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# Evidence for the flexo-electric effect in a nematic liquid crystal cell

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# Evidence for the flexo-electric effect in a nematic liquid crystal cell

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We present evidence for the flexo-electric effect in a hybrid aligned nematic cell containing 4-methoxybenzylidene-4′-*n*-butylaniline (MBBA) subject to d.c. excitation, by comparing the theoretical curve derived by including the flexo-electric term in the free energy with the experimental points. The agreement appears to be very good for MBBA doped with a small amount of an additive which shows reversible electrochemical reactions at very low redox potential; this allows us to determine the sum of the flexo-electric coefficients and the anchoring energy on the planar wall. For undoped MBBA the experimental results can be justified only by assuming a low voltage screening of the electric field, due to the presence of ionic impurities in the material.

## 1. Introduction

In a previous paper [1] we reported electro-optical properties of hybrid aligned nematic (HAN) cells [2–5]. As observed by Matsumoto *et al.* [2], under a.c. fields, the HAN cell does not exhibit a threshold voltage for electro-optical effects, unlike planar or homeotropic configurations. In [1] we verified this response, showing that the main contribution to the molecular reorientation results from the dielectric term in the elastic free energy. In the same study we noticed that a different behaviour occurs in the d.c. case at low fields; qualitatively we ascribed this effect to a flexo-electric contribution.

More recently Madhusudana and Durand [6], measuring the electrically-controlled birefringence of a similar cell, found a linear response for positive and negative voltages of magnitude greater than $|1{\cdot}5|$ V, which they attributed to flexo-electricity. To justify the observed non-linearity between $-1{\cdot}5$ and $+1{\cdot}5$ V these authors assumed low voltage screening by counter-ions, followed by the appearance of electrochemical effects near the redox potential of the liquid crystal.

In this paper we report more accurate and systematic measurements on the d.c. electro-optical behaviour of a thin HAN cell containing 4-methoxybenzylidene-4′-*n*-butylaniline (MBBA). Moreover we present data obtained with MBBA doped with

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a small amount of ferrocene, an electrochemical standard material, in order to clarify the competition between flexo-electricity and electrochemical processes. Finally we try to fit our results with a theoretical curve obtained by including the flexo-electric term into the free energy.

## 2. Experimental

HAN cells incorporating nematic MBBA are obtained by coating a conductive glass surface with ODS-E from Chisso Co. (homeotropic alignment) and the other one with a polymeric thin film of formvar (homogeneous alignment), as described elsewhere [1]. The experimental measurements reported in this paper refer to 6 $\mu$m thick cells investigated in the arrangement described previously [1]; the curves correspond to the automatic registration of the light intensity transmission as a function of the applied voltage.

To provide a better explanation of the observed electro-optical effects we carried out experiments under different experimental conditions, trying to isolate contributions arising from flexo-electricity, impurities and electrochemical behaviour. Flexoelectricity has been detected by observing the difference between d.c. and high frequency (500 Hz) a.c. behaviour and by studying the effect of field polarity reversal for d.c. fields. To check the role of impurities in MBBA we filled the cells with differently aged materials (having nematic–isotropic transition temperatures ranging between 35 and 41·5°C). To prevent electrochemical degradation of the liquid crystal, and mainly to avoid complete screening by counter-ions, some experiments were performed by doping MBBA with a small quantity ($2 \times 10^{-3}\,M^{-1}$) of ferrocene (bicyclopentadienyl iron). This additive shows reversible electrochemical behaviour occurring with fast kinetics at a very low value of the applied field (half-wave potential of 0·08 V referred to the reference electrode $Ag/Ag^+$, $10^{-2}\,M^{-1}$ in acetonitrile [7, 8]). Cyclic voltammograms of MBBA (reduction waves at $-2{\cdot}06$ and $-2{\cdot}6$ V, oxidation at $+1{\cdot}45$ and 2·0 V versus saturated calomel) are found in the literature [9].

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As a typical example we report in figure 1 the behaviour obtained in a d.c. field with undoped MBBA. The general feature, as observed for many samples, can be summarized as follows. The HAN cell is much less sensitive to small fields than under a.c. excitation; near zero voltage the intensity of the transmitted light remains practically constant or shows a small field-dependence (i.e. a plateau), whose width and shape is asymmetric as regards polarity. The behaviour at slightly higher voltages is polarity-dependent. At higher voltages the behaviour is qualitatively the same in d.c. or a.c. fields, showing successive minima and maxima in the transmitted light; however these oscillations are shifted towards higher voltages under d.c. excitation.

No appreciable effect arises from the investigation of MBBA samples with different nematic–isotropic transition temperatures, that is with different concentrations of impurities.

MBBA doped with ferrocene behaves under an a.c. field in the same way as the undoped material. The curves obtained for d.c. fields are shown in figure 2. The main result is the disappearance of the plateau near zero voltage; we find an almost linear dependence of the transmission against voltage between $-1{\cdot}5$ and $+1{\cdot}5$ V. At higher fields the curves are similar to those reported for undoped MBBA. A typical measurement, such as those reported in figures 1 and 2, requires approximately 2 hours. The applied voltage changes by steps of 0·1 V every 2 min. An analysis of the dynamical processes that take place when a voltage is applied, and their characteristic times $\tau$,

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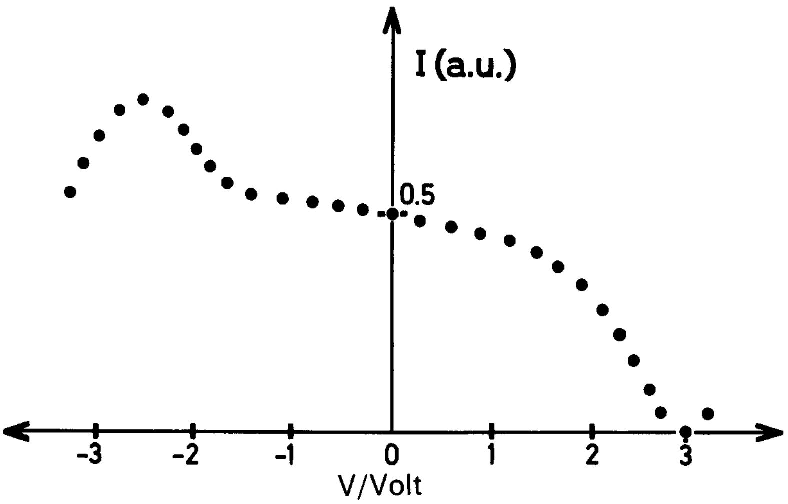

Figure 1. Intensity of the transmitted light for a HAN cell with undoped MBBA.

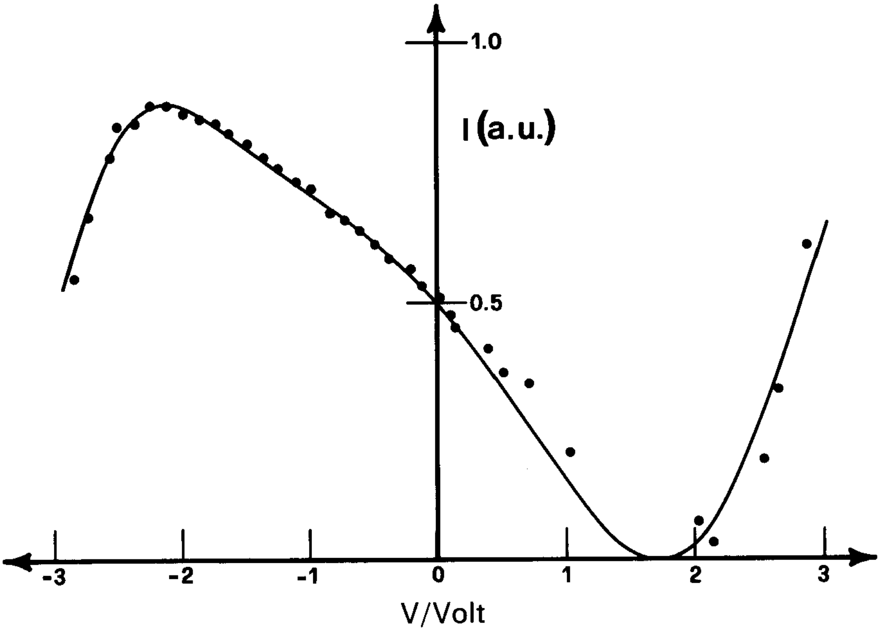

Figure 2. Comparison between the calculated curve and experimental points for doped MBBA.

is reported in [10]. These processes are: (1) field induced distortion (rot), (2) elastic relaxation (elast), (3) ion transport under a field (tr), (4) thermal diffusion (diff), (5) dielectric response time (diel). The connected characteristic times, for a cell of thickness $d \simeq 6\,\mu\mathrm{m}$, are of the order of magnitude [10] $\tau_{\mathrm{rot}} \simeq 10\,\mathrm{ms}$, $\tau_{\mathrm{elast}} \simeq 100\,\mathrm{ms}$, $\tau_{\mathrm{tr}} \simeq 50\,\mathrm{ms}$, $\tau_{\mathrm{diff}} \simeq 5\,\mathrm{s}$, $\tau_{\mathrm{diel}} \simeq 5\,\mathrm{ms}$, for a typical liquid crystal. Since $\tau_i \ll t_m$, where $i$ = rot, elast . . . and $t_m \simeq 2\,\mathrm{min}$, we can suppose that the measurements are made under equilibrium conditions.

## 3. Theoretical analysis

Let us consider a HAN cell of thickness $d$ and suppose that: (i) the single constant approximation is adequate ($K_{11} = K_{33} = K$ [11]); (ii) the anchoring energy on the homeotropic wall is infinitely large due to the silane treatment [12]; (iii) the anchoring energy on the planar wall is small (formvar treatment). Since, in our sample, the width is very much greater than the thickness, and the surface treatment is uniform, every physical quantity is a function of the $z$ coordinate only (the $z$ axis is normal to the boundary walls). Consequently the equation

$$\mathrm{rot}\,\mathbf{E} = 0$$

gives

$$\mathbf{E} = E_3\mathbf{K}.$$

Furthermore the continuity equation for the electric current density, in the steady state, reads

$$\mathrm{div}\,\mathbf{j} = 0,$$

and hence $j_3$ is constant. By Ohm's law we obtain

$$j_1 = \sigma_{13}(\phi)\,E_3$$

and

$$j_3 = \sigma_{33}(\phi)\,E_3,$$

where the $\sigma_{ij}(\phi)$ are the components of the conductivity tensor and $\phi$ is the angle between the director $\mathbf{n}$ and the $z$ axis. It follows that

$$E_3 = j_3/\sigma_{33}(\phi) \qquad (1)$$

and hence

$$V = \int_0^d E_3\,dz = j_3\int_0^d dz/\sigma_{33}(\phi), \qquad (2)$$

where $V$ is the applied voltage. From equations (1) and (2) we obtain

$$E_3 = \frac{V}{\sigma_{33}(\phi)}\Bigg/\int_0^d dz/\sigma_{33}(\phi). \qquad (3)$$

Equation (3) shows that the electric field is non-local [13], whereas equation (2) shows that $j_3$ is a functional of $\phi$. In our case $\mathbf{j}$ is not parallel to the $z$ axis, and it is found to be

$$\mathbf{j} = [(\sigma_{13}(\phi)/\sigma_{33}(\phi))\,\mathbf{i} + \mathbf{K}]\,j_3, \qquad (4)$$

but $j_1$ does not enter the problem, given the hypothesis that it is unidimensional.

The free energy $F_{\mathrm{flexo}}$ related to the flexo-electric polarization is

$$F_{\mathrm{flexo}} = -\int_0^d \mathbf{P}_{\mathrm{f}}\mathbf{E}\,dz, \qquad (5)$$

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where

$$\mathbf{P}_f = e_{11}\mathbf{n}\,\mathrm{div}\,\mathbf{n} - e_{33}\mathbf{n} \times \mathrm{rot}\,\mathbf{n};$$

$e_{11}$ and $e_{33}$ are the flexo-electric coefficients. Taking into account that

$$\sigma_{33}(\phi) = \sigma_{\parallel}\cos^2\phi + \sigma_{\perp}\sin^2\phi$$

and

$$\sigma_{13}(\phi) = (\sigma_{\parallel} - \sigma_{\perp})\sin\phi\cos\phi,$$

where $\sigma_{\parallel}$ and $\sigma_{\perp}$ are the conductivities of the nematic along **n** and normal to it, and by substituting equation (1) in equation (5) we obtain

$$F_{\text{flexo}} = -\frac{e_{11} + e_{33}}{\sigma_{\parallel} - \sigma_{\perp}} j_3 \ln(1 - \sigma\sin^2\phi_2), \qquad (6)$$

where $\sigma = 1 - (\sigma_{\perp}/\sigma_{\parallel})$ is the relative anisotropic conductivity, $\phi_2 = \phi(d)$ and we have supposed that $\phi_1 = \phi(0) = 0$ (strong anchoring hypothesis on the homeotropic wall). Equation (6) tells us that the total free energy due to the flexo-electric effect is, actually, a surface contribution. In the hypothesis of small $\sigma$ equation (6) becomes

$$F_{\text{flexo}} = \frac{1}{2}\frac{e_{11} + e_{33}}{\sigma_{\parallel}} j_3 \sin^2\phi_2. \qquad (7)$$

Let us suppose that the surface free energy due to the nematic–substrate interaction is of the kind [14]

$$F_S = (1/2)\,W\cos^2\phi_2, \qquad (8)$$

where $W$ is the so-called anchoring energy and the easy axis is parallel to the $x$ axis (planar alignment). In this case the total surface free energy is, from equation (7),

$$\begin{aligned} f_S &= F_S + F_{\text{flexo}} \\ &= \frac{1}{2}W - \frac{1}{2}\left(W - \frac{e_{11} + e_{33}}{\sigma_{\parallel}} j_3\right)\sin^2\phi_2. \end{aligned} \qquad (9)$$

Equation (9) shows that the flexo-electric effect changes the anchoring energy. In the case of $e_{11} + e_{33} < 0$ equation (9) for $j_3 > 0$ ($\Rightarrow V > 0$) gives an effective anchoring energy

$$W^* = W - j_3(e_{11} + e_{33})/\sigma > W,$$

i.e. the anchoring energy is greater than that for $V = 0$, and hence $\phi_2 > \phi_2(V = 0)$. In the opposite case ($V < 0$) we have $W^* < W$ and hence $\phi_2 < \phi_2(V = 0)$. In order to solve the problem it is necessary to derive the surface torque

$$\tau_S = \partial f_S/\partial\phi_2.$$

From equation (9) we deduce

$$\tau_S = -\tfrac{1}{2}\{W - j_3(e_{11} + e_{33})/\sigma_{\parallel}\}\sin 2\phi_2 + \frac{1}{2}\frac{e_{11} + e_{33}}{\sigma_{\parallel}}\frac{\partial j_3}{\partial\phi_2}\sin^2\phi_2. \qquad (10)$$

For small $\sigma$ equation (2) gives

$$j_3 = \frac{\sigma_{\parallel}V}{d}\{1 - \sigma\langle\sin^2\phi\rangle\} \qquad (11)$$

and hence

$$\partial j_3/\partial\phi_2 = -(\sigma\sigma_\parallel V/d)\frac{\partial}{\partial\phi_2}\langle\sin^2\phi\rangle,$$
$$= O(\phi_2).$$

Consequently the last term in equation (10) is of the order of $O(\phi_2^3)$, whereas the other one is of the order of $O(\phi_2)$. It follows that for small $\phi_2$ we can consider $j_3$ to be constant with respect to $\phi_2$ [13]. Of course this approximation is better for $V < 0$, since in this case the anchoring energy is lower than that for $V = 0$, for small voltages. In the following we suppose $j_3$ to be independent of $\phi$, and hence we assume as the surface torque, for any $\sigma$,

$$\tau_S = j_3\frac{e_{11} + e_{33}}{2\sigma}\frac{\sin 2\phi_2}{1 - \sigma\sin^2\phi_2} - \frac{W}{2}\sin 2\phi_2. \tag{12}$$

From the previous discussion our problem is now equivalent to that of studying the orientation induced by an electric field in a conducting nematic, with surface anchoring energy given by equations (6) and (7). With these hypotheses $\phi(z)$ is determined by

$$K d^2\phi/dz^2 - (\varepsilon_a E_3^2/4\pi)\sin\phi\cos\phi = 0, \tag{13}$$

where $\varepsilon_a$ ($\equiv \varepsilon_\parallel - \varepsilon_\perp$) is the dielectric anisotropy. The first term in equation (13) is the elastic torque and the last one is the dielectric torque. In equation (13) $E_3$ is a function (non-local) of the deformation $\phi$ (cf. equation (3)). In an insulator the electric field **E** is obtained by the condition div **D** = 0, where **D** is the dielectric displacement. Of course a nematic material is very far from being a perfect insulator [15]. Then, as we have already pointed out, the field into the sample is determined by the conduction anisotropy [16]. In this case the back electric field connected with the flexo-electric polarization is suppressed by the free charge [17] and, in the hypothesis of small $\phi$, the flexo-electric effect gives only a surface contribution [18].

By substituting equation (1) into equation (13), after standard calculations, we find

$$\left(\frac{d\phi}{dz}\right)^2 - \frac{\varepsilon_a j_3^2}{4\pi K\sigma\sigma_\parallel^2}\frac{1}{1 - \sigma\sin^2\phi} = B^2, \tag{14}$$

where $B$ is a constant of integration. The boundary condition is

$$K\frac{d\phi}{dz} + \frac{j_3(e_{11} + e_{33})}{2\sigma_\parallel}\frac{\sin 2\phi_2}{1 - \sigma\sin^2\phi_2} - \frac{W}{2}\sin 2\phi_2 = 0 \tag{15}$$

derived by equating the volume torque to the surface torque. Equations (14) and (15) lead to

$$B = \frac{1}{d}\int_0^{\Phi_2}\left(\frac{1 - \sigma\sin^2\phi}{1 - \mu^2 - \sigma\sin^2\phi}\right)^{1/2} d\phi, \tag{16}$$

$$B = \frac{(W/2K)\sin 2\phi_2}{\sqrt{[(1 - \mu^2 - \sigma\sin^2\phi_2)/(1 - \sigma\sin^2\phi_2)] + (\zeta/2K\sigma_\parallel)(e_{11} + e_{33})\mu\sin 2\phi_2/(1 - \sigma\sin^2\phi_2)}} \tag{17}$$

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where $\zeta = 2\pi\sqrt{(\pi K\sigma/-\varepsilon_a)}$ and $\mu = j_3/B\zeta$. From equations (16) and (17) we can deduce $\mu = \mu(\phi_2)$ and then $B = B(\phi_2)$, $j_3 = j_3(\phi_2)$. Consequently, from equation (2), we find

$$V = \frac{\zeta}{\sigma_\parallel}\mu\int_0^{\phi_2}\frac{d\phi}{\sqrt{[(1-\sigma\sin^2\phi)(1-\mu^2-\sigma\sin^2\phi)]}}, \tag{18}$$

which gives $\phi_2 = \phi_2(V)$.

We determine the tilt angle distribution $\Phi(z)$ by measuring the transmitted light intensity as a function of the applied voltage; the aim is to obtain information on the anchoring energy due to the formvar treatment and on the flexo-electric coefficients. In our experimental arrangement the optical path difference between the ordinary and the extraordinary ray is given by

$$\Delta l = n_o\left\{\int_0^d (1 - R\sin^2\phi)^{-1/2}dz - d\right\} \tag{19}$$

with $R = 1 - (n_o/n_e)^2$. The intensity of the transmitted light can be written in the form [1]

$$I = I_o\sin^2\left(\frac{\pi}{\lambda}\Delta l\right). \tag{20}$$

Figure 2 shows a comparison between experimental points of the transmitted intensity and the theoretical curve computed with $\varepsilon_a = -0{\cdot}7$ [19], $\sigma_\parallel = 6 \times 10^{-11}(\Omega\,\mathrm{cm})^{-1}$ [20], $\sigma_\perp \simeq 4 \times 10^{-11}(\Omega\,\mathrm{cm})^{-1}$ [20], $\sigma = 0{\cdot}32$, $\lambda = 0{\cdot}6\,\mu$m, $K = 7 \times 10^{-7}$dynes [11], $R = 0{\cdot}207$ [11], $n_o = 1{\cdot}57$ [11]. The agreement is very good in a relatively large range of the applied voltage. From the best fit we can derive a value for $e_{11} + e_{33}$ of $-4{\cdot}5 \times 10^{-4}(\mathrm{dynes})^{1/2}$, consistent with the measurements of Madhusudana and Durand [6], and for $W$ a value of $5{\cdot}8 \times 10^{-3}\,\mathrm{erg/cm^2}$.

We point out that the agreement is better for $V < 0$ than for $V > 0$. This is connected with the approximation that $j_3$ is a constant—which implies small $\phi$ values (and hence a small effective anchoring energy). The same fit does not work on the experimental points reported in figure 1, since the screening effect by counter-ions is present, and hence the electric field inside the sample is very different from that applied [10]. An analysis of the curve of $I$ versus $V$ for liquid crystals without dopant is very difficult and requires a detailed model for the screening effect; this will be attempted in the future.

## 4. Conclusions

We have studied the flexo-electric properties of the HAN cell from both an experimental and a theoretical point of view. The measurements carried out on commercial MBBA show a transmitted intensity which is approximately constant for small applied voltages. The same measurements performed on MBBA doped with ferrocene, show an enhanced linear behaviour of the light intensity versus the applied voltage, when it is smaller than about |2|V. We interpret these last experimental results as due to the flexo-electric effect. The best fit obtained with the assumption that the electric field into the sample is due to the effective conduction anisotropy, gives for $e_{11} + e_{33}$ a value of $-4{\cdot}5 \times 10^{-4}(\mathrm{dynes})^{1/2}$ and for the anchoring energy, $W$, $5{\cdot}8 \times 10^{-3}\,\mathrm{erg/cm^2}$, both consistent with other researchers' results. In the former case, i.e. without dopant, the experimental data are interpretable only by supposing a screening effect by counter-ions, present in MBBA. While at low fields this screening can be almost complete, at higher voltages the MBBA molecules play an important role in the conduction phenomena and degradation is produced.

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## Appendix

In equation (9) we have neglected the energy connected with the surface polarization [21, 22]. As is well known, near a boundary surface a nematic polarization $\mathbf{P}_S$ can exist. $\mathbf{P}_S$ then gives a further surface term of the type $\mathbf{P}_S \cdot \mathbf{E}(d)$; molecular models [23] give, for $\mathbf{P}_S$, the expression

$$\mathbf{P}_S = \tfrac{1}{2}\alpha \cos\phi\, \mathbf{n} + \beta(\mathbf{n} \times \mathbf{K}) \times \mathbf{n},$$

where $\alpha$ and $\beta$ are $z$ dependent. It follows that the surface energy connected to $\mathbf{P}_S$ is

$$\begin{aligned} F_P &= -\tfrac{1}{2}\alpha \cos^2\phi E_3, \\ &\simeq -\tfrac{1}{2}\alpha \frac{j_3}{\sigma_{33}(\phi_2)} \cos^2\phi_2, \\ &\simeq -\tfrac{1}{2}\alpha \frac{j_3}{\sigma_\parallel} \cos^2\phi_2 \end{aligned}$$

for small $\phi_2$. Equation (9) should then be rewritten as

$$\tilde{f}_S = F_S + F_{\text{flexo}} + F_P = \tfrac{1}{2}W - \tfrac{1}{2}\left\{W - \left[\frac{e_{11} + e_{33} + \alpha}{\sigma_\parallel}\right] j_3\right\} \sin^2\phi_2.$$

Hence, as pointed out by Petrov *et al.* [21], the effective flexo-electric coefficient is $e_{11} + e_{33} + \alpha$. Our measurements give this effective coefficient.

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