



Molecular Crystals and Liquid Crystals Science and Technology. Section A. Molecular Crystals and Liquid Crystals

Publication details, including instructions for authors and subscription information:

<http://www.tandfonline.com/loi/gmcl19>

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Version of record first published: 24 Sep 2006

To cite this article: P. Sixou, C. Gautier & H. Guillard (2001): Passive Broadband Reflector: Elaboration and Spectral Properties, *Molecular Crystals and Liquid Crystals Science and Technology. Section A. Molecular Crystals and Liquid Crystals*, 364:1, 665-678

To link to this article: <http://dx.doi.org/10.1080/10587250108025035>

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Passive Broadband Reflector: Elaboration and Spectral Properties

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We present passive broadening obtained by assembling two layers of cholesteric photo-polymerisable monomers having different pitches at the same temperature. A more or less large diffusion occurs between the two layers, which is dependent of various parameters such as degree of cross-linking of the two layers, temperature dependence, time evolution at a given temperature, thickness of the layers... The presented work consider the spectral properties of the sandwich structure and its correlation with the diffusion profile at the interface between the two layers. The band shape of the reflected and transmitted light were studied. Then, the reflected intensity shows a widening of the reflection associated with the existence of a pitch gradient inside the sample, which can be blocked by photo-polymerisation. At short time, the two peaks corresponding to each liquid crystal polymer are still present but the diffusion occurs at the interface. After a longer time, the broadening band still decreases and a single large peak is observed.

Keywords: broadband; passive reflector; optical properties

INTRODUCTION

A first technique to produce passive reflector in a wide range of wavelength is polymerisation of a cholesteric network in which the helical pitch gradually changes over the cross-section in the film [1-4]. Recently, J.F. Li *et al.* developed, with this technique, a single layer reflective polarizers, which can be electrically switched from broadband to narrow band and vice versa [5]. An alternative of this method to produce passive filters consists in superposition of network layers constituted with different helical pitches [6].

This preliminary experiments purpose the study of superposition of nematic and cholesteric layers containing various chiral agents, leading to a passive broadband reflector. The interest of this work consists in the pitch gradient blocking, at any time during chiral agent diffusion process, using photo-polymerisation. Moreover, these films will allow to understand the complex diffusion phenomena that occur at the interface of the layers, on the one hand, and, on the other hand, will be applied to make reflective polarising filters [7,8].

The nematic and cholesteric monomer films were deposited by spin coating and then assembled. The composition gradient in the cell induces diffusion over the film thickness leading to a gradually change of the helical pitch over the cross-section of the cell. The influence of chiral agent nature and contact time on wavelength shift were studied using optical measurements.

EXPERIMENTAL

The polymer layer was deposited by spin coating with a P-6000 Spin Coater. However, the solid polymer material must be fluidified for coating and two methods were used : heating and dissolution in solvent. Concerning the heating, a thermal element was inserted in the top of the spin coater and the layer was deposited at about 80°C. In spite of the fluidity improvement with temperature, polymer material must be diluted in cyclohexanone solvent, which is also a solvent of liquid crystal molecules.

Then, this paper reports the influence of adding a chiral agent in two cases of polymer nature on optical properties of one single layer coated on glass substrates. The passive cell is performed by the assembly of two polymer layers under vacuum. One of both films

contains polymer without chiral agent and the second one contains polymer with a chiral agent. The texture containing the pitch gradient is then blocked by photo-polymerisation, after a suitable time of the chiral agent diffusion process. So, we have studied the influence of the chiral agent nature on optical properties versus contact time and the influence of thermal annealing after polymerisation using a commercial UV light source, wavelength centred at 365 nm. The observations of layer morphology complete the spectral characterisations.

RESULTS AND DISCUSSION

In a first time, we present the optical properties of various chiral agents added to the BN5 liquid crystal (LC). Figure 1 expresses the variation of absorption maximum λ_{\max} for LC mixtures containing several kind of chiral agents. It appears that NXO and NXL, respectively left and right chiral agents, lead to the same helical pitch, whereas adding the CMR and CML ones lead to a lower helical pitch. Moreover, a weak variation of λ_{\max} versus temperature is observed for these mixtures up to 80°C, which corresponds to the beginning of the isotropic phase transition.

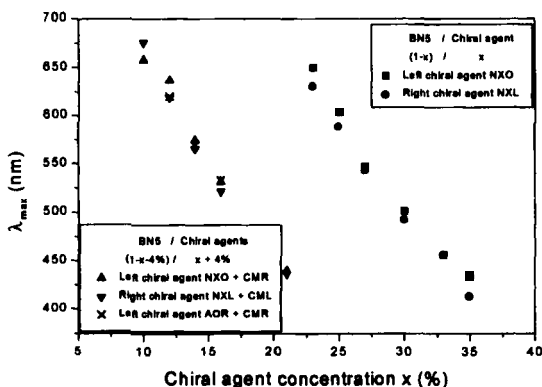


FIGURE 1 Influence of chiral agent nature and concentration on λ_{\max} .

One single layer

In a first time, the Spin Coater parameters were optimised. Four parameters are adjustable : acceleration, spin speed, deceleration and spin time. Moreover, a remark was extracted from the first experiments: the quantity of polymer material must be sufficient in order to obtain a good cover rate. The main experiments of coating polymer layers are summarised in Table 1.

The observations of polymer layer coated with pure polymer (100% concentration) expressed a poor cover rate and a very inhomogeneous thickness for each coating conditions, demonstrating the necessity of using diluted polymer. However, the use of diluted polymer improves the film homogeneity but thinner layers are obtained. Then, the deposition of thicker layer requires several successive coatings. Best results are observed with a 66% polymer concentration and two successive layers. A thin film with good homogeneous thickness can be obtained either by coating one single layer or by increasing spin speed and spin time parameters.

Polymer Concentration (%)	Number of layers	Observations
100	1	Very irregular layers, Inhomogeneous thickness, Poor cover rate
50	1	Best homogeneity of the thickness
	2	Good homogeneity, Thicker layer
66	2	<i>Good homogeneity, Thicker layer</i>

TABLE 1 Observations of polymer layer according to various coating conditions.

The optical properties were measured on various single layers. In the case of SN11 nematic polymer, an absorption peak appears by adding right chiral agent, in very high concentration (Figure 2).

The decrease of chiral agent concentration is accompanied by a shift of the absorption maximum towards the high wavelengths. Moreover, this phenomenon leads to a decrease of the maximum intensity that means the optical properties strongly depends on chiral agent concentration, in this concentration range.

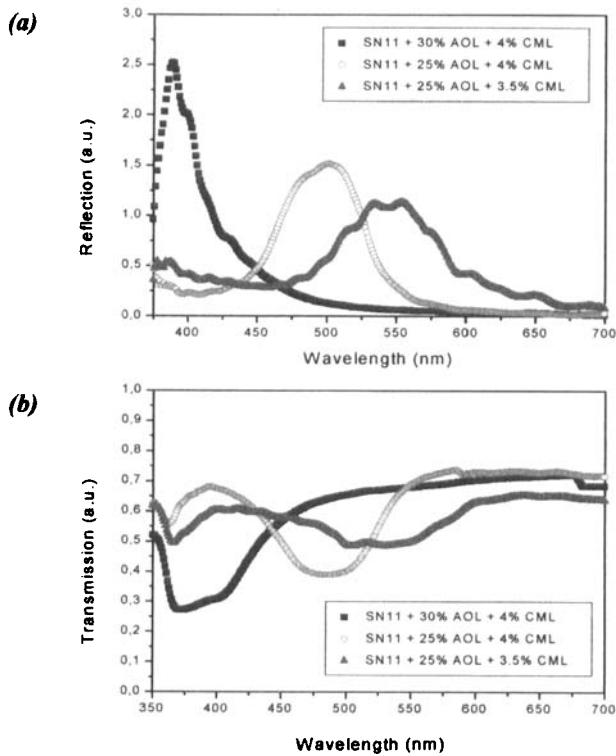


FIGURE 2 Optical properties of one single layer of SN11 nematic polymer : (a) reflection measurements, (b) transmission measurements.

In the case of CM14 left cholesteric polymer, the absorption maximum of the layer without chiral agent is observed at about 518 nm (Figure 3). By adding a left chiral agent, the absorption maximum shifts towards the low wavelengths. On the contrary, λ_{max} shifts towards the high wavelengths by adding a right chiral agent to polymer layer. The λ_{max} shift depends on chiral agent concentration.

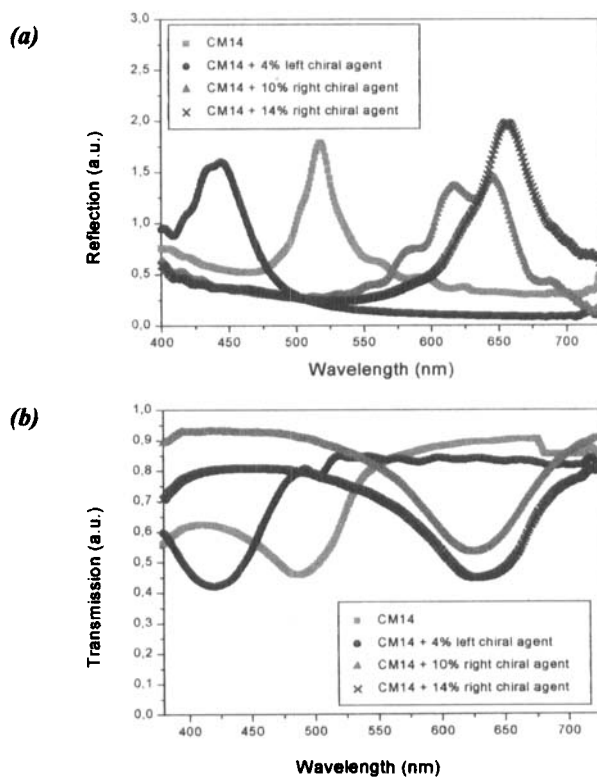


FIGURE 3 Optical properties of one single layer of CM14 left cholesteric polymer : (a) reflection and (b) transmission measurements.

Moreover, the reflection and transmission intensities at λ_{\max} are similar in every case that means optical properties are independent of chiral agent concentration for this polymer, in this concentration range. Then, from these results, we chose to study the optical properties of passive cell constituted with CM14-based layers.

Passive cells

The influence of left chiral agent nature on reflection and transmission properties is respectively presented in Figure 4 and Figure 5.

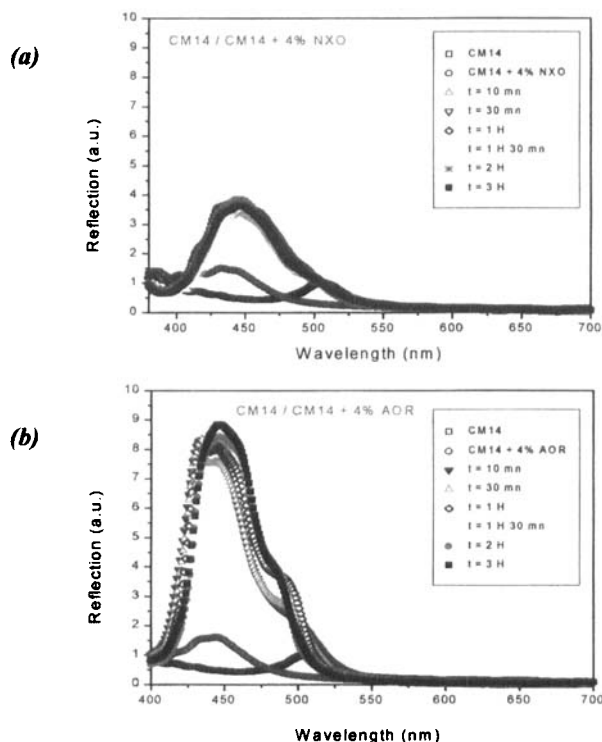


FIGURE 4 Influence of left chiral agent nature on reflection properties of passive cell : (a) NXO chiral agent, (b) AOR chiral agent.

With the both kinds of agent, an increase of reflective intensities is observed immediately after the contact due to a partial rearrangement of polymer molecules. However, this increase is stronger in the case of AOR chiral agent than in the case of NXO one. Moreover, similar transmission properties are observed with the both chiral agents.

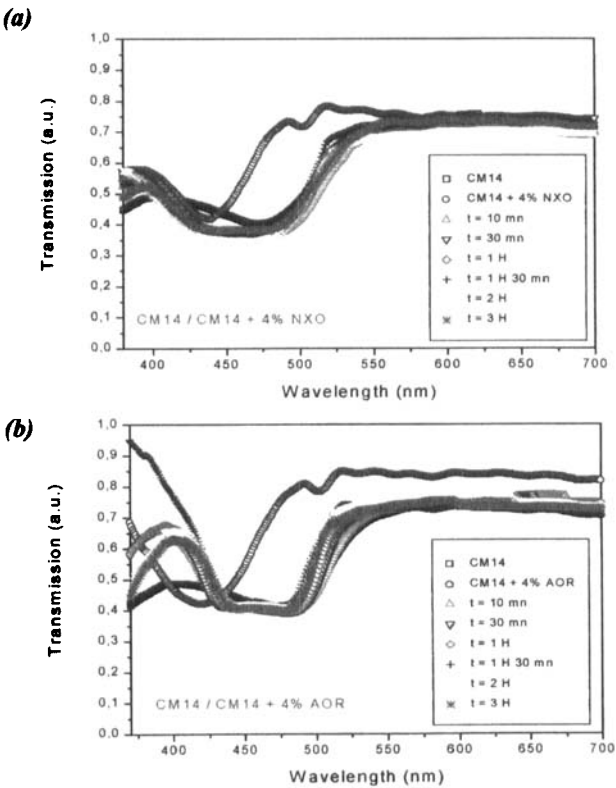


FIGURE 5 Influence of left chiral agent nature on transmission properties of passive cell : (a) NXO chiral agent, (b) AOR chiral agent.

The variation of broad band versus contact time is given in Figure 6. In both reflection and transmission spectra, the absorption

broadband shape of passive cell corresponds to the sum of each layer absorption peak. Moreover, the broadband shape evolves weakly in the studied range of contact time that means the chiral agent diffusion is ended. This rapid diffusion is probably due to a weak chiral agent concentration, on the one hand, and to layers too thin, on the other hand. In a general way, the AOR left chiral agent gives best results, particularly concerning the reflection properties and will be preferentially used to the NXO one.

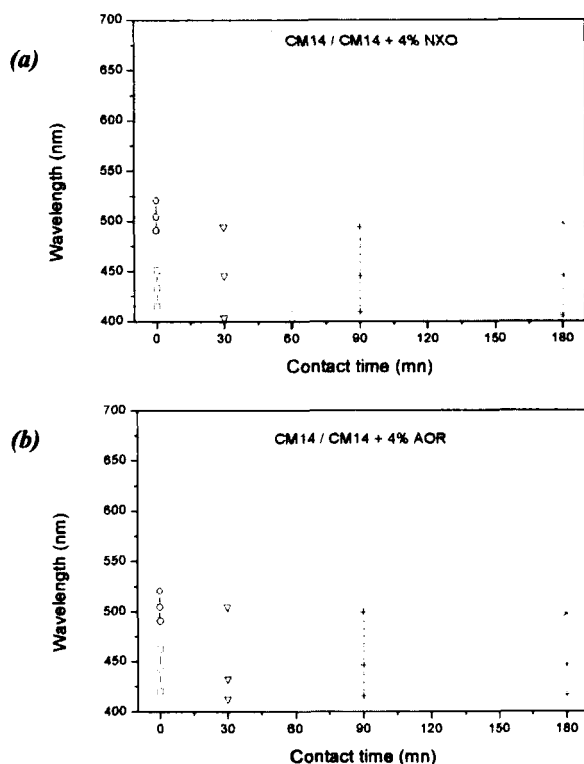


FIGURE 6 Variation of broad band versus contact time - Influence of left chiral agent nature : (a) NXO chiral agent, (b) AOR chiral agent.

The influence of right chiral agent nature on reflection and transmission properties is respectively presented in Figure 7 and Figure 8. As above-mentioned in the case of left chiral agent, an increase of reflective intensities is observed immediately after the contact, which is stronger with NXL chiral agent than with AOL one.

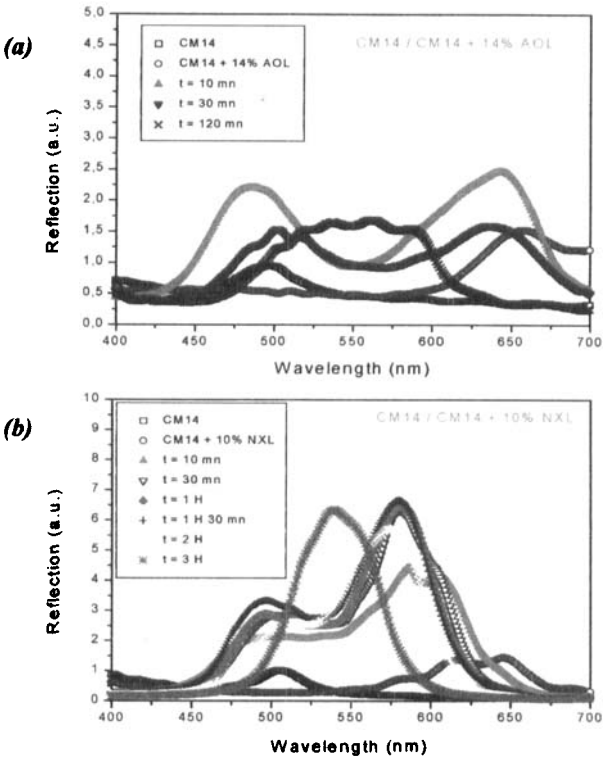


FIGURE 7 Influence of right chiral agent nature on reflection properties of passive cell : (a) AOL chiral agent, (b) NXL chiral agent.

Moreover, a best contrast of transmission is obtained using NXL agent. In the both cases, reflection and transmission

measurements indicate a strong evolution of the absorption broadband shape after contact traducing the diffusion of chiral agent molecules.

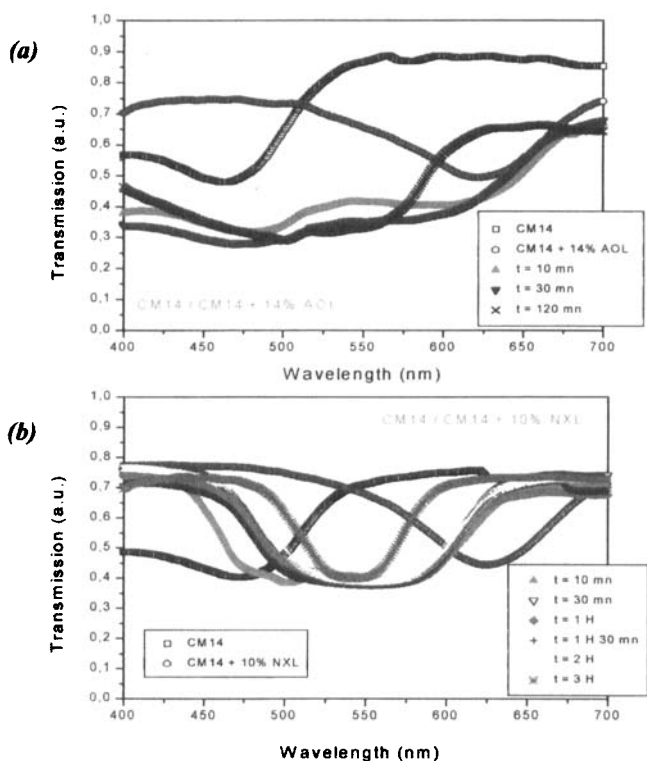


FIGURE 8 Influence of right chiral agent nature on transmission properties of passive cell : (a) AOL chiral agent, (b) NXL chiral agent.

The variations of broad band versus contact time given in Figure 9 put in evidence the evolving of absorption peak shape leading to a strong decrease of absorption wavelength range for a long contact time beyond 120 mn.

Moreover, the absorption maximum corresponds to the middle between the two initial peaks.

In a general way, the NXL right chiral agent gives best results in both the reflection and transmission properties and will be preferentially used than the AOL agent.

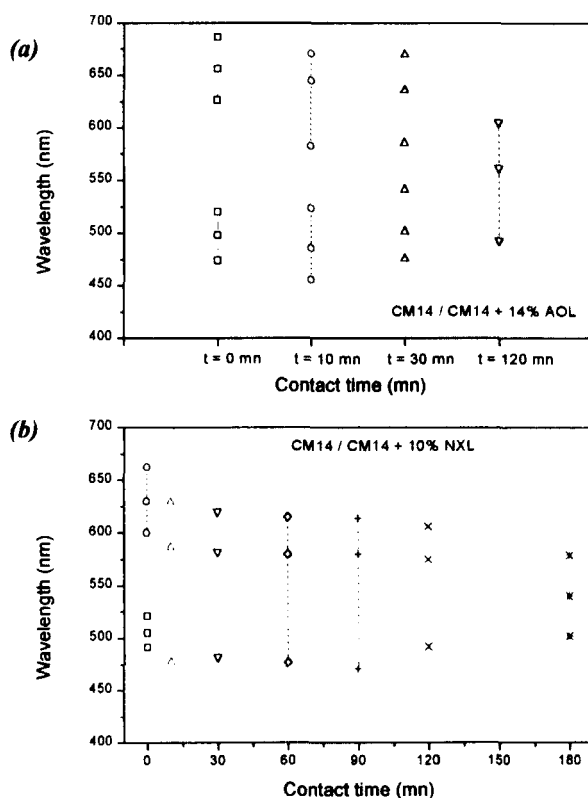


FIGURE 9 Variation of broad band versus contact time - Influence of right chiral agent nature : (a) AOL chiral agent, (b) NXL chiral agent.

Then, the following step concerns the polymerisation of polymer layers, which allows to stop on the suitable configuration. Figure 10 shows the influence of temperature on passive cells after polymerisation. One can be seen that increasing temperature doesn't modify the reflection properties up to 95°C that is very interesting for many applications and particularly for active cells.

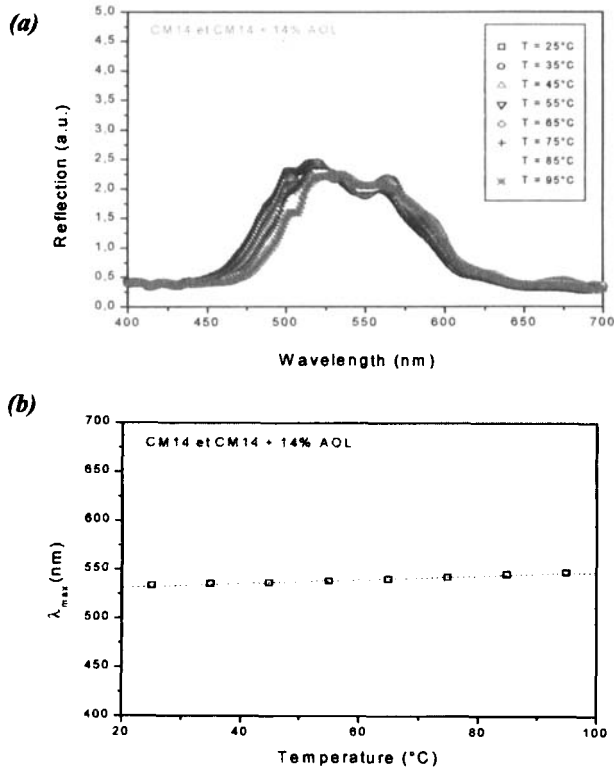


FIGURE 10 Influence of temperature on optical properties of passive cell after polymerisation : (a) reflection measurements, (b) variation of λ_{max} versus temperature.

CONCLUSION

The whole results indicate that the nematic and cholesteric polymers can be doped by adding chiral agents, adjusting the reflection wavelength in a wide range. Several kinds of stacked doped layers were studied. The optical properties of the sandwich traduce the diffusion of the chiral agent versus contact time. The kinetics of chiral agent diffusion in polymer layer depends on the film thickness and chiral agent concentration. At the beginning of the contact, the two peaks corresponding to each liquid crystal polymer are still present and, after a longer time, the broadening band still decreases up to a single peak. The absorption wavelength range can be chosen and fixed by photopolymerisation at the suitable time of the diffusion ; the passive cells remain then unmoved by thermal annealing up to 95°C.

ACKNOWLEDGMENTS

This work was supported by CCE under contract JOE3-CT970068

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