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Adsorption processes during the filling of a display with a smectic A LC mixture

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The separation of a smectic A liquid crystal (LC) mixture during the filling of a large area display was studied. This process is governed by the selective adsorption of the mixture's ionic dopant on the surface of the display substrates, leading to a non-uniform distribution of the dopant in the LC over the display area and resulting in defective regions. Conductivity measurements were performed to indicate the distribution of the dopant concentration in the LC over the display area. The consequences of mixture separation on the structure of the LC and on the display's electro-optical properties are discussed. Factors to reduce the extent of separation of the mixture are revealed using a general theory of chromatography. Increasing the cell gap, decreasing the area of the glass surface by reducting the gap between the ITO electrodes, and increasing the back pressure during filling, effectively reduce the significance of the separation process. The results obtained can be used when designing and filling displays based on the electrically reversible memory effect in a smectic A LC or displays based on dynamic light scattering in a nematic LC.

1. Introduction

In recent years interest in displays possessing bistable optical properties has increased. The main feature of such displays is the ability to remember information once written. Bistability removes the restriction on the number of rows that can be addressed by the passive matrix method. Furthermore, with bistable displays the state of the pixel is only required to change when the information content changes; this may be beneficial to the overall power consumption. Another desirable property is a wide viewing angle. Displays based on the electrically reversible memory effect in smectic A liquid crystals (LC), discovered in the late 1970s [1, 2], show both these useful characteristics.

In this paper we discuss one challenge in the manufacturing of large area smectic A (SmA) LC displays based on the electrically reversible memory effect. It was noticed that regions located far from the filling hole of the display in certain configurations were defective in terms of either a poor response or the absence of a response of the LC to the applied driving signals. Assuming that the LC is homogeneous prior to filling the display, this non-uniform effect was suggested to be caused by a separation of the LC mixture on the inner surface of the display substrates during the filling process. It was noted that with large cell areas and small cell gaps this can result in the appearance of defective regions far from the filling hole.

Previous papers (see, for example, [3, 4]) have discussed the influence of ion adsorption on the substrate on the display performance. The ionic dopant precipitating out of the LC onto the surface of the substrates during the filling of test cells with a SmA LC was mentioned by Coates *et al*. [2], but to the best of our knowledge there are no literature reports on how to minimize this process.

In order to test our hypothesis, conductivity and electro-optical properties were measured in display cells based on the electrically reversible memory effect in a

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SmA LC. Our results are presented and discussed in the context of a chromatographic separation of the LC mixture during the filling process. We also discuss methods to reduce this chromatographic effect.

2. Description of the electro-optical effect and role of ionic dopant

The electrically reversible memory effect utilizes the transition between the two distinct structures of the SmA liquid crystal: namely, the focal-conic (FC) and the homeotropic (HT) structures. A low frequency electric field initiates an electrohydrodynamic (EHD) instability in the SmA LC, resulting in strong scattering of the incident light. On removing the electric field, the EHD vortices relax to the FC structure, with an accompanying small decrease in the light scattering efficiency. The application of an electric field with a frequency higher than the critical frequency leads to a dielectric reorientation of the LC molecules. The molecules of the LC mixture with a positive dielectric anisotropy will then be aligned with the electric field vector, thus leading to the formation of a transparent HT structure. Figure 1 shows a schematic representation of these transitions. The transitions to the HT structure and to the EHD instability both have a threshold character. The visco-elastic properties of the smectic A LC make these extreme states, as well as intermediate ones, stable for a long time. No alignment layers are required for this effect. The advantages of devices based on the electrically reversible memory effect are the multistability (long term pixel memory), grey scale definition, wide viewing angle, the efficient light scattering of the FC structure and good transparency of the HT structure of the SmA LC leading to good contrast, a design containing no polarizers, a simplified production process, and the availability of both reflective and transmitting modes.

In order to generate the electrohydrodynamic instability, the LC mixture must contain ions. When a low frequency field is applied, the movement of the ions leads to a distortion of the SmA layer structure, and eventually to a circular movement of the LC material in the plane of the substrates. In order to obtain optimal display properties, a carefully chosen ionic dopant is added in controlled amounts to a deionized LC matrix. When the period of the applied electric field is much shorter than the space-charge relaxation time, the ions are 'frozen', and the dielectric reorientation of the LC molecules occurs.

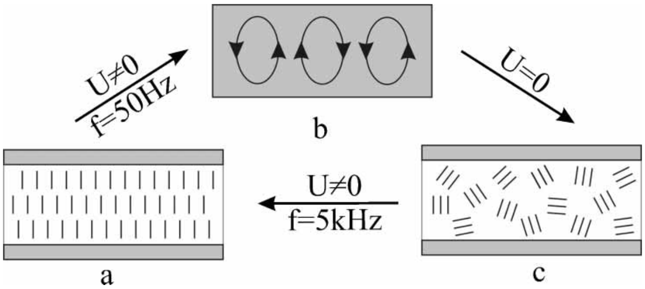
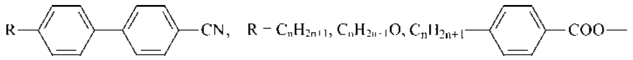


Figure 1. Schematic representation of the electrically reversible memory effect: (*a*) the homeotropic light transmitting structure of the LC; (*b*) the electrohydrodynamic instability (top view through substrate); (*c*) the focal-conic light scattering structure.

3. Materials and technique

In order to investigate the separation of the LC mixture during the filling of a display, the following materials and equipment were used. For the SmA matrix we made use of a multicomponent mixture containing alkylcyanobiphenyls, alkyloxycyanobiphenyls and cyanobiphenyl esters of alkylbenzoic acid:



The mixture showed a smectic A phase between −20 and 73°C. The dielectric anisotropy ($\Delta\varepsilon = \varepsilon_{\parallel} - \varepsilon_{\perp}$) and the anisotropy of conductivity ($\Delta\sigma = \sigma_{\perp}/\sigma_{\parallel}$) of the LC matrix were 11.2 and 2.1, respectively. A tetraalkylammonium salt of an alkyloxybenzoic acid was used as the ionic dopant.

The experiments were performed with displays having a 25.4 cm diagonal. We noted that the separation of the doped LC mixture was more pronounced when the ITO electrodes were covered by a layer of silica. A smaller cell gap further enhanced the separation of the mixture. In order to provide good conditions for the separation of the LC mixture, an 11 μm gap and substrates with ITO electrodes covered by silica were used. Some reference to good and moderate results of filling, however, are made for samples prepared without a silica coating.

Due to the high viscosity of the SmA LC mixture, the displays were filled with the LC in the isotropic state at $T = 120$°C using a two-hole method in which one of the substrates has two holes in opposite corners. Air was evacuated from the display package as well as from the retort containing the LC mixture until the pressure reached 0.01 mbar. The LC was then directed to the filling hole and a dry nitrogen back pressure of 780 mbar was applied in order to accelerate the filling.

The conductivity of the LC was measured using an HP4284A precision LCR meter with a 1 V a.c. signal voltage at a frequency of 200 Hz. Electro-optical characteristics were obtained by measurements of monochromatic light transmission. A red semiconducting laser ($\lambda = 650$ nm) was used as a light source. The light intensity was detected by a silicon pin diode photodetector, the detector signal being amplified by a trans-impedance amplifier. The transformations of the LC structures were studied using a Leitz Orthoplan polarizing microscope.

4. Results and discussion

The defect regions which arise because of the separation of the LC mixture during the filling of the display are shown schematically in figure 2; two cases of separation are depicted. The LC inlet filling hole is shown in the top right corner of the drawings. In typical good cells, the entire display is addressable with equal electro-optical parameters over the whole display area. Figure 2 (*a*) shows the situation for a large cell with no silica coating on the ITO electrodes. In this configuration, most pixels are addressable (region 1), i.e. the LC can be switched between FC and HT structures. In two small regions (region 3) close to the edges of the cell near the outlet hole, the LC does not respond to a low frequency electric field, so the FC structure cannot be formed there.

When the cells were prepared with a silica coating on the inner surfaces of the substrates, mixture separation became more pronounced. The stronger polarity of silica than that of ITO causes a better adsorption of ionic dopants and explains the difference in the extent of separation between display cells with and without a silica coating. As seen in figure 2 (*b*), the addressable region became much smaller while the defective region became correspondingly larger. The latter region was completely unresponsive to low frequency drive signals even with voltage levels exceeding the normal driving voltage by several times. The boundary between these regions had a shape that corresponds well with the shape of the LC mixture front in this region during filling.

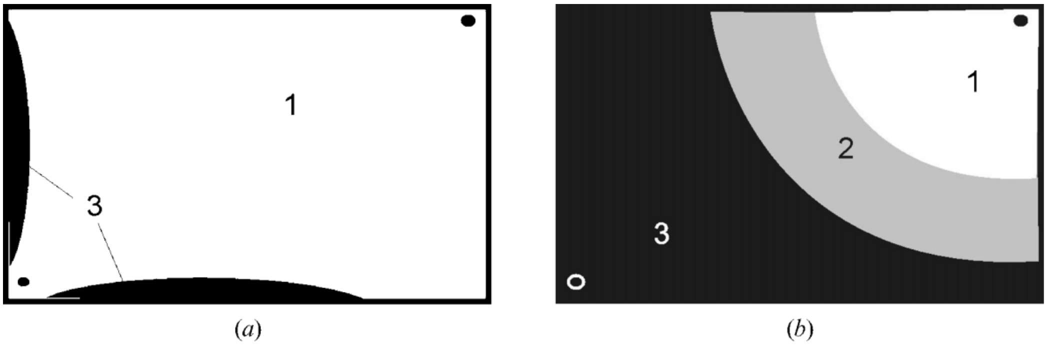
One peculiar effect of the silica coating is that a good homeotropic alignment is obtained upon filling. This happens due to the long alkyl chains of the adsorbed ionic dopants acting like an alignment layer on the LC mixture [5]. This alignment reduces the stability of intermediate FC light scattering states (grey levels). After removing the low frequency electric field, weakly scattering regions relax to the HT structure.

In cells with silica covered ITO, we see a relaxing defective region; figure 2 (*b*), region 2. The LC in this region responds poorly to applied low frequency signals. The FC structure with saturated light scattering cannot be formed without increasing the driving voltage, so a relaxation from a weakly scattering state to HT structure occurs. It was possible to reduce the size of this relaxing defective region by increasing the driving voltage.

After filling the cells having silica covered ITO, the LC was perfectly homeotropically aligned in the addressable and relaxing regions. The microscopic texture of the addressable region in crossed polarizers is shown in figure 3 (*a*). In the non-responding defective region the LC had a weakly scattering FC structure, figure 3 (*b*).

All the observations described indicate a reduced amount of ionic dopant in the bulk of the LC in defective regions. There is no electrohydrodynamic instability induced in the non-responding defective region and there is no dopant adsorbed by the surface of the silica to produce an alignment layer. In the relaxing defective region there was sufficient dopant available in the bulk of the LC to provide the functionality of an adsorbed alignment layer, but the remaining ionic dopant in the LC was insufficient to provide a strong EHD instability and produce a stable strong scattering FC structure.

The threshold voltage of an EHD instability in the addressable region increases on approaching the



(*a*) (*b*)

Figure 2. Functionally different regions obtained due to separation of the LC mixture in the display during filling: (*a*) common case for separation on ITO and glass in a large area cell; (*b*) worst case of separation on a silica coating. 1 = addressable region, 2 = relaxing defective region, 3 = non-responding defective region. Filling inlet holes are at the upper right corners of the drawings, outlet holes are at the lower left corners.

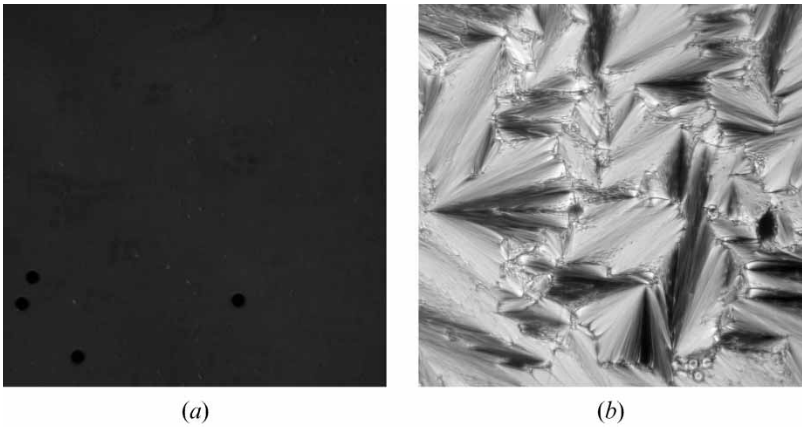

(*a*) (*b*)

Figure 3. Optical textures of (*a*) the SmA LC homeotropicaly aligned by the ionic dopant adsorbed on the silica surface, and (*b*) of the LC in a weakly scattering focal-conic state on the surface of silica with no dopant present. The pictures were taken with crossed polarizers in regions 1 and 3, respectively at a 140 × magnification; see figure 2 (*b*).

relaxing defective region and undergoes a high increase in the relaxing region. This observation also supports our hypothesis that a deviation in performance for defective display regions located far from the LC inlet hole is caused by the decrease in the ionic dopant concentration in the bulk of the LC, as a consequence of the selective adsorption of the ionic dopant on the surface of the substrates during the filling of the display cell. As the LC mixture fills the cell, the filling front of the LC gradually becomes depleted of the ionic dopant. This causes a non-uniform distribution of ionic dopant concentration over the display area. The concentration of ionic dopant actually approaches zero in the non-responding defective region.

To test further our hypothesis concerning mixture separation, estimates were made of the ionic dopant concentration distribution in the volume of the LC matrix over the display area. The conductivity of electrolyte depends on the bulk concentration of charge carriers according to

$$\sigma = \alpha z F c (u_+ + u_-) \qquad (1)$$

where σ is the specific conductivity for a binary electrolyte, α is the degree of dissociation, z is the charge of the ion, F is the Faraday constant, c is the concentration of ionic dopant in mol m^{-3}, and u_+ and u_- are the mobilities of the cation and anion, respectively. Thus, it is possible to estimate the distribution of ionic dopant bulk concentration over the display area by the distribution of the local conductivity of the LC.

Accurate measurement of the local conductivity of the LC is not straightforward in matrix LCD cells since d.c. measurements should be avoided and capacitive coupling between electrodes will affect the results of a.c. measurements. Nevertheless, a.c. measurements were performed in two configurations on a silica coated cell corresponding to that shown in figure 2 (*b*). First, the measuring voltage was applied between a single column and a single row of the cell. Since the conductivity of the SmA LC is anisotropic, and thus depends on its alignment, the LC in the entire cell had first been transferred to the HT state by the application of a high frequency signal. The results of such measurements are shown in figure 4.

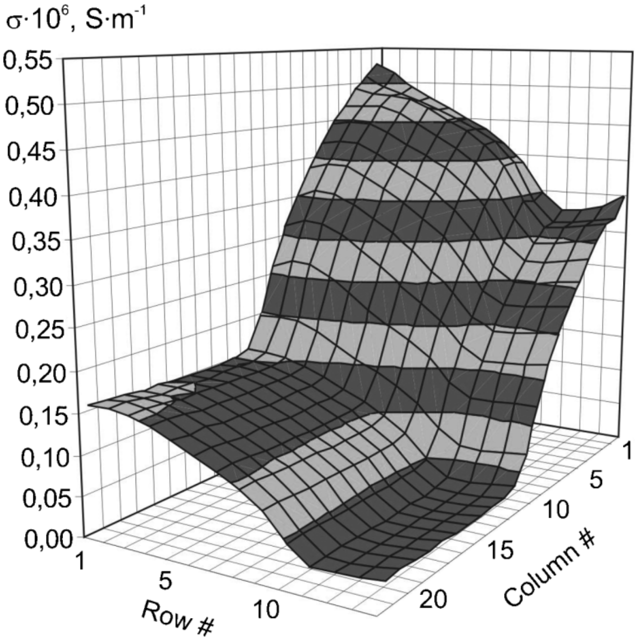


Figure 4. Distribution of LC conductivity over the area of display cell. Pixel (1;1) corresponds to the inlet filling hole, pixel (14;22) corresponds to the outlet hole.

From this figure it can be seen that the region of highest conductivity is near the filling hole. In this region the concentration of the ionic dopant is a maximum and the EHD instability can be easily generated. The conductivity decreases with the distance from the filling hole. At a certain distance, the conductivity reaches a plateau value indicating that no ionic dopant is left in the volume of the LC. The location of the plateau corresponds to the non-responding defective region, figure 2 (*b*), region 3.

Unfortunately these measurements do not show the exact conductivity of each single pixel. The values also include contributions from the remaining pixels of the row and of the column, as they are capacitively coupled through the rest of the display. This makes the plateau distorted—the measured conductivity becomes higher for pixels included in the rows or columns containing pixels with higher conductivity. Attempts at recalculating the data failed due to high relative errors in the measured data. Qualitatively, however, such a recalculation would have the effect of levelling the plateau down to the value of the conductivity near the outlet hole.

It was possible, however, to measure precisely the LC conductivity in entire columns. This was accomplished by applying the probe voltage between a single column and all the rows. Information on the conductivity and thus the dopant distribution in the second dimension along the columns is, however, lost when using this procedure. Figure 5 shows the conductivity of the columns measured in this configuration.

We note that the conductivity of the LC in columns near the filling hole is close to the conductivity of the initial mixture, whereas the conductivity measured near the outlet filling hole corresponds to the conductivity of the LC matrix before the ionic dopant is added.

The process of separation of the LC mixture observed during filling the display can be considered as a chromatographic process. Multifold adsorption–desorption processes of the mixture's components take place on the walls of the flat capillary during filling. The frequency and the speed of these processes influence the migration rate of the substances along the capillary. As a result, different components of the originally uniform LC mixture may migrate with different velocities, and a separation of the mixture components occur. The separation of the LC matrix's components is insignificant because they all have similar polarity. However, the ionic nature of the dopant differs significantly from those of the LC matrix components. The ionic dopant moves more slowly along the capillary than the LC matrix, which results in a non-uniform distribution of dopant concentration over the display area. The separation of the LC mixture during the filling of the display is thus an inevitable process, as long as the mixture contains components which interact differently with the surface of the display substrates. The separation cannot be eliminated completely, but the separation efficiency may be reduced according to the general principles of chromatography [6].

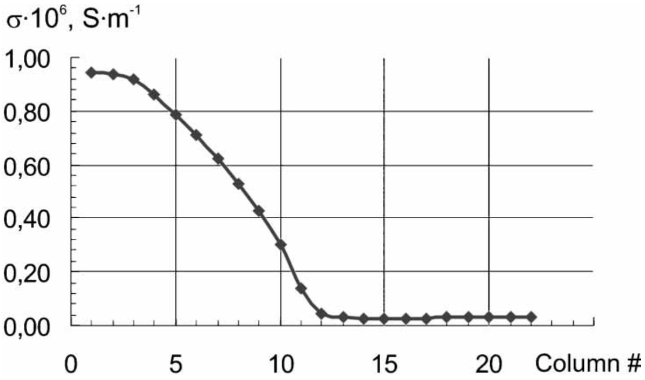


Figure 5. Conductivity of the LC in the individual columns of the display.

The filling process is similar to thin layer plane chromatography, but the separation takes place between two substrates in a flat capillary under pressure. This resembles high performance liquid chromatography, which is one of the most efficient modern chromatographic methods of separation. The LC matrix can be treated as a mobile phase, while the substrate surfaces can be regarded as a stationary phase. We will now consider factors that influence the separation efficiency and discuss methods by which to reduce it.

The separation efficiency depends strongly on the nature of the stationary phase, of the adsorbing substance and of the mobile phase. It is not possible to reduce the extent of separation by varying the LC matrix or the ionic dopant, as these materials are optimized with respect to the display of electro-optical properties. As we have shown already, the modification of the ITO surface by the introduction of a more polar silica layer increases the separation efficiency, figure 2 (*b*). The application of low polarity coatings will reduce the undesirable effect of separation, but the change of substrate surface may also affect the electro-optical properties of the display. In addition, such a modification would add a complication to the display production. Since the polarity of the glass surface is close to the polarity of silica, the polarity of ITO is lower than the polarity of glass. This means that the reduction of the gap between adjacent electrodes on the same glass will accordingly reduce the separation efficiency, as this will decrease the area of free glass surface.

We now consider how the dimensional parameters of the display cell and the conditions of filling will influence the separation efficiency. The efficiency of a chromatographic column is expressed by the number of effective plates N [6]:

$$N=\frac{L}{H} \tag{2}$$

where L is the length of the chromatographic column and H is the height equivalent to an effective plate (HEEP). The larger the number of effective plates means better separation.

For open tubular chromatographic columns the HEEP is determined by the Golay equation [7]:

$$H=\frac{2D_\mathrm{m}}{u}+\frac{f_1(k)r^2}{D_\mathrm{m}}u+\frac{f_2(k)d_\mathrm{f}^2}{D_\mathrm{s}}u \tag{3}$$

here D_m and D_s are the diffusion coefficients of the adsorbing substance in the mobile phase and in the stationary phase, respectively; u is the velocity of the mobile phase, r is the radius of the column, d_f is the thickness of the stationary phase film, $f_1(k)$ and $f_2(k)$ are functions depending on the column geometry, and capacity factor $k=n_\mathrm{s}/n_\mathrm{m}$, where n_s and n_m are the number of the molecules of the adsorbing substance in the stationary phase and in the mobile phase, respectively.

The first term of equation (3) represents the contribution from the diffusion of the adsorbing substance in the mobile phase, and dominates the HEEP value for very small velocities. A slow filling process should thus result in a more uniform distribution of components in all directions due to the molecular diffusion of the ionic dopant under the influence of a concentration gradient. For large area displays impractically slow filling processes would, however, be required. Taking into account a low value of the diffusion coefficient for the adsorbing substance in a liquid phase, this term in the equation can be neglected for practical velocities of the mobile phase.

The second and the third terms are the contributions of resistance to the adsorbing substance mass-transfer from the mobile phase to the stationary phase (second term) and *vice versa* (third term). The radius r of the tubular column in the Golay equation came from Einstein's diffusion law and it implies a distance over which the molecule has to diffuse, at random, from the bulk of the phase to reach the interface. We can substitute this parameter with half the distance between the display substrates ($d/2$). It should be noted that the form of functions $f_1(k)$ and $f_2(k)$ for a tubular column may change when applied to a flat capillary. In our case of separation during the filling of the display, the adsorption occurs on the substrate surface. Hence, the thickness of the stationary phase d_f is very low in comparison with the distance between substrates (cell gap) and the third term of equation (3) has no significant contribution. Summarizing, we can obtain a simplified Golay equation for HEEP in a flat capillary:

$$H=\frac{f_1(k)d^2}{4D_\mathrm{m}}u. \tag{4}$$

The mean velocity of a viscous incompressible fluid flowing steadily between two parallel surfaces under laminar conditions is determined by [8]:

$$V=\frac{\Delta P d^2}{12\eta L} \tag{5}$$

where ΔP is the pressure difference causing the flow of the fluid, η is the dynamic viscosity of the fluid and L is the length of the display. Assuming a steady flow over a small length dL during the filling of the display we can find the time required for filling a display with a length L:

$$t=\int\limits_{L=0}^{L}\frac{1}{V}\mathrm{d}L=\int\limits_{L=0}^{L}\frac{12\eta L}{\Delta P d^2}\mathrm{d}L=\frac{6\eta L^2}{\Delta P d^2}. \tag{6}$$

Thus, the average velocity of the fluid during the filling of a flat capillary will be:

$$u=\frac{L}{t}=\frac{\Delta P d^2}{6\eta L}. \tag{7}$$

It should be stressed that during the filling of the display the velocity of the mobile phase decreases hyperbolically with the filled length, and the separation efficiency increases accordingly. By introduction of an average velocity we obtain an average separation efficiency ($\bar{N}$) over the whole length of the display. Combining equations (2), (4) and (7) we obtain the dependence of the average separation efficiency during the filling of the display on the conditions of filling, and the dimensions and material parameters of the display cell:

$$\bar{N}=\frac{24}{f_1(k)}\cdot\frac{D_\mathrm{m}\eta L^2}{\Delta P d^4}. \tag{8}$$

This final equation confirms our observation that the separation during filling is more significant for large area displays, i.e. displays with a long filling length L, as the increase of the filling length promotes the separation efficiency.

The separation efficiency will be effectively reduced with an increased cell gap d due to two factors. On increasing the gap between the substrates the average distance over which the adsorbing substance has to diffuse from the bulk of the mobile phase to reach the surface of adsorbent, increases. This decreases the separation efficiency, equations (4) and (2). Furthermore, increasing the gap increases the velocity of the mobile phase, equation (7), which also leads to a reduction in the separation. The negative consequence of increasing the cell gap will be an increase in the threshold voltage both for the initiation of the EHD instability and for dielectric reorientation, but the light scattering efficiency of the FC structure improves, meaning an increase of the display brightness in the reflective mode.

A higher filling pressure will also help to make the separation less pronounced, as it increases the velocity of the mobile phase. Increasing the filling pressure and the cell gap also have the positive technological effect of reducing the time required for filling the display.

The influence of temperature on the separation efficiency is complex. Increasing the temperature leads to a decrease in the LC matrix viscosity. The temperature dependence of viscosity is quite strong for LCs near the mesophase transition temperatures. As shown already, this makes the separation less pronounced, equation (8), as the mobile phase velocity increases, equation (7). On the other hand, the diffusion coefficient of an adsorbing substance in the mobile phase D_m, which determines the time required for a substance to reach the surface of the adsorbent, is inversely proportional to viscosity, as shown by the Stokes–Einstein equation for isolated Brownian spheres:

$$D_m = \frac{k_B T}{6\pi\eta r_s}. \qquad (9)$$

Here, k_B is Boltzmann's constant, T is the absolute temperature and r_s is the radius of spheres. The decrease of the viscosity with increased temperature will reduce the diffusion coefficient and increase the separation. These two factors balance each other, resulting in a weak temperature dependence of the separation efficiency near mesophase transition temperatures.

At the same time, the temperature influences the position of the adsorption equilibrium, described by a distribution constant K, and thus the capacity factor k:

$$K = A\exp(-\mathrm{d}H/RT) \qquad (10)$$

where $K = C_s/C_m$ is a distribution constant, C_s and C_m are the concentrations of the adsorbing substance in the stationary and the mobile phase, respectively, A is the preexponential factor, dH is the enthalpy of sorption and R is the absolute gas constant. The direction of the shift in the adsorption equilibrium depends on the sign of the sorption enthalpy, which in turn is determined by the nature of the adsorbing substance, and of the mobile and stationary phases.

Taking into account all these factors, we completely eliminated all visual and electro-optical consequences of LC mixture separation during the filling of the display. Displays of diagonal 25.4 cm, without any additional coating on the surface of the ITO and with the gap between the substrates increased by 50%, were successfully filled using a 20% increased back pressure. In addition, a further 30% increase of the cell gap and a decrease of the gap between adjacent ITO electrodes allowed us to increase the size of the display several times.

5. Conclusion

Conductivity measurements on filled display cells have confirmed our hypothesis that the problem observed during the filling of large area display cells is caused by the separation of the LC mixture. Such separation is caused by selective adsorption of the ionic dopant on the surface of the display substrates and leads to a non-uniform distribution of the ionic dopant concentration in the volume of the LC matrix over the display area. It has been shown that this process may be considered to be chromatographic in nature. By applying the general theory of chromatography we have shown that increasing the cell gap and decreasing the area of free glass by a reduction in the gap between electrodes will effectively reduce the significance of the separation process. Likewise, increasing the back pressure while filling has been shown to reduce the effect of the separation process and to result in more uniform LC cells. Effectively, the observed problem has been eliminated by addressing the factors revealed theoretically.

The factors discussed and the results obtained should be taken into account when designing and filling displays based on the electrically reversible memory effect in a smectic A LC or displays based on dynamic light scattering in a nematic LC, in which an ionic admixture has to be used. In addition, these observations can be applied to any case of display cell filling, when the contribution of the chromatographic effect is significant.

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