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To cite this article: Taiju Takahashi, Shusuke Nakamura & Yukihiro Kudoh (2015) Measurement of Flexo-coefficients for Nematic LCs without the Influence of the Impurity Ions by Using the Chromatographic Isolation Method, *Molecular Crystals and Liquid Crystals*, 611:1, 146-152, DOI: [10.1080/15421406.2015.1030211](https://doi.org/10.1080/15421406.2015.1030211)

To link to this article: <http://dx.doi.org/10.1080/15421406.2015.1030211>



Published online: 06 Jul 2015.



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Measurement of Flexo-coefficients for Nematic LCs without the Influence of the Impurity Ions by Using the Chromatographic Isolation Method

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The sum of the flexo-coefficients ($e_{11}+e_{33}$) was measured by the capacitance characteristic depending on the applied dc voltage in the HAN cell. The voltage for the minimum value of the capacitance was shifted by the influence of flexoelectric effect. One of the important problems for the evaluation for the flexo effect was the influence of impurity ions. Then, the chromatographic isolation phenomenon was used to separate impurity ions when the LC material was injected into the empty cell by the capillary action. The coefficient ($e_{11}+e_{33}$) of ZLI-4792 was measured, and a value of 26.0 pC/m was obtained.

Keywords flexoelectric; flexo-coefficients; HAN cell; impurity ions; Chromatographic Isolation

1. Introduction

The flexoelectric effect has been theoretically proposed by R. B. Mayer [1] in 1969. There is a possibility of the realization for the director switching in the nematic LC cell depending on the polarity of the applying electric field due to the flexoelectric polarization. The bistable switching LCDs operated by using the flexo effect have been proposed and investigated by some research groups, for example the achievements made by G. Durand's group around 1900 are widely known in the literature [2–6], though their work did not result in a practical method.

In the recent years extensive developments in the display modes have occurred and in the high definition LCD panel, some defects have been observed to respond to the polarity of the applied voltage. It has been said that some defects such light leakage or non-uniform optical transmittance at electrode edges in the pixel have occurred by the flexoelectric effects [7]. On the other hand, there is a proposal to actively use the flexoelectric effect for the low voltage operation of the IPS mode in LCD [8–9].

The measurement of the flexo-coefficient has been attempted by a number of researchers. The problem is that each researcher has reported a different value for the same

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material and these values are distributed over a broad range of values. An accurate value is required for the measurement of the flexoelectric coefficients. The observation or detection for the flexoelectric effect is not so easy because the effect occurs over a small area in the sample. Furthermore, one of the important points for the evaluation of the flexo effect is to separate the effect of impurity ions. Avoiding the influence of impurity ions contained in LC materials is difficult when a characteristic of the LC materials is obtained under applying the dc voltage. Because flexoelectric effects were induced by the dc applying voltage, and, also impurity ions responded to the dc voltage, i.e., it meant that it was difficult to observe those two effects as an independent phenomenon.

Several years ago, our research group attempted to measure the flexo-coefficients using the HAN cell by fitting method which compared the measurement results with the numerical calculated results for the electro-optical characteristics [10–11]. However, it seems that the countermeasure against impurity ions was not sufficiently at that time, when we think back now. Then, in this paper, we have proposed a novel measurement method where we used the chromatographic isolation phenomenon by avoiding the influence of the impurity ions. And, the measurement was done by using the capacitance against the dc applying voltage characteristics (C-V curves) of the HAN cell, instead of measuring the conventional optical transmittance characteristics.

2. Measurement for Flexo-coefficients ($e_{11}+e_{33}$)

Flexo-coefficients e_{11} and e_{33} pertain to the splay distortions and the bend distortions, respectively. The measurement for obtaining the value as an independent coefficient is difficult. In many cases, the value is determined as the sum or difference of e_{11} and e_{33} . We propose a novel measurement method for the sum of flexo-coefficients ($e_{11}+e_{33}$) by minimising the effects of impurity ions as much as possible. We paid attention to the chromatographic isolation phenomenon in the LC cell, it has been shown to be of benefit for removing the impurity ions. In general, this effect is observed when LC materials are injected into the empty cell. A voltage holding ratio (VHR) is one of the evaluation methods amount of impurity ions contained in the LC materials. Then, the VHR may show higher value in the area of the cell where the distance is far from the injection gate [12, 13].

Furthermore, the electro-optical measurement was not used in this experiment. As a reason, the measurement error easily appears under the optical measurements of the cell. For instance, a misalignment of the optical incident angle to the cell is given the error, etc. In this paper, a method to measure the capacitance characteristics in the cell was attempted. The optical adjustment in the measurement system was not necessary for the measurement of the capacitance characteristics. In addition, the capacitance characteristics corresponding to the director reorientation to the varying applied voltage are more sensitive compared with the optical characteristics in the LC cell. The dc voltage was applied parallel across the cell thickness in the HAN cell and the capacitance depending on the voltage was measured. Of course, before carrying out each measurement, the ac voltage was applied to the cell for diffusing impurity ions in the cell. The voltage that shows the minimum value of the capacitance was shifted by the effect of the induced flexoelectric polarization in the HAN cell. The sum $e_{11}+e_{33}$ was determined by comparing the measurement value and the calculated value for the amount of shifted voltage. Here, the absolute value of the capacitance was not an important parameter.

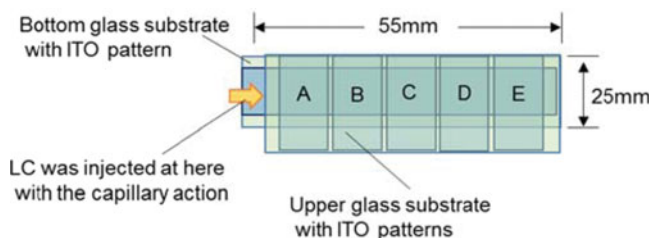


Figure 1. Electrode patterns of the cell for measurement.

2.1. Fabrication of measurement cells

The rectangle-shaped HAN cell as shown in Fig. 1 was fabricated. It was expected that impurity ions on the surface of orientation film were adsorbed by the chromatographic isolation effect during the injection process of LC materials. The ITO pattern in the cell was separated in five parts to observe the characteristics for each area. These were numbered as A to E from the injection gate as shown in Fig. 1. The polyimide alignment material RN-1338, (Nissan Chemical industries) was used for both the vertical and horizontal alignment films. The pretilt angle was able to control by the baking temperature; the baking temperature was 250, for the horizontal alignment and 180, for the vertical alignment, respectively. Then, the rubbing treatment was done to only horizontal alignment substrate. The cell was assembled with a cell thickness of $10\mu\text{m}$. The LC material was injected into the cell from the injection gate by the capillary action in the isotropic phase. By the way, it seemed that an LC material consisting of a single compound was better than the mixture of compounds for observing the flexo-effects because the origin of the inducing flexo-polarization depends on the shape of the LC molecules. However, we used the LC mixture material ZLI-4792 (Merck) in this experiment. This material has been known as a fluorine series and shown a high VHR due to a very low amount of content of impurity ions. And, in our previous other experiments [9], the bistable switching of the LC cell injected with ZLI-4792 was realized under applying the dc electric field. So, this LC material was selected by showing a possibility to induce the flexo-polarization.

2.2. Measurement system

The block diagram of measurement system is shown in Fig. 2. The impedance analyzer SI 1260 (Solartron) was used. The voltage waveform shown in Fig. 3 was applied to the cell. The applied voltage of the polarity was alternately switched, and the magnitude of voltage was reduced from 5V to zero in steps of 0.1V, as shown in Fig. 3. Each measurement was carried out under applying the dc voltage after applying the ac voltage due to diffusing

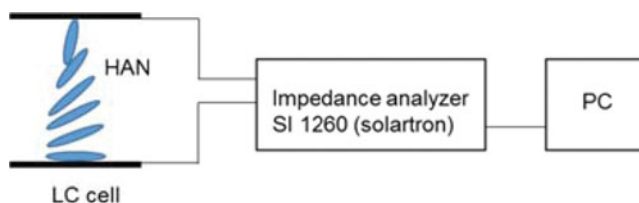


Figure 2. Block diagram of measurement system.

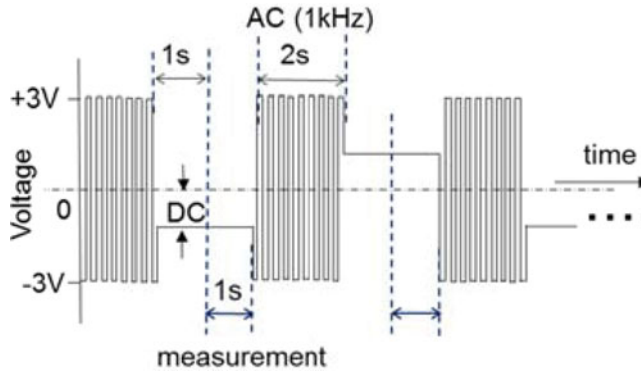


Figure 3. Schematic chart for the applied voltage.

impurity ions. The measurement was done at the room temperature. In this measurement, the absolute value of capacitance was not required, however, the voltage V_s which showed a minimum value of the capacitance was important. The least-square technique was used to find the voltage V_s in the measurement data. Then, the resolution in the measurement data can increase by this numerical technique. The sum of coefficients $e_{11}+e_{33}$ was determined by the fitting of V_s with the numerical calculation.

3. Theoretical Equations

Some equations for the numerical simulations by the continuum theory were shown as follows.

The direction of the applying electric field was along with the cell thickness direction.

$$\vec{E} = (0, 0, E_z)$$

LC director $\vec{n} = (\cos \theta, 0, \sin \theta)$.

Displacements in x -axis and y -axis directions are $\frac{\partial}{\partial x} = 0$, $\frac{d}{dy} = 0$, respectively.

The free energy density in the bulk part including the flexoelectric term with the electrode is written as follow;

$$W = \frac{1}{2} f(\theta) \left(\frac{\partial \theta}{\partial z} \right)^2 - \frac{1}{2} \varepsilon(\theta) \left(\frac{\partial V_\phi}{\partial z} \right)^2 + \frac{1}{2} (e_{11} + e_{33}) \sin 2\theta \left(\frac{\partial \theta}{\partial z} \right) \left(\frac{\partial V_\phi}{\partial z} \right)$$

where, $f(\theta) = K_{11} \cos^2 \theta + K_{33} \sin^2 \theta$, $\varepsilon(\theta) = \varepsilon_0 (\varepsilon_p \sin^2 \theta + \varepsilon_n \cos^2 \theta)$, and $\frac{\partial V_\phi}{\partial z} = \text{grad} V_\phi = -E_z$, V_ϕ is the electric potential.

Table 1. Parameters for numerical simulations (LC material parameters of ZLI-4792)

K_{11} [pN]	13.2	A_0 [J/m ²]	1×10^{-3}
K_{33} [pN]	13.2	A_d [J/m ²]	1×10^{-4}
ε_p	8.3	θ_0 [deg.]	2.0
ε_n	3.1	θ_d [deg.]	89.9
d [μ m]	10.0		

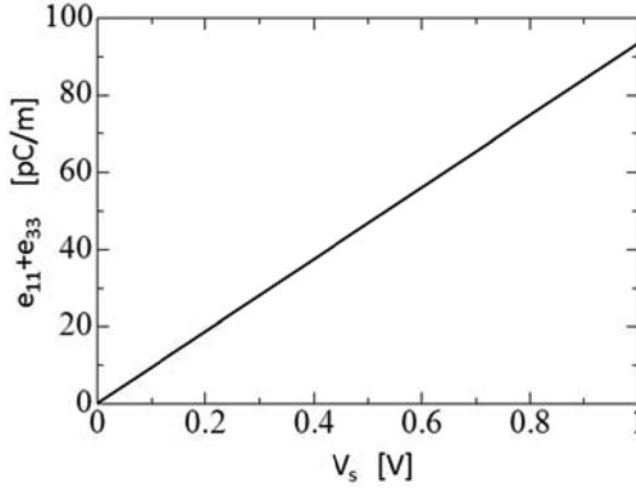


Figure 4. Flexo-coefficients ($e_{11}+e_{33}$) vs. V_s (Theoretical characteristic).

The surface anchoring energy density on each substrate surface is

$$f_0 = \frac{1}{2} A_0 \sin^2 (\theta_{[0]} - \theta_0) \text{ at } z = 0, \text{ and } f_d = \frac{1}{2} A_d \sin^2 (\theta_{[d]} - \theta_d) \text{ at } z = d,$$

where, A_0 and A_d are anchoring energy coefficients at $z = 0$, $z = d$, respectively.

Then, whole energy density in the unit area F is written as

$$F = \int_0^d W dz + f_0 + f_d.$$

The Euler-Lagrange equation with θ and V_φ about the bulk equation is given as

$$2f(\theta) \left(\frac{\partial^2 \theta}{\partial z^2} \right) + f'(\theta) \left(\frac{\partial \theta}{\partial z} \right)^2 + \varepsilon'(\theta) \left(\frac{\partial V_\varphi}{\partial z} \right)^2 + (e_{11} + e_{33}) \sin 2\theta \left(\frac{\partial^2 V_\varphi}{\partial z^2} \right) = 0$$

$$\text{where, } \frac{\partial V_\varphi}{\partial z} = \frac{e_{11} + e_{33}}{2\varepsilon(\theta)} \sin 2\theta \left(\frac{\partial \theta}{\partial z} \right) - \frac{\alpha}{\varepsilon(\theta)},$$

$$\text{and } \alpha = \left\{ \int_0^d \frac{(e_{11}+e_{33}) \sin 2\theta \left(\frac{\partial \theta}{\partial z} \right)}{2\varepsilon(\theta)} dz - V \right\} / \int_0^d \frac{1}{\varepsilon(\theta)} dz.$$

The Euler-Lagrange equation about each surface equation is given as

$$f(\theta_{[0]}) \left(\frac{\partial \theta}{\partial z} \right) \Big|_{z=0} + \frac{1}{2} (e_{11} + e_{33}) \left(\frac{\partial V_\varphi}{\partial z} \right) \Big|_{z=0} \sin 2\theta_{[0]} - \frac{1}{2} A_0 \sin \{2(\theta_{[0]} - \theta_0)\} = 0$$

at $z = 0$, and

$$f(\theta_{[d]}) \left(\frac{\partial \theta}{\partial z} \right) \Big|_{z=d} + \frac{1}{2} (e_{11} + e_{33}) \left(\frac{\partial V_\varphi}{\partial z} \right) \Big|_{z=d} \sin 2\theta_{[d]} + \frac{1}{2} A_d \sin \{2(\theta_{[d]} - \theta_d)\} = 0$$

at $z = d$.

It is clear from those equations that the e_{11} and e_{33} cannot be separated, when the cell in which the voltage is applied across the cell thickness direction. The numerical calculations

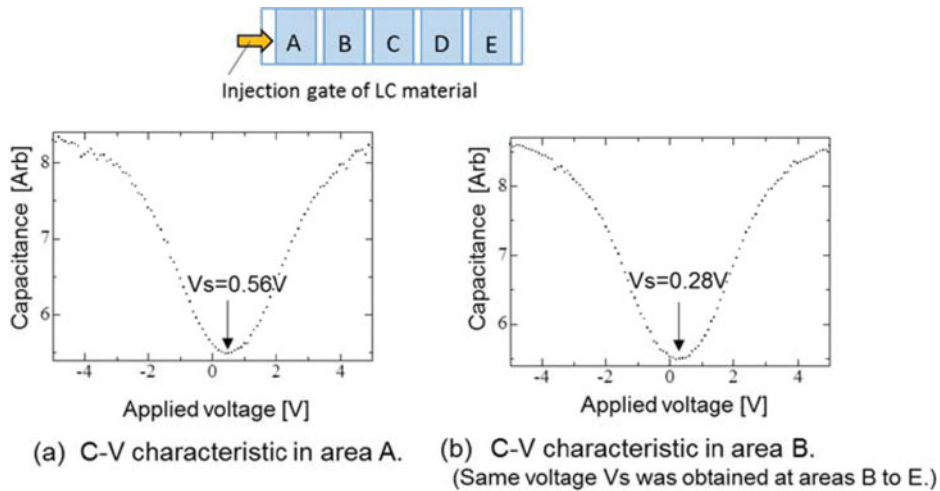


Figure 5. Measurements curves of C-V characteristics (ZLI-4792).

of director distribution taking account of flexoelectric effects were carried out by using material parameters of ZLI-4792, as shown in Table 1. Then, the C-V characteristics were calculated, and the voltage V_s which gave the minimum value of the capacitance was extracted and plotted, then the theoretical characteristics of $e_{11}+e_{33}$ vs. V_s were obtained, as shown in Fig. 4.

4. Results and Discussion

The measurement results of C-V characteristics were shown in Figs. 5(a) and (b). The least-square technique was used to determine the value of V_s . The result at the measurement area A in the cell was shown in Fig. 5(a), 0.56V of V_s was obtained. In areas B through E, almost same characteristics were found, then, the C-V curve at the area B was shown as a representative property. In these area, the same value, $V_s = 0.28V$, was obtained, i.e., the characteristics were saturated. It was confirmed that the farther area from the injection gate of the cell, the smaller value of V_s was shown and the value of V_s tended to saturate in general. This meant that impurity ions were reduced at the farther area from the injection gate by the chromatographic adsorption effect. Then, the acceptance value V_s for determining the flexo-coefficient should be selected in such areas. The value of $V_s = 0.28V$ was applied to the theoretical characteristic for the fitting (Fig. 4) to determine the sum of flexo-coefficients $e_{11}+e_{33}$, then, the coefficient $e_{11}+e_{33}$ of the nematic mixture ZLI-4792 was determined as 26.0 pC/m.

5. Conclusion

We proposed the novel measurement method to determine the sum of flexo-coefficients $e_{11}+e_{33}$ avoiding the influence of impurity ions by using the chromatographic isolation technique. In our method, the capacitance in the HAN cell under applying the dc voltage was measured. This method does not use any optical characteristics, and it has a possibility of reducing the measurement error which occurs in the electro-optical measurement system,

for example an adjustment of the optical alignment. Of course, we have to verify measurement errors in our method, as rapidly as possible. In the future, the relationship of the VHR and flexo-coefficients should be measured and clarified if possible. The flexo-coefficients for other LC materials (5CB, E7, ZLI-2293, etc) will also be measured.

Acknowledgments

We would like to thank Merck for supplying the LC materials, we also thank Nissan Chemical Industries Japan for supplying the polyimide material.

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