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- [17] From the DSC data the activation energy of glass transition (E_g) in KSb₅S₈ using the Kissinger formula [15] was determined to be 476 ± 28 kJ mol⁻¹. This is a rather large activation barrier, compared to many non-stoichiometric chalcogen-rich glasses, and is probably a consequence of the rigid nature of the [Sb₅S₈]⁻ framework and of the absence of floppy regions or van der Waals gaps in the structure.
- [18] For glasses to nucleate, a certain period of incubation is necessary. As the heating rate increases, the time available for a desired temperature to be reached is reduced and thus crystallization lags and initiates at relatively higher temperatures, i.e., the T_p correspondingly shifts to higher temperature.
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Light-Induced Reorganization in Self-Assembled Liquid Crystal Gels: Electrically Switchable Diffraction Gratings**

By Yue Zhao* and Xia Tong

Diffraction gratings with electrically controllable efficiency are useful for many applications such as fiber optic switches and dynamically variable focal length lenses.^[1] Discovery of new materials and fabrication processes is of general interest for the development of devices operating with switchable diffraction gratings. Liquid crystals (LCs) are promising materials in this regard due to their large birefringence change induced by an electric field.^[2,3] Among the known approaches, holographically recorded gratings in polymer-dispersed liquid crystals (PDLCs) are probably the most interesting system, for which a photopolymerization-induced segregation of the polymer from the LC droplets is responsible for the grating formation.^[1] More recently, electric field induced pattern formation in cholesteric LCs has been exploited as diffraction gratings,^[4,5] and the use of a covalent polymer network was shown to be able to stabilize the gratings.^[6] If no polymer or polymer network is used with LCs, a spatially controlled electric field, produced from structured electrodes, is necessary.^[7,8] In this communication, we report, for the first time, that self-assembled LC gels can form electrically switchable volume gratings, without the use of patterned electrodes, as a result of a light-induced structural reorganization process in this type of materials.

A self-assembled LC gel refers to a LC host contained by a physical network of fibrous aggregates of a compound called a gelator.^[9,10] Generally, the gel is obtained by cooling a homogeneous mixture of the LC and gelator from the isotropic phase; the gelation occurs at lower temperatures when the aggregation of the gelator molecules is initiated by strong specific and highly unidirectional intermolecular interactions such as hydrogen bonding. We used a gelator containing amide groups and an azobenzene moiety in its structure (Fig. 1) and dissolved it in a cholesteric LC host composed of a nematic LC, BL006 ($T_{ni} = 115$ °C), and a chiral dopant R811 (Merck). As will be shown later, the use of a cholesteric LC is necessary because the threshold voltage for its switching in an electric field is very sensitive to the presence of a physical network, which is not the case for the nematic LC.^[10b,11] When the mixture with 1 wt.-% gelator and 5 % dopant is filled in a 10 μm, non-rubbed and indium-tin-oxide-coated electrooptic cell and rapidly cooled from 130 °C to room temperature, a LC gel is formed. After removal of the LC host in hexane, scanning

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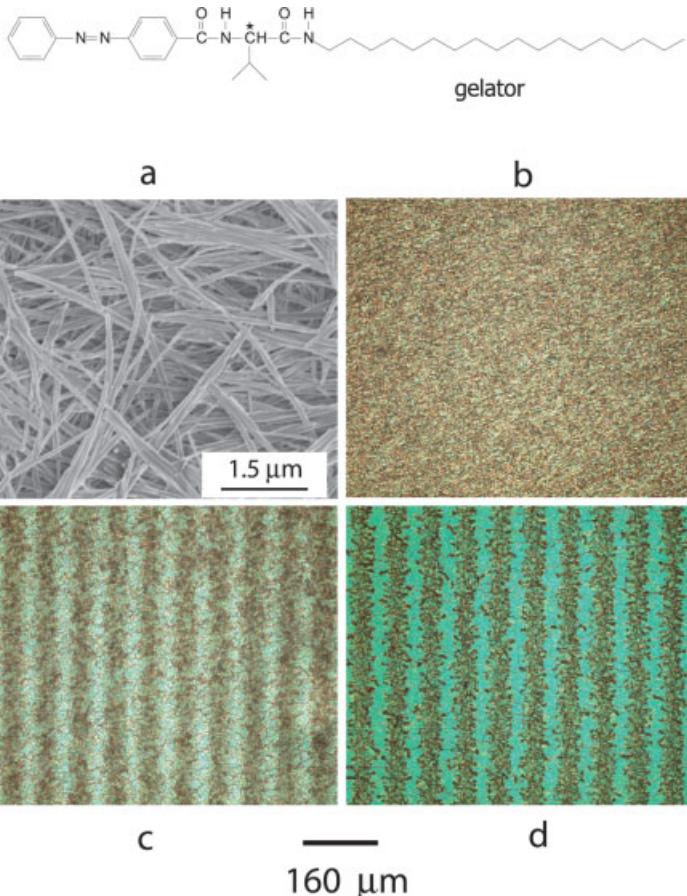


Fig. 1. a) SEM image of the fibrous aggregates formed in the LC gel with 1 % gelator and 5 % chiral dopant; b) photomicrograph of the LC gel before irradiation through a photomask c) after 5 min irradiation and d) after 10 min irradiation. The chemical structure of the gelator is shown at the top.

electron microscopy (SEM) revealed that the physical network is built up by very thin hydrogen (H-) bonded fibrous aggregates of the gelator, whose diameters are in the range of 100 nm, as shown in Figure 1a. When the LC gel is heated to temperatures close to the melting point of the aggregates, which is about 95 °C, and exposed to UV light (centered at 360 nm, ~100 mW cm⁻²) through a photomask with parallel transparent strips, a light-induced reorganization in the gel takes place, resulting in an apparent transport of the aggregates from the irradiated area to the non-irradiated area and, consequently, the formation of a grating. Figure 1 also shows polarizing photomicrographs of the LC gel before irradiation (Fig. 1b) and after 5 and 10 min irradiation at 90 °C (Figs. 1c,d). Before irradiation, the network is homogeneous and there is no grating, while after 5 min irradiation the grating emerges as the aggregates start to leave the irradiated area. With sufficiently long exposure time, 10 min in this case, the aggregates can be completely removed from the irradiated area, resulting in an increased network density in the non-irradiated area. The aggregates are orange-colored due to the azobenzene chromophore. Once the grating is formed, either under or without irradiation, subsequent cooling to room tem-

perature results in no reappearance of aggregates in the irradiated area, and the grating remains. The grating can be erased by heating the sample into the isotropic liquid phase to redissolve the gelator molecules in the LC, followed by cooling to ambient conditions. For reasons related to the use of photomask and the geometry of the electrooptic cell, which will be explained later, only gratings with large periods were obtained, and the diffraction is in the Raman–Nath regime.

Gratings formed in the self-assembled cholesteric LC gels display electrically switchable diffraction efficiency, which is determined by the response of the LC to the electric field. Figure 2 shows an example of plotting changes in the first order (+1) diffraction efficiency as function of applied voltage (root mean square, rms) for a grating obtained from the gel with 1 % gelator and 5 % chiral dopant (period ~40 μm). The first time the electric field is applied, i.e., during the first voltage increase, the diffraction efficiency changes in a complex fashion. It drops first at about 7 V and then rises at around 16 V. To explain this behavior, it is noted that after the grating is formed at 90 °C and cooled to room temperature the cholesteric LC in the irradiated area, where the aggregates are moved away by light, has a planar texture (Fig. 1d), for which the helices formed by the director (orientation direction) of the LC molecules are normal to the plates of the cell. Conversely, in the non-irradiated area the LC should have randomly aligned helices, i.e., the focal conic texture, due to the presence of the aggregates. This explains the refractive index modulation leading to the diffraction at the field-off state. As the electric field is applied, it destabilizes the planar texture into a disordered state (focal conic texture) as in the non-irradiated area, which results in the drop of diffraction at about 7 V. When the voltage is increased to 16 V, the electric field is strong enough to unwind

the helices in the irradiated area, resulting in a new planar texture with a different orientation than the original one. This causes a second increase in the diffraction efficiency at 16 V. After the first voltage increase, the grating is destroyed and the LC is in the isotropic liquid phase. Subsequent cooling to room temperature results in no reappearance of aggregates in the irradiated area, and the grating remains.

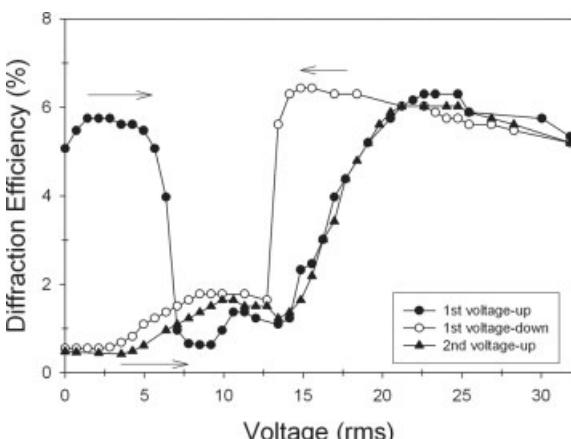


Fig. 2. 1st order (+1) diffraction efficiency vs. applied voltage for a grating formed in the LC gel with 1 % gelator and 5 % chiral dopant.

the helices and align the LC molecules along the field direction (homogeneous texture) in the irradiated area, but it cannot do that in the non-irradiated area because of the higher threshold voltage of the cholesteric LC caused by the physical network.^[11] This difference results in the switch from low diffraction to high diffraction. The high diffraction shows little changes until near 30 V when the LC in the regions with the aggregates starts to align along the field direction, which reduces the index modulation. On decreasing the voltage, the diffraction switches from high to low efficiency at about 14 V, which is caused by the usual hysteresis effect of the LC. At low voltages, the initial high diffraction efficiency is not recovered because the initial planar texture cannot be formed in the irradiated area. As is shown by the curve obtained during the second voltage increase in Figure 2, there is only one stable switch of diffraction efficiency at around 16 V on subsequent scans, which is associated with the transition between the focal conic and homogeneous texture of the LC in the irradiated area. Before switching, the low diffraction is due to the small index modulation mainly contributed by the physical network of aggregates, since the LC molecules are not aligned throughout the sample and incident light sees essentially an admixture of the ordinary (n_o) and the extraordinary (n_e) refractive index of the LC. As the electric field aligns the LC molecules in the irradiated area, the index modulation rises because incident light now sees the ordinary refractive index in these regions. This interpretation is also supported by the observation that the high diffraction efficiency shows no dependence on the polarization direction of the incident light. In essence, this electrical switchability of diffraction efficiency is made possible by the delayed orientation of the LC molecules in the gel areas due to the interaction with the physical network of the gelator. Note that the influence of the physical network on the threshold voltage is important only for cholesteric LCs. We used the gels prepared from pure BL006 to record gratings and found that the sharp increase in diffraction efficiency occurs over a narrow range of voltages (~2 V) before dropping to the low level. This is caused by the similar threshold voltages of BL006 with and without the network of the gelator.^[10b]

We also investigated the dynamic behavior of the electrically switchable diffraction gratings. The example in Figure 3 is the results for a grating (period ~40 μm) formed in the LC gel with 1% gelator and 2% chiral dopant. Figure 3a shows the recorded switching of the first order (+1) diffraction efficiency between 0 V (off-state) and 20 V (on-state) of a square-wave electric field (10 s duration for field-off and field-on). The switching behavior is relatively stable. Figure 3b shows the change in diffraction efficiency for a pulse wave of 200 ms, from which the high diffraction turn-on time was estimated to be about 65 ms and the turn-off time near 50 ms.

As mentioned above, this light-induced grating formation in self-assembled LC gels appears to be effective only at writing temperatures close to the melting of the hydrogen-bonded aggregates of the gelator; under the same experimental conditions no clear gratings were obtained at temperatures below

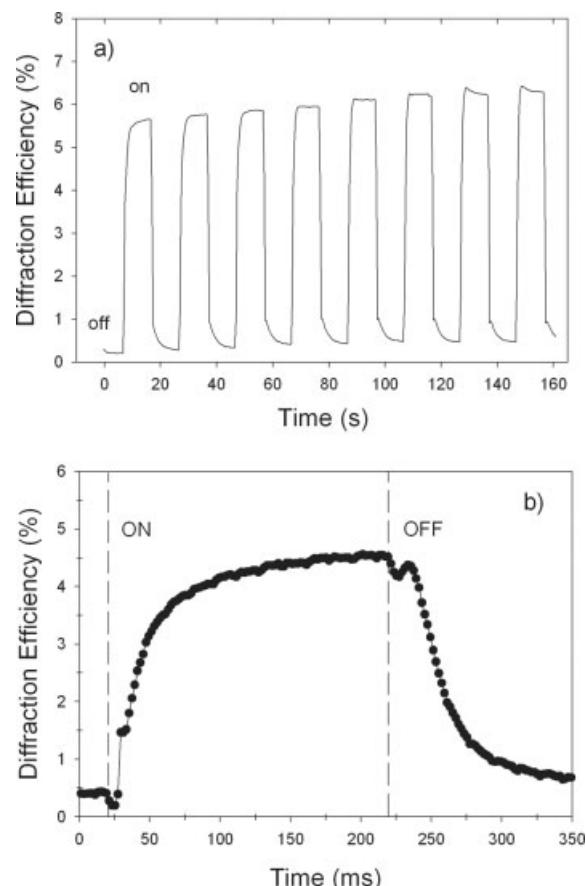


Fig. 3. Switching of diffraction efficiency for a grating formed in the LC gel with 1% gelator and 2% chiral dopant: a) between 0 V (off-state) and 20 V (on-state) of a square-wave electric field; and b) dynamic response to a pulse of 200 ms duration.

80 °C or above 95 °C. This observation implies that a gel state with the gelator molecules starting to dissolve in the LC may be crucial for the observed light-induced reorganization of the gel. We propose a mechanism that is schematically illustrated in Figure 4a. Before irradiation, there is a homogeneous physical network of thin aggregates dispersed in the LC, and the gelator molecules are on the verge of dissolving into the LC (it is also possible that a small amount of the gelator molecules are already dissolved in the LC). During irradiation through a photomask, light may alter the equilibrium in the irradiated area by bringing more gelator molecules from the aggregate surface to the LC. That is, the action of light may increase slightly the solubility of the gelator in the LC host through some mechanisms. Once this happens, the dissolved gelator molecules would diffuse from the irradiated area into the neighboring non-irradiated area in order to make the concentration uniform in the whole sample. In the non-irradiated area, an increased concentration of dissolved gelator molecules would shift the equilibrium towards the formation of more aggregates as the gelator molecules condense into the existing aggregates leading to a denser physical network. This process of light-triggered dissolving, diffusion, and aggregation will continue until the aggregates in the irradiated area

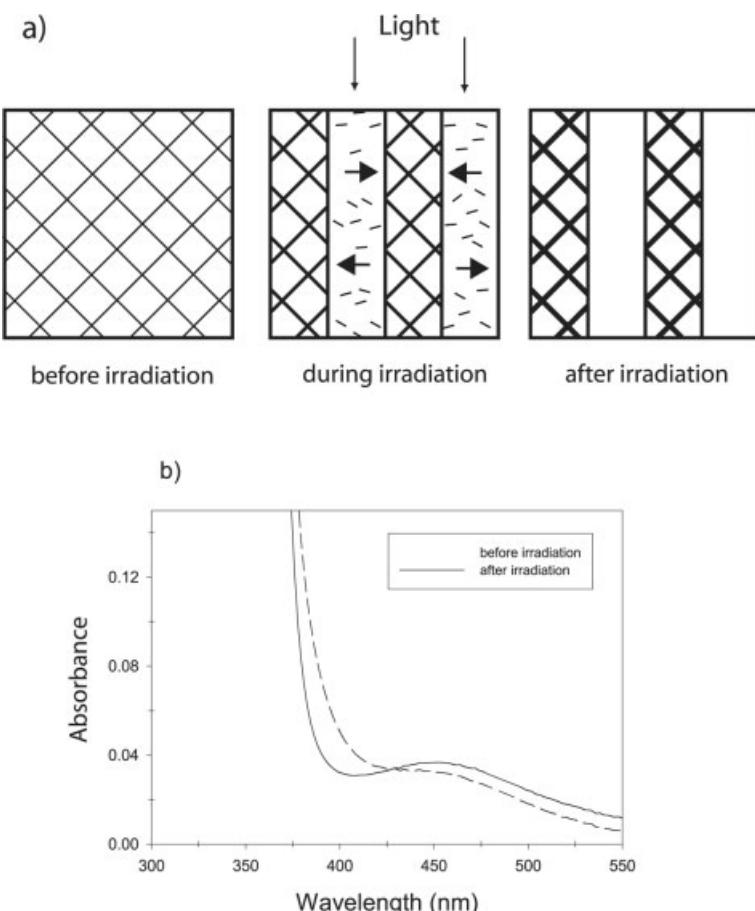


Fig. 4. a) Schematic illustration of the mechanism for the light-induced reorganization in self-assembled LC gels and the grating formation (the LC host is not shown); and b) UV-vis spectra of a mixture containing 1 % gelator before and after 10 min irradiation at 120 °C, showing the trans-cis photoisomerization of the azobenzene gelator molecules.

are completely dissolved and reformed in the non-irradiated area. Therefore, after irradiation a grating is formed with regions containing essentially the pure LC and regions of the LC gel. Now, the question raised is how light may increase the solubility of the aggregates in the irradiated area. We feel that the photochromic azobenzene group in the gelator (Fig. 1) may be a reason for that. Figure 4b shows the photoisomerization of 1 % azobenzene gelator dissolved in the cholesteric LC at 120 °C. Due to the low concentration of the gelator, the absorption peak of *trans*-azobenzene at 328 nm is masked by the absorption of the LC host, but after 10 min irradiation of the mixture the increased absorption peak of *cis*-azobenzene at about 450 nm is clearly visible. When the gel sample is heated to temperatures close to the melting of the aggregates, the gelator molecules on the surface would be mobile enough to undergo the *trans*-*cis* photoisomerization, which could not be detected because of the extremely low concentration of the azobenzene molecules involved (only those on the surface of the 1 % aggregates). In other words, it is likely that the gelator molecules on the surface were converted from the *trans*- to the *cis*-isomer by light, which may increase the surface tension

of the aggregates^[12] and, as a result, increase the dissolution of the gelator molecules in the LC. We also used irradiation light at 440 nm that is quite far from the absorption maximum of the azobenzene group. Gratings could still be formed even though the process seemed to be less efficient, since a longer irradiation time was needed. This observation suggests that a thermal effect due to irradiation may be another factor bringing more gelator molecules from the aggregate surface into the LC, although the intensity of light used is relatively low. To clarify the role of the azobenzene moiety, it would be interesting to see if this light-induced reorganization phenomenon can be observed in self-assembled LC gels with gelators bearing no azobenzene group. Even though the use of a photoactive gelator may not be a condition should a thermal effect be the trigger for the whole process, a large amount of gelator molecules on the aggregate surface must be important for the effectiveness of the grating formation mechanism. The LC gel studied in this work has nanometer-sized fibrous aggregates (Fig. 1a), which indeed ensures a large specific surface of the aggregates.

We have found a new type of materials, namely self-assembled LC gels, for making electrically switchable diffraction gratings based on a light-induced reorganization process. The grating is volume grating and extremely easy to fabricate. The grating obtained from the cholesteric LC gel investigated in this work may fall short for any practical applications, but given the possibility that this approach may be generalized for many systems made from designed gelators and various types of LCs, the interest of exploiting self-assembled LC gels and the new grating formation process leaves no doubt. The key to the grating formation in these materials is the ability for the physical network to be rearranged by light, which may necessitate a network built up from very thin fibrous aggregates and probably be helped by the presence of photochromic groups such as azobenzene. This work also demonstrates the unique features of the physical networks in LCs, because the observed light-induced structural reorganization obviously cannot occur in LC gels with covalent polymer networks. Finally, note that the large grating periods in this work were due to the limitation of the recording technique employed. Knowing that the electrooptic cell composed of two plates has a total thickness of 2.25 mm and the photomask is placed in front of it, incident light, which is diffracted when passing through the mask, should travel some 1125 µm before hitting the LC gel sample. This means that with masks having a period below 30 µm, incident light actually irradiates the whole film and no grating can be formed. Gratings with smaller periods might be obtained using other techniques such as holographic recording.

Experimental

The synthesis of the azobenzene gelator has already been reported [10a]. To prepare a self-assembled cholesteric gel, a homogeneous mixture of BL006/R811/gelator was obtained by first dissolving all compounds in a common solvent, tetrahydrofuran (THF), and then evaporating the solvent under reduced pressure. The mixture was flow-filled, at 130 °C, into the 10 µm thick electrooptic cell, and subsequent fast cooling of the mixture to room temperature resulted in the gel sample with a uniform physical network built up from nanometer-sized hydrogen-bonded fibrous aggregates (Fig. 1a). To record a grating, a photomask was positioned on one side of the cell, and the whole was placed into a thermostat hot stage and heated to the recording temperature. A curing system (Novacure apparatus) was used to expose the sample to UV light centered at 360 nm. Once the irradiation was completed, the sample was cooled to room temperature, either under irradiation or with irradiation turned off. The gratings were observed on a polarizing optical microscope (Leitz DMR-P apparatus). For SEM observations of the aggregates, on a Hitachi S-4700 FEG SEM apparatus, the cell was carefully opened and the cholesteric LC host was extracted in hexane.

For the measurements of the diffraction efficiency under an electric field, a He-Ne laser (633 nm) was used as the incident light source (normal to the cell) and the 1st order (+1) diffraction signal, I_d , was monitored using a high-speed photodetector (Displaytech) collected to a digital oscilloscope (Tektronix, TDS 420 A apparatus). A high-voltage waveform generator (WFG500 apparatus, FLC Electronics) was used to apply the AC (1000 Hz), square wave, and pulse electric fields through the cell. The diffraction efficiency was calculated as the ratio of I_d over the intensity of the laser beam before reaching the cell.

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The Chiroptical Properties of a Thermally Annealed Film of Chiral Substituted Polyfluorene Depend on Film Thickness**

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In the last decade, there has been an intense and sustained research effort into polyfluorenes as potential materials for

polymeric organic light-emitting diodes (OLEDs), with special attention afforded to them as blue emitters.^[1] The observation of thermotropic liquid crystallinity^[2] paved the way for alignment of the polymer chains and subsequent linearly polarized electroluminescence (LPEL) from a fluorene homopolymer.^[2,3]

Introduction of two chiral side chains at the 9-position of the fluorene units of the polymer was found to result in large degrees of circular polarization in the absorption and emission of the polyfluorene.^[4] It was noted that the chiroptical properties in the solid state developed after annealing the film in its thermotropic phase. Subsequent work by Chen and co-workers on well-defined fluorene oligomers showed that, thermally annealed films of these materials have a vitrified chiral nematic morphology.^[5] For the treated films of fluorene polymers, a Schlieren texture is observed when placed between crossed polarizers, indicating that for these polymers a glassy cholesteric structure is also likely. More recently, large degrees of circular polarization in the absorption of light have been reported for films of a poly(phenyleneethylene) derivative.^[6] These materials also show liquid crystal (LC) behavior and the circular dichroism for the polymer with chiral pendants develop after thermal treatment.

The degree of circular polarization in absorption can be expressed as a dissymmetry ratio or g_{abs} value. This number is defined as $2(A_L - A_R)/(A_L + A_R)$ where A_L and A_R denote the absorbance of left- and right handed circularly polarized light, respectively. When only left (right) circularly polarized light is absorbed, $g_{abs} = 2(-2)$. For thermally treated films of chiral polyfluorene, $g_{abs} > 0.15$ has been reported by Oda et al.,^[4] while for the poly(phenyleneethylene) values up to $g_{abs} = 0.38$ have been found by Bunz and co-workers.^[6] These g values are considerably higher than those reported for films of poly(thiophene),^[7] poly(thiénylenevinyline),^[8] and poly(*p*-phenylenevinyline) (PPV)^[9] type conjugated polymers with chiral sidechains. These polymers do not show liquid crystalline behavior at high temperature and circular dichroism measurements are commonly performed on spincoated films. Typical values observed are $g_{abs} \sim 10^{-2}$ for polythiophene derivatives and $g_{abs} \sim 10^{-3}$ for PPV derivatives. For polythiophene, values up to 8×10^{-2} have been observed for aggregates of polymer in decanol.

Values of g_{abs} up to 10^{-3} are compatible with the range of g values observed for dipole allowed spectroscopic transitions of small chiral molecules in dilute solution. For these solutions, the circular polarization necessarily reflects the local chirality of the chromophore. For these dilute solution the g value is an intrinsic property of the molecule and therefore independent of concentration and optical path length. Comparing typical g_{abs} values for solutions of small molecules with those reported for the LC conjugated polymers, we note that values for the polymers are exceptionally large and the intriguing question arises whether they can be regarded as an intrinsic, microscopic property of the polymer or whether the circular polarization originates on a macroscopic level from, e.g., interference or selective reflection effects. The latter are well-known phenomena for low molecular weight LC materials.

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