Factors influencing flexoelectric polarization in liquid crystals

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Abstract. The work experimentally investigated the factors affecting the formation and physical parameters of flexoelectric polarization in liquid crystals (LC) with nematic as well as smectic A- and C-phases. Factors such as the heating temperature of the sample, orientation effects near the nematic – isotropic state (N-I) phase transition, and the initial orientation of LC molecules were studied. The thickness of the studied layers is from 10 to 100 μm, the frequency of exposure is 1000 Hz. It was found that the flexosignal takes maximum value in homeotropically oriented samples in the nematic phase. When approaching the N-I phase transition, the magnitude of the flex signal increases, which is associated with an increase in the deviation angle of the director of the LC. As the dipole moment grows, the quadrupole moment also increases. This is due to the tendency of LC molecules to form dipole pairs with antiparallel orientation of electric moments. The estimate of the flexural value $U_{1\omega}$ yielded (for example, for MBBA) with a planar orientation 3 times less than with a homeotropic one. The presence of the second harmonic $U_{2\omega}$ is a consequence of the orientational instability of surface polarization. The rapid growth of $U_{2\omega}$ is associated with an increase in the square of the director deviation angle $\theta_d^2$.

1. Introduction

The experimental study of the flexoelectric effect in liquid crystals (LC) is difficult to overestimate. Interest in his research is associated with the constant search for new effects and developments for the creation of touch devices, as well as displays of a new generation. The flexoelectric effect is observed at various kinds of deformations (shift or bending) of the surface layer of a liquid crystal both in the nematic phase and in the vicinity of the nematic - isotropic phase transition. When a flexoelectric effect occurs, polarization appears on the surface of the liquid crystal layer, as a result of which a number of harmonics of both the emf and the optical signal that passes through the object of study are generated.

The nature of the harmonics of the emf generated by the LC layer under external influence was investigated in [1–7]. They show that two mechanisms of molecular orientational flexoelectric polarization of LCs are known today: dipole and quadrupole. Based on empirical data, most authors are inclined to conclude that the main of the two mechanisms is quadrupole. The last conclusion can be made using the analysis of the dependences of the piezoelectric temperature signal. However, an unambiguous opinion on this issue has not yet been drawn up, as a result of which our immediate task will be to clarify the role played by both the dipole and quadrupole mechanisms in the induction of flexoelectric polarization of the LC, as well as the study of the behaviour of surface
polarization as a function of temperature. We now turn to the study of the behaviour of the spectral components of the piezoelectric response from temperature.

2. Objects of research and experimental setup
To solve this problem, liquid crystals were studied (Table 1) with both negative and positive dielectric anisotropy, the molecules of which have a different geometric shape (for example, drop-shaped - cyanobiphenyls, drop-like - MBBA, HBACE), as well as different dipole moments. In addition, substances with both a purely nematic phase and smectic A- and C-modifications were investigated.

Table 1. Objects of research.

| Liquid crystals | Formula |
|-----------------|---------|
| MBBA            | n-methoxybenzylidene-n-butylaniline |
| HBACE           | heptylbenzoic-acid-cyanophenyl-ester |
| NFOOB           | hitrophenyloctyl-oxybenzoate |
| OCB             | 4-octyl-4-cyanobiphenyl |
| WHAOB           | will-heptanilase-hydroxybenzene |

To study liquid crystals with a thickness \( h=10-100 \) μm, we used the setup shown in Figure 1a. A spectrophotometric prefix recorded the variable component of the light signal intensity, and a direct current millivoltmeter measured its constant component. An LC sample was placed on a microscope stage. Under it was a source of bending vibrations. The membrane of the oscillation source and the experimental cell were connected by a waveguide through which oscillations were transmitted. The capacitor cell was collected from glass plates (Figure 1b), separated by gaskets, and a liquid crystal was placed in the free space.

![Figure 1](image1.png)

**Figure 1.** Installation for studying the flexoelectric effect in an LC: a) 1 - a source of bending vibrations; 2 - thermostat with an LC cell; 3 - polarizing microscope with a spectrophotometric nozzle; 4 - differential thermocouple; 5 and 6 - voltmeters (direct current); 7 - selective amplifier; 8 - analog-to-digital converter; 9 - computer; b) 10 - liquid crystal; 11 and 12 - glass plates; 13 - gaskets that specify the thickness of the LC; 14 - waveguide; 15 is a coating that reflects light.
3. Experimental results and discussion

3.1. Flexoelectric effect in the vicinity of the nematic - isotropic state phase transition

Let us consider the temperature dependencies of the flexoresponse $U_{1\omega}$ recorded at an excitation frequency of $\omega=1000$ Hz (Figure 2) ($1000$ Hz is the most convenient frequency for investigation because of the minimal influence of impurities $\sigma$ on the magnitude and behavior of $U_{1\omega}$).

First, we compare the magnitudes of the observed signals in various liquid crystal modifications (the initial orientation is homeotropic). Flexoresponse reaches its highest value at the excitation frequency in the nematic phase. This is due, as noted in [8], to a greater sensitivity of nematic ordering to external perturbations, since NLCs have less “rigidity” in comparison with a layered structure, for example, smectic $A$, and therefore, for the same deformations in the bulk of the smectic phase, the induced perturbations are weaker. The latter does not apply to the pre-transition phenomena observed in the vicinity of solid phase - smectic, smectic - nematic phase transitions, where a number of material parameters of the medium: elastic moduli, orientational viscosities behave anomalously, due to an increase in the fluctuation $S$-mode.

According to the data of [9], in flexure $A$, flexocoefficients $e_{ij}$ ($S$ is the deformation) can significantly exceed the flexocoefficients in nematics, namely, in the smectic NFOOB, but, for example, in smectics $C$ in the OCB, as experience shows (Figure 2b), the signal $U_{1\omega}$ $10^2$ – $10^3$ times less than in the nematic phase, which indicates weak flexoelectric properties or their absence.

Next, we discuss the temperature behavior of flexosignals $U_{1\omega}$. Far from the nematic – isotropic phase transition, the value of the signal $U_{1\omega}$ changes slightly, which coincides with the results of studies [10]. However, in the vicinity of the nematic – isotropic $N$–$I$ phase transition, the values of $U_{1\omega}$ do not change, gradually decreasing to zero, as the theory predicts [11], but are characterized by spasmodic behavior (Figure 2a). Therefore, a special place in the study of the temperature dependencies of the induced polarization $P^\omega$ is the elucidation of the behavior of $U_{1\omega}$ in the vicinity of the nematic – isotropic liquid transition, since only this will solve the problem of contributions to the flexopolization of the dipole and quadruple mechanisms.

3.2. Orientational effects near the nematic - isotropic state phase transition

It is known that the order parameter $S$ undergoes a jump in the $N$–$I$ phase transition [12]. The elastic module, the orientational viscosity [13], and the anisotropy of the magnetic and dielectric properties [14] behave in a similar way. Characteristic parameters of the anisotropic medium, which decrease their values when approaching the temperature $T_{NI}$. In this case, the flexocoefficients $e_{ij}$ must also
have corresponding dependencies; in general, they are functions of $S$ and $S^3$ [15], by some combination of the form $e_{ij} - \alpha S_i + \beta S_2$. However, such a functional dependence does not in any way describe the observed behavior of the $U_{1,\omega}$ signal in the crystals under study. To solve this problem, we turn to the expression

$$e_{obs} = \frac{\varepsilon \omega \tau}{1 + \omega^2 \tau^2},$$

(1)

where $e_{obs}$ is the recorded potential difference, $\varepsilon$ is the EMF arising from the disturbance of molecular LC ordering, and $\tau$ is the relaxation time.

It follows from (1) that the first harmonic is proportional to the flexocoefficient, the sample thickness, and the average director deviation angle $\bar{\theta}_d$ in the LC layer, i.e. $U_{1,\omega} \approx \varepsilon_1 h \bar{\theta}_d$. As Gaviller [16] showed, in the region of the phase transition, the average director deviation angle abnormally increases in the orienting magnetic field, which is induced by bias. This means that the observed temperature dependence of the recorded signal can be associated with a change in the director deviation during heating. In this connection, the dependence of the magnitude of the acoustooptical effect — the magnitude of the light flux recorded at the doubled excitation frequency $I_{2,\omega}$ on the temperature, which is expressed in terms of the crystal parameters and the deviation angle $\bar{\theta}_d$ by the formula:

$$\frac{I_{2,\omega}}{I_o} = \frac{1}{4} \bar{\theta}_d^2 \beta h \sin(\bar{\theta}_d^2 \beta h),$$

(2)

where $\beta = 2\pi \Delta n/\lambda$. It follows from expression (2) that the value of $I_{2,\omega}$ should oscillate depending on the temperature, which is confirmed experimentally (Figure 3a), because the sine argument includes temperature-dependent optical anisotropy and $\bar{\theta}_d \neq 0$.

![Figure 3](image)

**Figure 3.** Temperature dependencies of the relative light intensity $I_{2,\omega}/I_o$ reflected from a cell with an LC modulated with a frequency of $2\omega$: a) strain amplitude $a > a_{cr}$; b) the amplitude of the perturbation $a = a_{cr}$. 

In addition, in the vicinity of the phase transition, the value of $I_{2,\omega}$ sharply increases and then drops to zero (Figure 3b), which cannot be explained by a regular change in the birefringence parameter $\Delta n$, which is a consequence of an increase in the angles $\bar{\theta}_c$ and $\bar{\theta}_d$. To exclude the influence on the measurement results of the acoustooptical effect of the stationary distribution of the director in the volume, we studied the dependencies at small strain amplitudes $a$, when $\bar{\theta}_c = 0$. In this case, the functional dependence $I_{2,\omega}/I_o$ will have the form:
Hence, taking into account that, at small amplitudes \( a \sim (\bar{\vartheta}_d^2)^{1/2} \), and, knowing the behavior of \( \Delta n(T) \) for the crystals under study [12–14], we calculate, according to formula (3), the dependencies \( \bar{\vartheta}_d \) and \( \vartheta_d \) on temperature as well (Figure 4).

\[
\frac{I_{2\vartheta}}{I_\vartheta} \sim \frac{1}{8} (\beta \vartheta)^2 (\bar{\vartheta}_d^2)^2 .
\]  

(3)

Figure 4. Temperature dependencies: a) squared director deviation angle \( \bar{\vartheta}_d^2 \); b) its root \( \vartheta_d \sim (\bar{\vartheta}_d^2)^{1/2} \) in the investigated LC (\( a \geq a_0 \)).

It can be seen from Figure 4 that in the vicinity of the phase transition, the magnitude of the director deviation angle and the square of the angle increase abnormally. For example, at \( \Delta T \approx T_{NI} - 0.5 \) °C (MBBA), the angle \( \bar{\vartheta}_d \) is 4–5 times larger than at \( \Delta T = 20 \) °C, and \( \bar{\vartheta}_d^2 \) 20 times. For NLCs with \( \varepsilon_\alpha > 0 \), for example, OCB and HBACE, the changes are slightly less, \( \bar{\vartheta}_d \) 2.5–3 times, \( \bar{\vartheta}_d^2 \) 10 times.

From the results obtained, the behavior of flexosignals in the studied LC systems becomes clear. Far from the phase transition, when \( \bar{\vartheta}_d \) changes slightly, the value gradually decreases. When approaching the phase transition, it increases, which is associated with an increase in the amplitude of the director’s oscillations \( \vartheta_d \), and the final value of the recorded signal is due to a jump in the magnitudes of the flexocoefficients \( e_{ij} \), because they are functions of the order parameter \( S \).

Now, based on the foregoing, it is possible to obtain the temperature dependencies of flexoelectric coefficients \( e_{11} \). In the general case, the temperature behavior of \( e_{11} \) is fairly well described by the functional dependence of \( \alpha S + \beta S^2 \) (Figure 5).

Figure 5. Temperature dependencies of flexoelectric coefficients \( e_{11} \) of the studied substances.

For example, for MBBA \( \alpha \approx 0.2, \beta \approx 0.8 \) (relative calculation error of 10%). This means that the contribution of the dipole mechanism of molecular orientation polarization is \( -0.8 \) of the total value, and the quadruple 0.2. For the remaining crystals, the coefficient values are given in Table 2.
A comparison of the $\alpha$ and $\beta$ values of the studied systems leads to the important conclusion that, in anisotropic media such as nematic LCs, as the absolute value of the dipole moment in the molecules increases, the “weight” of the quadrupole mechanism also increases. This indicates the tendency of molecules to form dipole pairs with antiparallel orientation of electric moments. The latter is especially characteristic for LCs with large positive dielectric anisotropy, in which the dipole moment is directed mainly along the long axis of the molecule. For example, the dipole moments of HBACE $p$~4.5 D; OCB $p$~5.0 D; NFOOB $p$~3.5 D, and the coefficient $\alpha$~1 is the same for all three crystals. The values of the coefficients $\sim$1 apparently explain the small difference in the values of flexomodules between MBBA ($p$~2.6 D) and the listed crystals, although their dipole moments differ significantly. A similar situation is due to the fact that the quadrupole polarization is due to the next term in the expansion of the potential $\phi(r)$ induced by inhomogeneous distortions, and the decrease in the magnitude of this term is compensated by the large dipole moment and the geometry of the mutual arrangement of the molecules that make up the quadrupole.

3.3. The initial orientation of the liquid crystal molecules

We now turn to studying the effect of the initial orientation of LC molecules on the magnitude and behavior of harmonics as a function of temperature. According to the expression

$$\mu=(\varepsilon_1 T_1 k M/4 \pi p N_A)^{1/2},$$

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$$U_h = e_{11} \frac{h}{R} \partial_d e^{i\omega t} - (e_{11} - e_{33}) \frac{h}{R} \partial_d e^{i2\omega t} \quad \text{and} \quad U_p = e_{33} \frac{h}{R} \partial_d e^{i\omega t} + (e_{11} - e_{33}) \frac{h}{R} \frac{\partial \bar{\partial}}{\partial r} e^{i2\omega t},$$

(where \(h\) – LC layer thickness, \(R\) – cell radius in the sample plane, \(\bar{\partial}_d\) - means averaging over the thickness of the layer), an estimate can be obtained for the crystals under study, for example, in MBBA with a planar orientation of the quantity \(U_{1\omega}\) about 3 times less than with homeotropic, which gives an estimate \(e_{11}-10^{-3}\) units CGSE/cm, and in NLC with \(\varepsilon_{\alpha}>0\), the value of \(e_{11}<<e_{11}\) and has a value of \(~10^{-5}\) units CGSE/cm.

It should be noted that, in the vicinity of the nematic –isotropic phase transition for MBBA, the value of the first harmonic oscillates and, increasing, reaches the values observed in the homeotropic orientation. Polarization-optical observations showed that the director angle \(\bar{\partial}_d\) decreases and the layer becomes quasi-homeotropic. Since the orientation of the molecules of the LC films depends on the boundary conditions and the state of liquid crystal ordering on the surface, in this case the issue of studying the surface properties and, in particular, the surface polarization of the LC is of great importance. Moreover, this issue has an independent interest, which stimulated further research.

It was established above that the second harmonic is a consequence of the orientational instability of surface polarization and the magnitude of the detected signal should depend on the initial orientation of the molecules on the surface. Let us consider the temperature dependencies of \(U_{2\omega}\) at different molecular orientations in the cell. An increase in the director’s angle of inclination relative to the normal leads in the NLC with \(\varepsilon_{\alpha}<0\), first to a small increase, and then to a sharp decrease in the absolute value of the signal (Figure 7a).

At an orientation angle \((\theta_c \sim 80^\circ)\), the signal appears only in the vicinity of the phase transition. The latter correlates with the behavior of the director angle in the cell, which tends to zero in the phase transition region, i.e. the orientation of the sample becomes quasi-homeotropic (Figure 7b), which
leads to the appearance of a second harmonic. In the case of positive anisotropy far from the phase transition at small initial angles \((\theta_0 \leq 10^\circ)\), the angle of inclination tends to zero.

To clarify the causes of the second harmonic and its relation to the orientation of the sample molecules, we consider the following model. Suppose that on the surface of the LC there is a polarized layer of molecules that creates some effective surface electric field \(E_s = \mu_0 P_{sat} \) directed perpendicular to the surface. As shown in [17], there exists a critical sample thickness \(h_c\) that determines the uniformity of director orientation in LC films, i.e. if the sample thickness is greater than \(h_c\), then a spontaneous orientational deformation is induced in the sample. For \(h < h_c\), the sample is not deformed. In our case, \(h_c \approx 2k / \tilde{W}\), where \(\tilde{W} = W_s + \varepsilon_c E_n^2 \delta / 8 \pi \) [18], \(W_s\) is still the surface energy in the sense of Rapini, and \(\varepsilon_c E_n^2 \delta / 8 \pi\) is the term describing the interaction between the anisotropic part of the NLC dielectric constant and by the surface field, \(\delta\) is the effective thickness of the polarization layer. If the initial orientation is homeotropic, then at \(h \geq h_c\) the layer becomes quasi-homeotropic and the general condition for finding the orientation angle \(\theta_c^*\) on the surface is a transcendental equation of the type:

\[
h_c \cdot \theta_c^* \cdot \sinh \theta_c^* = 0.\]

If \(h < h_c\), then \(\theta_c^* = 0\), but if \(h > h_c\), then \(\theta_c^* \neq 0\). A detailed analysis of this equation under the condition that \(\theta_c^* \ll 1\) is small is given in [19, 20]. Because the critical thickness \(h_c\) depends on the elastic modulus \(k(T)\) and on the generalized surface energy \(|\tilde{W}|\), then it should depend on temperature and in the vicinity of the phase transition can become larger than the thickness of the sample, which leads to condition \(\theta_c^* \approx 1\), those the sample becomes quasi-homeotropic. In particular, \(h < h_c\) at \(\Delta T - T_{N-I} \approx 2\Delta T - T_{N-I}\) in MBBA, since \(|\tilde{W}| = 0\) due to the equality \(|\tilde{W}| = 0\) due to the equality \(|W_s| = |\varepsilon_c E_n^2 \delta / 8 \pi|\) and the negative dielectric constant, this implies a decrease in the initial director orientation angle on the surface \(\theta_c^* \ll 1\) and the appearance of the signal at the second harmonic. A similar situation takes place in WHAOB. The described effect, in combination with optical methods, can also be used to diagnose orientational structural-phase transformations, as well as to measure the binding energy of LC molecules with a solid substrate. In the case of MBBA and WHAOB, the adhesion energy turned out to be equal to \(W_r \approx 0.8 \cdot 10^{-3}\) erg/cm\(^2\) and \(W_s \approx 10^{-3}\) erg/cm\(^2\), respectively.

A simpler situation is with the initial homeotropic and quasi-homeotropic orientation of the LC molecules. Let us analyze this case. It should immediately be noted that there is a significant difference in the nature of the behavior of the second harmonic. Compared to the temperature dependencies, \(U_{2\omega}\) increases in the entire temperature range up to the temperature of the phase transition to the isotropic phase. The behavior of the harmonic becomes especially critical in such LCs as HBACE and NOOFB (Figure 7b), which is inexplicable only by a change in the LC parameters near phase transitions in such ordered systems. The unusual behavior of the EMF generated at the doubled excitation frequency is apparently connected, as for the first harmonic, with the temperature dependence of the director oscillation angle \(\theta_d^*\) at the LC – solid substrate interface. Indeed, the observed signal is \(U_{2\omega} \approx U_\delta (\bar{\theta}_d^*)^2\), where \(U_\delta\) is the voltage drop across the surface polarization layer, and \((\bar{\theta}_d^*)^2\) is the average square of the director deviation on the surface, which is \(-0.05 (\bar{\theta}_d^*)^2\) \((\bar{\theta}_d^*)\) is the angle induced by the oscillating flow and \(\bar{\theta}_d^* \sim \bar{\theta}_c < \mu_0 \frac{\partial \theta_d^*}{\partial z}\) is equal to [1], where \(< \sim\) is the time averaging operation). But \(\theta_c\) can be determined for small perturbations from the data on photoelasticity by expanding in a series the sine in the formula

\[
U_{2\omega} = \frac{h}{2} P' (z) \int (\bar{\theta}_d^*)^2 dz = P' \delta (\bar{\theta}_d^*)^2 \cos \theta_d^* = U_\delta \cos \theta_d^* (\bar{\theta}_d^*)^2,
\]

where \(P'\) – surface polarization, \(U_\delta = P' \delta\) - voltage drop on the surface layer.

Then we get the ratio
\[ \bar{\theta}^2_c = \frac{4I_{2\omega}}{(\beta\hbar)^2 I_\theta'(\bar{\theta}'_d)}, \]

which is true for \(|a-a_o|<<1\), i.e. in the vicinity of the threshold of occurrence of \(U_{2\omega}\) and a constant tilt angle induced by a viscous wave in the LC \((a\geq a_o)\). From this, the behavior of the second harmonic becomes clear, namely, its magnitude increases due to the rapid growth of the square of the director oscillation angle \(\bar{\theta}'_d\). Knowing the temperature dependence \(\bar{\theta}^2_c\) and optical anisotropy \(\Delta n(T)\) dependence, we find the temperature changes \(U'_{2\omega}\) for the studied LCs (Figure 8).

Figure 8. Temperature dependencies of the voltage drop \(U'_{2\omega}-U_\delta\) on the surface polarization layer.

Let us analyze the behavior of the renormalized quantity \(U'_{2\omega}\). Firstly, it is obvious that \(U'_{2\omega}\) is maximum in the nematic phase and is practically not observed in smectics A and C and in the isotropic phase. This is consistent with the data of [19], in which the temperature dependence of the surface polarization \(P'\) was studied. In addition, the temperature dependence of \(U'_{2\omega}\) is actually a change in the drop in \(U_\delta\) potential on the surface polarization layer with the effective molecular thickness \(\delta\). For the studied series of crystals, \(U'_\delta\) are presented in Table 3.

Table 3. Potential drop on the surface polarization layer for various LC.

| Liquid crystals | \(U'_\delta\) \(10^{-1}\) V |
|-----------------|-----------------|
| MBBA            | 5.5             |
| WHAOB           | 3.0             |
| HBACE           | 5.5             |
| OCB             | 8.0             |
| NFOOB           | 4.5             |

The latter is in fairly good agreement with the data for MBBA, WHAOB and NFOOB obtained in [8]. For example, the potential jump in MBBA on the surface polarization layer, determined by the value of electroreflection from related substrates used in this work, turned out to be \(\Delta U_\delta\sim1\) V. If we take into account the estimated values of the polarized layer \(\delta\sim10^3\) Å obtained there, then we can calculate the polarization value \(P\sim3\cdot10^2\) units CGSE/cm². It should be emphasized that the obtained value is average and at the surface itself can reach a value of \(P\sim10^3\) units CGSE/cm².

4. Conclusion

In ordered media - liquid crystals formed by anisotropic molecules, a flexoelectric effect takes place. In such NLCs as MBBA and WHAOH, there are two flexocoefficients \(e_{11}\) and \(e_{33}\), because the shape of the molecules can be approximated by either a drop or a banana, and the dipole moment is directed at an angle to the long axis of the molecule. Such NLCs as HBACE, OCB, NFOOB also have two coefficients, but \(e_{11}\) is significantly larger than \(e_{33}\). This is due to the fact that the dipole moment is mainly directed along the long axis of the molecule, and the molecules themselves have an internal splay deformation.
An important result is the separation of the dipole and quadrupole mechanisms of molecular-orientation flexoelectric polarization. An increase in the electric moment of the molecule and its orientation in the direction of the long axis of the molecule leads to an increase in the “weight” of the quadrupole mechanism. Such a situation is typical for NLCs with high dielectric anisotropy, whose molecules tend to form pairs. In smectic A, flexoeffect coefficients are comparable and even somewhat larger than in nematic, but in smectic C, the flexoeffect could not be detected.

In the investigated LCs, another mechanism of molecular orientation polarization was discovered, which is a consequence of the orientational instability of molecules in a near-surface polarized layer in the field of acoustic disturbances. The latter is characteristic only for the nematic phase of liquid crystal ordering, which is apparently due to the smallness of the surface polarization in smectic and the greater orientational stability of the molecules of this phase to acoustic disturbances.

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