Light-scattering liquid-crystal composites with reduced off-axis haze

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Abstract — The majority of liquid-crystal (LC) composites operating in a light-scattering mode suffer from scattering of the obliquely incident light in the field on-state (off-axis haze effect). This is evident in the angularly selective viewing-angle characteristic with a maximal transmittance corresponding to the normally incident light. We consider methods to control the viewing-angle characteristic of polymer-dispersed LC (PDLC) and filled LC. For PDLC samples, this control is realized by modification of the refractive index of the polymer matrix with highly refractive nanoparticles (NP) having a low rate of aggregation in the polymer. By proper optimization of NP concentration, one can bring the refractive index of the polymer matrix in the range needed to reduce the haze problem. In filled LC, the viewing-angle curve depends on the refractive-index mismatch between the LC and NP. By optimization of this parameter, one can flatten the angular characteristic or obtain selectable viewing angles in the desirable range. These results allow for the construction of optical shutters and a scattering-type LCD with controllable viewing-angle characteristics, particularly with low off-axis haze.

Keywords — PDLC, filled LC, nanoparticles, off-axis haze.

1 Introduction

It is well known that liquid crystals (LC) are rather attractive for displays and optical shutters because of two basic effects: electrically controlled birefringence and electrically controlled light scattering. The former effect, clearly apparent in monodomain LC samples, is widely used in nearly all commercial display technologies.¹ The latter one is in polydomain samples, especially in composite systems having enhanced light-scattering properties.^{2,3} The devices working in the scattering mode are distinguished by high brightness, wide viewing angle, and fast switching. They do not require polarizers, compensation films, alignment layers, and high uniformity of cell gap that greatly simplifies the preparation process. At the same time, samples of this type have several drawbacks, which seriously hinder their wide application. First is the low switching contrast, which cannot compete with the contrast of monodomain samples commonly used in commercial LCDs. Several approaches recently developed give hope to substantially improving this characteristic.^{4–6} The other problem concerns the angular dependence of the transmittance of the sample in the transparent field-on state; the sample is highly transparent at normal observation but is hazy at oblique viewing angles. This characteristic is known as off-axis haze.⁷ In the present work, we consider LC composites with controllable viewing-angle characteristics and, in particular, with substantially reduced off-axis haze over a wide viewing angle. The conditions for this improvement and of the underlying scattering mechanisms are discussed.

2 Background

2.1 Electrically controlled scattering LC composites

To explain our approach, first we shall consider scattering LC composites capable of electric switching and the corresponding mechanisms of light scattering. The most wellknown of these are polymer-dispersed $L\bar{C}$ (PDLC)^{3,8,9} and filled LC.^{10–13} Typical PDLCs consist of a large amount of spherical bipolar droplets of LC with positive dielectric anisotropy chaotically distributed in the polymer (P) matrix. The refractive index of the polymer, n_p , is usually adjusted to be close to the ordinary refractive index of LCs, n^{o}_{LC} . In the absence of a driving electrical field, the composite strongly scatters light. The main reason for scattering is the mismatching of the refractive indexes of the polymer and chaotically oriented LC droplets. The droplets orient in the external electric field so that their refractive index $n_{LC} = n^o{}_{LC}$ approaches n_p . For normal light incidence, this matching of the refractive indices leads to the high on-state transmittance of PDLC layers. At the same time, for oblique light, the refractive indices are mismatched causing the scattering to become stronger while the on-state transmittance becomes smaller. In other words, off-axis haze takes place.

The other popular system, filled LC, is a suspension of small particles (usually aerosils) dispersed in LC. The light scattering in such composites is mainly caused by the disturbance of the LC director field in the vicinity of particles or their aggregates. In the field on-state, the layer of filled LC becomes transparent due to the orientation of the LC, leading to a large diminution of the number of orientational defects. The angular characteristics of light transmittance have not been systematically studied for these composites. This problem is considered in Sec. 4.

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2.2 Reduction of off-axis haze

Great efforts have been spent to overcome off-axis haze in PDLCs.^{14–16} The proposed methods concern driving schemes, insertion of additional films, and optimization of the optic properties of coexisting phases. First of all, to reduce haze at any viewing angle, it is suggested to power the PDLC sample by using a square-wave power source rather than by a sine-wave power source.¹⁴ A square-wave voltage reverses the polarity almost instantaneously so that the liquid crystal does not have time to switch between a distorted and an aligned state, and thus the match between n^{o}_{LC} and n_p is maintained during each cycle of the applied field. The simple way to reduce off-axis haze consists of placing the polarizer in front of the PDLC film with the polarization direction perpendicular to the light-incidence plane.¹⁵ Indeed, light from this type of polarization (s-wave) is not sensitive to changes in the light-incidence angle. However, the polarizer decreases the brightness of PDLC film at least by a factor of 2, which makes this method not very practical. The other solution¹⁶ suggests the replacement of the isotropic polymer by an anisotropic one with ordinary and extraordinary refractive indices approximately equal to n^{o}_{LC} and n^{e}_{LC} , respectively. A serious problem in this method is the selection of an anisotropic medium and its proper alignment in PDLC.

Finally, the most common and realistic way consists of the selection of isotropic polymer with the refractive index satisfying the inequality^{7,14}

$$n_{LC}^o < n_p \ll n_{LC}^e. \tag{1}$$

When Eq. (1) is true, then the peak maximal transmittance for the PDLC is shifted away from normal incidence so that range of viewing angles with sufficiently high on-state transmittance becomes broader.

3 Our concept

To lower off-axis haze in PDLC, we propose a modified version of the last approach. To meet inequality (1), instead of choosing polymer with a proper n_p , we suggest the modification of the polymer already optimized for the formation of PDLC with certain LC. According to Ref. 17, the refractive index of the polymer can be continuously varied by doping it with nanoparticles having distinctly different indices of refraction. In good approximation, the refractive index of modified polymer \tilde{P} is a linear function of NP concentration:

$$n_{\widetilde{P}} = n_P \phi_P + n_{NP} \phi_{NP}, \qquad (2)$$

where φ_P and φ_{NP} are volume fractions of the polymer and NP.¹⁸ This nonsynthetic method to broaden the refractiveindex range of organic materials is currently rather attractive for a number of optical applications (telecommunication devices, information systems, *etc.*). To realize this approach, we can control the phase separation in LC-P-NP systems by directing the NP to polymer. Fortunately, in the course of photoinduced phase separation of a LC-P-NP mixture with a small amount of NP, the NP are mainly absorbed in the polymer phase.¹⁹ Presumably, they serve as polymerization centers and thus are naturally involved by the polymer. However, at small concentrations (less than 3 vol.%), NP do not noticeably change the PDLC morphology of LC-P composites. To be in a range of low concentrations and, simultaneously, to achieve a considerable increase in n_p , the NP of highly refractive materials, usually inorganic ones, should be utilized.

A similar concept can be used to reduce off-axis haze of filled LC. If NP filler intensively aggregate with an aggregate size comparable or higher than the wavelength of the scattered light, the mismatch in the refractive indices of LC and NP can be an important factor in light scattering. It can be the major factor in the field-on state, when orientation defects disappear. For SiO₂ particles, $n_{NP} = 1.46$ is close to $n^o{}_{LC} = 1.52$ so that layer of filled LC should be transparent for on-axis viewing, but hazy at off-axis viewing. To reduce the off-axis haze, by analogy with inequality (1), the refractive index of NP should satisfy

$$n_{LC}^o < n_{NP} << n_{LC}^e. \tag{3}$$

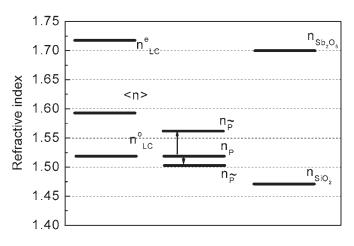
The light scattering due to the refractive-index mismatch should not be essential in the composites with weakly aggregated NP (the aggregate size is lower than wavelength of scattering light). Consequently, the viewing-angle characteristics of these composites in the on-state are expected to be rather flat.

4 Experimental

As LC, we used nematic liquid-crystal-mixture E7 from Merck. It is characterized by a relatively broad mesophase (from -10° C to 58°C), strong optical anisotropy (refractive indices are $n^{o}_{LC} = 1.5216$ and $n^{e}_{LC} = 1.7462$) and positive dielectric anisotropy ($\Delta \varepsilon = 13.8$). As photopolymerizing material, we employed UV curable adhesive No. 65 from Norland, Inc. (USA) with a refractive index $n_p = 1.524$. The combination of LC E7 and Norland's photopolymer No. 65 has been widely used to prepare LC composites.^{5,19,20}

As NP filler, we employed silica (SiO_2) and antimony pentoxide (Sb_2O_5) NP obtained from Nissan Chemical Industries, Ltd. (U.S.A.) as colloidal dispersions in methanol with a particle content of 30 and 35 wt.%, respectively. The particles were of spherical shape with a diameter of 10–20 nm (SiO_2) and 7–11 nm (Sb_2O_5) . The selection was determined by the substantial difference in their refractive indices, which are 1.46 and 1.7 for SiO₂ and Sb₂O₅ NP, respectively. As can be seen from the refractive-index diagram shown in Fig. 1, the refractive indices of SiO₂ and Sb₂O₅ NP are close to the ordinary and extraordinary refractive indices of LC.

In fact, we studied only LC-P-NP composites based on colloidal NP. Instead of filled LC, we used their polymerstabilized analogues because of intensive aggregation of col-



 $\ensuremath{\textit{FIGURE 1}}$ — The refractive-indices diagram for the nanoparticles, polymer, and LC.

loidal NP. The formation of a polymer network in such suspensions is considered to be an effective method to stabilize the structure and prevent phase separation.²¹ All composites were prepared in the following manner. At first, the LC was thoroughly mixed with the colloidal dispersion of NP. In this process, alcohol was evaporated. The complete removal of alcohol was achieved by applying a vacuum to the suspension at 10^{-2} Torr over 0.5 hours. In the following, a photopolymer was added to the LC-NP composition. The components were mixed under red light to avoid polymerization, which is not desirable at this stage. The mixtures with different concentrations of LC, P, and NP were prepared. They can be divided in two groups with concentrations $\varphi_P = 32-35$ vol.%, $\varphi_{NP} = 1-3$ vol.%, $\varphi_{LC} = 62-67$ vol.% and $\varphi_P = 5$ vol.%, $\varphi_{NP} = 1-3$ vol.%, $\varphi_{LC} = 92-94$ vol.%, respectively. The first group includes NP-doped PDLC, while the second one comprises polymer-stabilized filled LC.

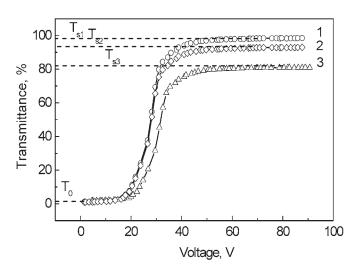


FIGURE 2 — *T* (*U*) dependences for pure PDLC and PDLC doped with NP. 1) $\varphi_{LC} = 63$ vol.%, polymer – the rest; 2) $\varphi_{LC} = 63$ vol.%, $\varphi_{NP} = 1$ vol.% (SiO₂ NP), polymer – the rest; 3) $\varphi_{LC} = 63$ vol.%, $\varphi_{NP} = 1$ vol.% (Sb₂O₅ NP), polymer – the rest.

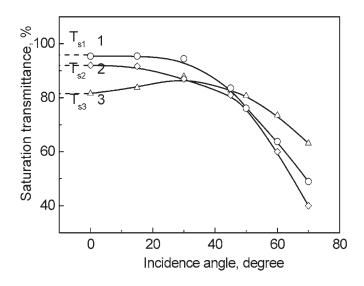


FIGURE 3 — The saturation transmittance T_s for pure PDLC and PDLC doped with NP as a function of light incidence angle θ . The light is p-polarized. 1 – pure PDLC ($\varphi_{NP} = 0$); 2 – $\varphi_{SiO_2} = 1$ vol.%; 3 – $\varphi_{Sb_2O_5} = 1$ vol.%. For all cases, $\varphi_{LC} = 63$ vol.%, the rest is polymer content. The lines are just guides for the eyes.

Additionally, as conventional filled LC, we prepared an E7-aerosil suspension using aerosil A300 from Degussa with an averaged particle size of 10 nm. The concentration of aerosil was about 1 vol.%.

We used a typical setup to measure the optical transmittance T of a laser beam ($\lambda = 635$ nm) through the tested sample as a function of applied voltage U.²² The angular dependency of the transmittance at a voltage corresponding to saturation of the T(U) curve were measured by means of sample rotation at 0, 15, 30, 45 50, 60, and 70°. The testing light was polarized in the incidence plane. For such polarization, the refractive index of the LC depends on the light incidence that maximizes off-axis haze.

5 Results and discussion

5.1 Nanoparticle-doped PDLC

The T(U) curves for pure PDLC and PDLC doped with 1 vol.% of inorganic nanoparticles of Sb₂O₅ and SiO₂ (Sb₂O₅-PDLC and SiO₂-PDLC, respectively) are presented in Fig. 2. Qualitatively, all dependences are rather similar: T rises with an increase in the driving voltage U until reaching saturation level T_s . At the same time, saturation transmittances are different for different values of the applied voltage. In the following measurements of the angular characteristics the applied voltage corresponded to the saturation state of all samples.

Figure 3 shows the dependences of $T_s vs.$ incidence angle θ for pure PDLC and PDLC doped with different types of NP. One can see that for the case of pure PDLC and SiO₂-PDLC samples (curves 1 and 2, respectively), T_s rapidly decreases with θ . In contrast, for Sb₂O₅-PDLC, T_s changes slowly and nonmonotonically going through maximum (curve 3 in Fig. 3). In the range of 0–50°, the transmit-

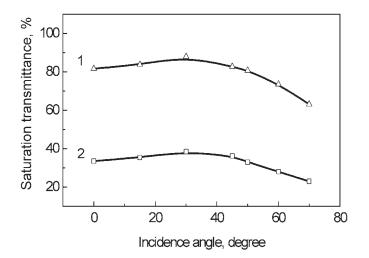


FIGURE 4 — The saturation transmittance $T_s vs.$ incidence angle θ curves for PDLC doped with a different amount of Sb₂O₅: 1 – φ_{NP} = 1 vol.%, 2 – φ_{NP} = 3 vol.%. For all cases φ_{LC} = 63 vol.%, the rest is polymer content. The lines are just to guide the eyes.

tance T_s changes insignificantly, while for the higher angles it does not decrease abruptly as is the case for other samples. In other words, the $T_s(\theta)$ curve considerably flattens, which is evidence of a substantial suppression of the off-axis haze.

According to Fig. 4 the shape of the $T_s(\theta)$ curve for the Sb₂O₅-PDLC composite is rather sensitive to the concentration of NP. The increase in the NP concentration leads to better flattening of the $T_s(\theta)$ curve, but, simultaneously, to lowering of the sample transmittance for small angles of light incidence. Thus, the concentration of NP should be well optimized to obtain samples with acceptable on-axis and off-axis electro-optic contrast.

According to the model described in Sec. 2, NP modify the refractive index of the polymer phase. These changes can be qualitatively analyzed using the refractive-indices diagram (Fig. 1). The refractive index of SiO₂ NP is close to the polymer refractive index. So particles of this type practically do not influence the refractive index of the polymer. This explains why the $T_s(\theta)$ curves for PDLC and SiO₂-PDLC are of similar shape and close each to other. For both of these systems, at $\theta = 0$, $n_P \approx n_{LC} = n^o{}_{LC}$ so that light transmittance is high. With an increase in θ , n_{LC} increases approaching n^{e}_{LC} at grazing incidence. This causes an increase in the refractive-index mismatch, $n_{LC} - n_P$, and light scattering in PDLC. From the Lambert-Beer law, light scattering is a decaying exponential function of the refractive-index mismatch and the optical path length. Consequently, light transmittance for SiO₂-PDLC should demonstrate a rapid decrease with the incidence angle that corresponds to experimental observation (curve 2 in Fig. 3).

The refractive index of antimony pentoxide is considerably higher than n_p (close to $n^e{}_{LC}$). So, doping PDLC with Sb₂O₅ NP increases the refractive index of polymer phase \tilde{P} so that $n^o{}_{LC} < n^e{}_{\tilde{P}} << n^e{}_{LC}$ (Fig. 1). Since $n^e{}_{\tilde{P}}$ does not match $n^o{}_{LC}$, T_s at normal incidence is not as high as that for PDLC and SiO₂-PDLC composites. The mismatch of n_p and n_{LC} changes non-monotonically with growth of θ : it

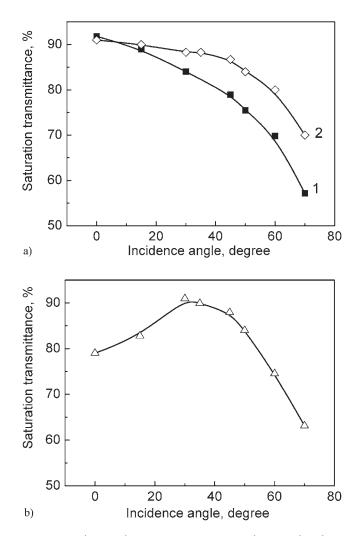


FIGURE 5 — The sample transmittance $T_s vs.$ incidence angle θ for LC filled with (a) SiO₂ and (b) Sb₂O₅ NP. (a) 1 – E7-colloidal SiO₂ NP ($\varphi_{\text{SiO}2}$ = 0.5 vol.%, φ_p = 5 vol.%); 2 – E7- aerosil A300 (φ_A = 1 vol.%); (b) E7-colloidal Sb₂O₅ NP ($\varphi_{\text{Sb}2O_5}$ = 0.5 vol.%, φ_p = 5 vol.%). The lines are just for guidance.

decreases, vanishes at some light incidence angle $\theta_c \neq 0$, and finally increases. The scattering tendency determined by the nonmonotonic refractive-index mismatch superimposes with the scattering trend connected to passing light increases with incidence angle. The result is that $T_s(\theta)$ is nonmonotonic: maximal transmittance is shifted away from the normal incidence to the angle $\theta_c \neq 0$. Simultaneously, the range of angles corresponding to relatively high transmittance becomes broader (curve 3 in Fig. 3).

5.2 Filled LC

Figure 5 shows the $T_s(\theta)$ curves measured for filled LC on the basis of (a) colloidal particles and (b) aerosil. The $T_s(\theta)$ curves for E7–SiO₂ samples (both colloidal particles and aerosil) show a monotonic decrease, while the curve for E7–Sb₂O₅ composite is nonmonotonic. In other words, curves for E7–SiO₂ and E7–Sb₂O₅ composites show angularly selective transmittance with, correspondingly, the on-axis and off-axis position of the transmittance maximum.

These results can be also explained by assuming light scattering due to the refractive-index mismatch between coexisting phases, *i.e.*, LC and NP aggregates. The refractive-index difference may go up or down depending on the particle sort. In E7 samples filled with silica NP, this value increases with θ . Superimposed with the lengthening of the optical path, this leads to the monotonic decrease of light transmittance [Fig. 5(a)]. For Sb₂O₅ filler, $n_{\text{Sb}_2\text{O}_5} \approx n_{LC}^{e}$ and the refractive-index difference decreases with incidence angle, approaching zero at the grazing angles. The nonmonotonic behavior of the $T_s(\theta)$ curve [Fig. 5(b)] is due to interplay between two competing factors: lengthening of the optical path and reducing the refractive-index mismatch between LC and NP that tend to decrease and to increase the transmittance T_s , respectively. Evidently, by changing the refractive index of NP, one can tune the viewing angles of the composites. In this particular case, when condition (3)is satisfied, the viewing-angle curve should be rather flat, indicated by low off-axis haze.

It is important to compare the $T_s(\theta)$ curves for the composites on the colloidal silica and aerosil base [curves 1 and 2 in Fig. 5(a)]. Despite the use of the same material and similar sized NP, these composites demonstrate different angular characteristics; the off-axis haze is considerably stronger in case of colloidal NP. This might be caused by a different aggregation rate of particles of these two types. According to Ref. 21, colloidal NP aggregate much stronger than aerosil particles. So, for smaller aerosil aggregates, the light scattering due to the refractive-index mismatch between LC and NP should be less effective than for larger aggregates of colloidal SiO₂ NP. Thus, the weakening of NP aggregation results in another trend to lower the off-axis haze of filled LC.

6 Conclusions

We considered viewing-angle characteristics of light-scattering LC composites containing inorganic NP in the translucent field-on state. It is shown that the doping polymer phase with NP having an optimized index of refraction can substantially reduce the off-axis haze of PDLC. The filling particles share the same properties as the polymer. Particularly, they modify the refractive index of polymer matrix bringing it in the range needed to reduce the haze at oblique viewing. Instead of finding polymer with a suitable refractive index, this method allows us to adapt any other polymer by changing its optical properties with NP. We believe that doping of the polymer phase with inorganic NP may lead to other improvements in PDLC, such as enhancement of the mechanical and thermal stability²³ and reduction of LC content in polymer.

The refractive properties of NP also influence the viewing-angle characteristic of filled LC, especially for the case of intensive particle aggregation (*e.g.*, colloidal NP). In

the common case, the viewing-angle curve is angularly selective and the angular range of maximal transmittance depends on the difference $n_{NP} - n^o{}_{LC}$. In a particular case, when the condition $n^o{}_{LC} < n_{NP} << n^e{}_{LC}$ is met, the viewing-angle curve should flatten.

The obtained results indicate the methods that can be used to control the viewing-angle characteristics of LC composites operating in a light-scattering mode. In particular, they help to prepare composites with low off-axis haze to be rather promising for optical shutters and scattering-type LCDs.

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