

INFLUENCE OF MOLECULAR STRUCTURE OF FERRIELECTRIC LIQUID CRYSTAL MIXTURES ON THEIR ELECTRO-OPTICAL PROPERTIES

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ABSTRACT

The effect of non-mesogenic chiral additives, such as *(S,S)*-bis-(1,1,1-trifluoro-oct-2-yl)-4,4"-terphenyl-dicarboxylate (FODTA-6) and *(S,S)*-(1-butoxy-1-oxopropan-2-yl)-[1,1':4'1"-terphenyl]-4,4"-dicarboxylate (SS-LACT-4), on the properties of ferrielectric liquid crystal (FiLC) mixtures FerriLCM-1 and FerriLCM-2 was investigated. FODTA-6 induced a positive sign for spontaneous polarization and handedness in mixtures of polar smectic C* liquid crystals, while SS-LACT-4 induced a positive sign for spontaneous polarization but a negative sign for handedness. A non-chiral smectic C liquid crystal 2-(4'-pentylbiphenyl-4'-yl)-5-hexyl pyrimidine (BPP-65) was used as a matrix. It was found that the absence of the chiral SS-LACT-4 dopant in FerriLCM-2 mixture resulted in a significant change of dielectric and electro-optical properties of the FiLC. In FerriLCM-2 mixture, several parameters decreased compared to FerriLCM-1: spontaneous polarization, electro-optical response time, rotational viscosity, electric susceptibility, and orientational Kerr-effect coefficient K_{kerr} . At the same time the electric critical fields E_{cp1} and E_{cp2} significantly increased. Within the framework of the existing phenomenological theory of chiral smectics C*, a quadratic dependence between orientational Kerr-effect coefficient and free relaxation time of helical structure was obtained. This quadratic relationship has been experimentally confirmed, yet it was also demonstrated that there are significant quantitative discrepancies between experimental data and theoretical calculations for FiLCs.

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ВЛИЯНИЕ МОЛЕКУЛЯРНОГО СТРОЕНИЯ ФЕРРИЭЛЕКТРИЧЕСКИХ
ЖИДКОКРИСТАЛЛИЧЕСКИХ СМЕСЕЙ НА ИХ ЭЛЕКТРООПТИЧЕСКИЕ СВОЙСТВА

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АННОТАЦИЯ

В работе исследовано влияние немезогенных хиральных добавок (*S,S*)бис-(1,1,1-трифторокт-2-ил)-4,4''-терфенилдикарбоксилата (FODTA-6) и (*S,S*)бис(1-бутоксипропан-2-ил)-[1,1':4'1''-терфенил]-4,4''-дикарбоксилата (SS-LACT-4) на свойства двух смесевых ферриэлектрических жидких кристаллов (ФЖК) FerriLCM-1 и FerriLCM-2. FODTA-6 индуцирует в смесях полярных смектических С* жидких кристаллов положительный знак спонтанной поляризации и волнового вектора, а SS-LACT-4 – положительный знак спонтанной поляризации, но отрицательный знак волнового вектора. В качестве матрицы используется нехиральный смектический С жидкий кристалл 2-(4'-пентилбифенил-4-ил)-5-гексил-пиримидин (BPP-65). Показано, что отсутствие в составе FerriLCM-2 хиральной добавки SS-LACT-4 приводит к существенному изменению диэлектрических и электрооптических характеристик ФЖК. В смеси FerriLCM-2, по сравнению с FerriLCM-1, уменьшаются следующие параметры: спонтанная поляризация, время электрооптического отклика, вращательная вязкость, диэлектрическая восприимчивость и коэффициент ориентационного эффекта Керра $K_{\text{Керр}}$. При этом существенно возрастают электрические критические поля E_{cp1} и E_{cp2} . В рамках существующей феноменологической теории хиральных смектиков С* получена квадратичная зависимость величины коэффициента ориентационного эффекта Керра от времени свободной релаксации возмущений геликоидальной структуры. Квадратичный характер этой зависимости подтвержден экспериментально, при этом показано, что существуют заметные количественные различия между экспериментом и результатами расчетов для ФЖК.

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Introduction

The phenomenon of polar liquid crystals (LC) has long been a subject of intense research interest among scientists. Following the discovery of ferroelectric liquid crystals (FLC) by Meyer [1], subsequent investigations have unveiled the existence of antiferroelectric liquid crystals (AFLC) [2, 3], as well as ferrielectric liquid crystals (FiLC) [4]. All the mentioned polar phases are chiral smectic C* LCs. There has been a wealth of research exploring the potential practical applications of FLCs and AFLCs [5–8]. However, the ferrielectric phase is often regarded as an intermediate state between antiferroelectric and ferroelectric phases due to its high temperature of existence and narrow temperature range [9, 10]. Therefore, most research on FiLCs has just focused on experimental and theoretical characterization of ferrielectric packages [11–14].

Previously, we have developed a liquid crystal mixture **FerriLCM-1** [15], which exhibits a ferrielectric phase over a wide range of temperatures, including room temperature. The mixture features a helix pitch $p_0 < 120$ nm, switching times $\tau < 300$ μ s in the DHF-mode (deformed helix ferroelectric [16]), high orientational Kerr-effect coefficient reaching up to 1700 nm/V² [17] and relatively low critical electric

fields $E_{\text{cpl}} < 0.5$ V/ μ m. All the above along with the electro-optical properties make this material highly promising for practical applications. In this paper, we investigate the correlations between molecular structure of ferrielectric liquid crystals and their electro-optical and dielectric properties. We also carried out an experimental verification of the applicability of the phenomenological theory developed for smectic C* ferroelectric liquid crystals to ferrielectric liquid crystals.

Materials and methods

Two helical ferrielectric liquid crystal mixtures: **FerriLCM-1** and **FerriLCM-2**, which have been developed by the authors at the Lebedev Physical Institute, were investigated. The mixtures consist of the following components: *2-(4'-pentylbiphenyl-4-yl)-5-hexylpyrimidine* (**BPP-65**) [18], which exhibits a non-chiral smectic C phase; *(S,S)-bis(1,1,1-trifluorooctan-2-yl)-[1,1':4',1''-terphenyl]-4,4''-dicarboxylate* (**FODTA-6**) [19] and *(S,S)-bis(1-butoxy-1-oxopropan-2-yl)-[1,1':4',1''-terphenyl]-4,4''-dicarboxylate* (**SS-LACT-4**), which are non-mesogenic chiral dopants. The composition of the used FiLCs, their signs of spontaneous polarization P_s [20] and helix handedness [21] (twist sign “+” for right-handed and “-” for left-handed) are presented in Table 1.

Table 1. The chemical compositions of ferrielectric liquid crystal mixtures

№	Compound	Chemical structure	Molar concentration in mixture, %		P_s sign	Handedness
			FerriLCM-1	FerriLCM-2		
1	BPP-65		61.8	69.2		
2	FODTA-6		28.7	30.8	+	-
3	SS-LACT-4		9.5	0	+	+

The **FerriLCM-2** liquid crystal is presented for the first time here. It consists only of **BPP-65** and **FODTA-6** compounds in similar molar ratio as for **FerriLCM-1**. The **FerriLCM-2** mixture undergoes the following phase transitions upon heating: solid crystal $\rightarrow +21$ °C \rightarrow solid crystal and ferrielectric smectic C*

phase $\rightarrow +45$ °C \rightarrow ferrielectric smectic C* $\rightarrow +95$ °C \rightarrow smectic A* $\rightarrow +100$ °C \rightarrow smectic A* and isotropic phase $\rightarrow +112$ °C \rightarrow isotropic phase. Upon cooling, the ferrielectric smectic C* phase exists in a supercooled state for an extended period (up to several years) without crystallizing at room temperature.

In our study we use planar-aligned FiLC layers in electro-optical sandwich-type cells, which are described in detail in papers [22, 23]. The thickness of the FiLC layer d was 1.8 μm for **FerriLCM-2** and 57 μm for **FerriLCM-1**. The helix pitch of the FiLCs was measured by Bragg reflections from the periodic homeotropically-aligned FiLC layers [24] using Ocean Optics USB 2000+ VIS-NIR-ER spectrometer.

Dependencies of the liquid crystal cells polarization P on the electric field E were measured via integrating the polarization reversal current by an external capacitor [25], and the spontaneous polarization P_s was determined from the level of saturation of the $P(E)$ dependence. The electric susceptibility $\chi(E)$ of supramolecular structure was obtained by differentiating the measured $P(E)$ dependencies:

$$\chi(E) = \frac{1}{\varepsilon_0} \frac{\partial P(E)}{\partial E} - \chi_\infty, \quad (1)$$

where ε_0 is vacuum permittivity, and χ_∞ is the high-frequency part of the electric susceptibility, which is related to the electronic polarizability of the LC structure. By analogy with ferroelectric liquid crystals, we have also calculated the electric susceptibility of the Goldstone mode χ_G as:

$$\chi_G = \lim_{E \rightarrow 0} \chi(E). \quad (2)$$

The values of critical electric fields E_{cp1} and E_{cp2} (which are observed in FiLCs [11, 15, 26]) were defined as maxima of $\chi(E)$ dependencies. Electro-optical response time $\tau_{0.1-0.9}$ in DHF-mode was measured similarly to [27]. The rotational viscosity γ_φ was calculated from the relation (3) according to [28]:

$$\tau_{0.1-0.9}^C = 2.2 \frac{\gamma_\varphi}{P_s E}, \quad (3)$$

where $\tau_{0.1-0.9}^C$ is a dielectric response time measured on external capacitor at $E > E_{cp2}$.

Under the electric field action, a quadratic variation in the electrically induced effective birefringence $\Delta n_{\text{eff}}^E(E, \lambda)$ of a helical structure is observed both in FLCs and FiLCs [29]:

$$\Delta n_{\text{eff}}^E(E, \lambda) = K_{\text{kerr}}(\lambda) \lambda E^2, \quad (4)$$

where K_{kerr} is the orientational Kerr-effect coefficient, λ is the radiation wavelength.

The electrically induced effective birefringence $\Delta n_{\text{eff}}^E(E, \lambda)$ and the effective birefringence $\Delta n_{\text{eff}}^0(\lambda)$ at $E = 0$, which is given by eq. (5), are the components

of the total effective birefringence $\Delta n_{\text{eff}}(E, \lambda)$ associated with the helical structure (6):

$$\Delta n_{\text{eff}}^0(\lambda) = \Delta n_{\text{mol}}(\lambda) \left(1 - \frac{3}{2} \sin^2 \theta \right), \quad (5)$$

$$\Delta n_{\text{eff}}(E, \lambda) = \Delta n_{\text{eff}}^0(\lambda) + \Delta n_{\text{eff}}^E(E, \lambda), \quad (6)$$

where $\Delta n_{\text{mol}}(\lambda)$ is the birefringence dispersion under condition $E > E_{cp2}$, and θ is the tilt angle of the liquid crystal director in smectic layers.

The light transmittance T_{cell} through a sandwich-type cell placed between crossed polarizers was measured directly [8]. It is described in [30] by eq. (7):

$$T_{\text{cell}} = \sin^2 2[\beta + \psi_d(E)] \sin^2 \left[\frac{\pi d \Delta n_{\text{eff}}(E, \lambda)}{\lambda} \right], \quad (7)$$

where β is an angle between the incident light polarization plane and the helix axis at $E = 0$, and $\psi_d(E)$ is an angle of the FiLC main optical axis rotation under the electric field E , measured in accordance with the methodology outlined in the SI of the work [31]. Then, according to eqs. (4)–(7), the values of $\Delta n_{\text{eff}}(E, \lambda)$, $\Delta n_{\text{eff}}^0(\lambda)$ and $\Delta n_{\text{eff}}^E(E, \lambda)$ were obtained.

The experiments were carried out using a semiconductor laser with $\lambda = 532$ nm, a photodetector (photomultiplier tube FEU-68), a Rigol DS1154 oscilloscope and an AKIP 3408/1 waveform generator. An alternating square-wave voltage with a zero-voltage interval was applied to FiLC cell, following the procedure outlined in [15] (see inset of Fig. 2, *a* of that work). In order to reliably maintain a static condition for electro-optical and dielectric characteristics of FiLCs, a signal frequency of $f = 10$ Hz was used [32]. In order to maintain a temperature accuracy of ± 0.1 °C, the FiLC cell was placed into a specially designed hot-stage.

Results and discussion

The chiral dopants **SS-LACT-4** and **FODTA-6**, used in **FerriLCM-1** mixture, induce opposite helix handedness, but the same spontaneous polarization sign (see Table 1). The spontaneous polarization of **FerriLCM-2** mixture, which is obtained by excluding **SS-LACT-4** from **FerriLCM-1**, decreases by 35 % and the tilt angle θ of the molecules in smectic layers increases from 35.3° to 36.6° (Fig. 1, *a*). The dielectric susceptibility χ_G decreases by 85 % (Fig. 1, *b*). **FerriLCM-2** mixture begins melting from the crystalline phase at 21 °C and exhibits a two-phase

region up to 45 °C. The ferrielectric phase persists for an extended period of time when cooled. The phase transition temperature to the paraelectric smectic A*

phase decreases from 100 °C (for **FerriLCM-1** mixture) to 90 °C (for **FerriLCM-2** mixture).

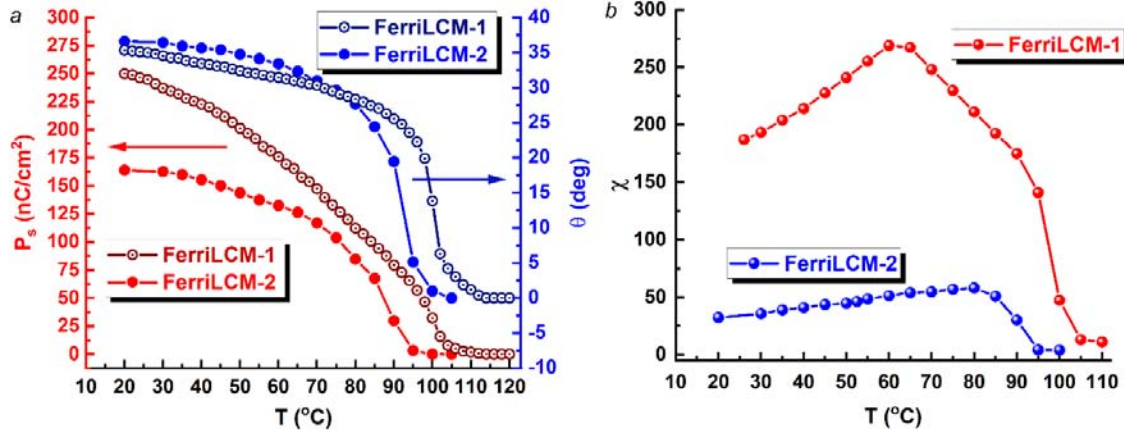


Fig. 1. Temperature dependences of: *a* – the spontaneous polarization P_s and the tilt angle θ for **FerriLCM-2** and for **FerriLCM-1**; *b* – the electric susceptibility χ_G during cooling for **FerriLCM-2** and **FerriLCM-1**

Ferrielectric liquid crystals are characterized by the existence of two critical electric fields (E_{cp1} and E_{cp2}), at which a nonlinear increase in polarization and the angle of the FiLC main optical axis rotation occurs. These critical electric fields correspond to the helix unwinding and the destruction of ferrielectric packages [14, 26]. The critical electric fields temperature dependencies are shown in Fig. 2, *a*. It can be seen that the critical electric field E_{cp1} of **FerriLCM-2** increases by almost 3.8 times, compared with **FerriLCM-1**, and E_{cp2} increases by 3.2 times. It

is worth noting that for both FiLCs, the value of E_{cp1} remains relatively constant within a certain temperature range until the temperature, at which the critical electric fields merge and become indistinguishable. These temperatures are 52.5 °C and 48 °C for **FerriLCM-2** and **FerriLCM-1**, respectively. During electro-optical switching in DHF-mode, the electric fields are limited to the values $E < E_{cp1}$, at which the condition $\chi(E) = const$ is fulfilled. The electric field strength E^{DHF} , at which this condition no longer holds, is shown in Fig. 2, *b*.

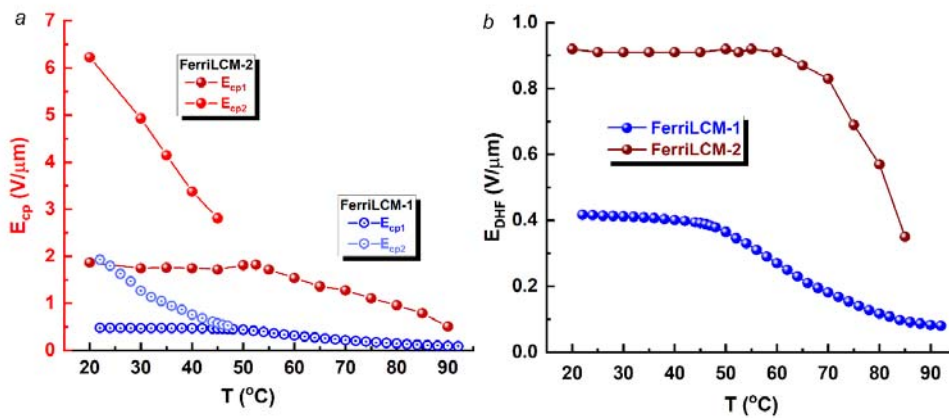


Fig. 2. Temperature dependencies of: *a* – the critical electric fields E_{cp1} and E_{cp2} ; *b* – the electric fields strength E^{DHF} , which limits the DHF-mode existence for **FerriLCM-1** and **FerriLCM-2**

According to eq. (7), such characteristics as $\psi_d(E)$ and $\Delta n_{\text{eff}}^E(E, \lambda)$ are of importance for the use of liquid crystals as electro-optical materials. Fig. 3 demonstrates the temperature dependencies of the maximum values of the angle of the FiLC main optical axis rotation $\psi_d(E^{\text{DHF}})$ and the electrically induced effective birefringence $\Delta n_{\text{eff}}^E(E^{\text{DHF}}, \lambda)$ achievable in DHF-mode for **FerriLCM-2** and **FerriLCM-1**.

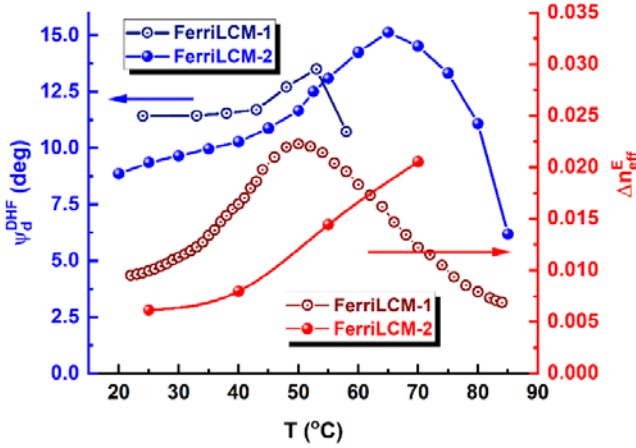


Fig. 3. Temperature dependencies of the maximum achievable values of the angle of the FiLC main optical axis rotation $\psi_d(E^{\text{DHF}})$ and effective electrically controlled birefringence $\Delta n_{\text{eff}}^E(E^{\text{DHF}})$ in the DHF-mode for **FerriLCM-1** and **FerriLCM-2**, $\lambda = 532$ nm

The angle ψ_d , at which the maximum value of $\sin^2 2[\beta + \psi_d(E)]$ of eq. (7) occurs in the most common case $\beta = 0$, is 45 degrees. Thus, it is advantageous for us to increase the angle $\psi_d(E^{\text{DHF}})$ when using liquid crystal cell between crossed polarizers. Therefore, **FerriLCM-1** has a slight advantage over **FerriLCM-2** in terms of providing maximum light transmittance. Additionally, the range of variation in Δn_{eff}^E is also larger for **FerriLCM-1** up to a temperature of approximately 62 °C. However, when using FiLC as an electro-optical medium for the phase modulators, in order to minimize ellipticity of the light

passing through a liquid crystal cell, it is important to minimize ψ_d [7]. From this perspective, despite the increased control voltages, **FerriLCM-2** is more profitable, since it has lower values of $\psi_d(E^{\text{DHF}})$, especially at room temperature. The electro-optical response time of **FerriLCM-2** is significantly less than that of **FerriLCM-1**. The on-time τ^{on} decreases from 301 μs to 98 μs , and the off-time τ^{off} decreased from 256 μs to 46 μs at $T = 25$ °C. The temperature dependencies of the electro-optical response time in the DHF-mode can be seen in Fig. 4.

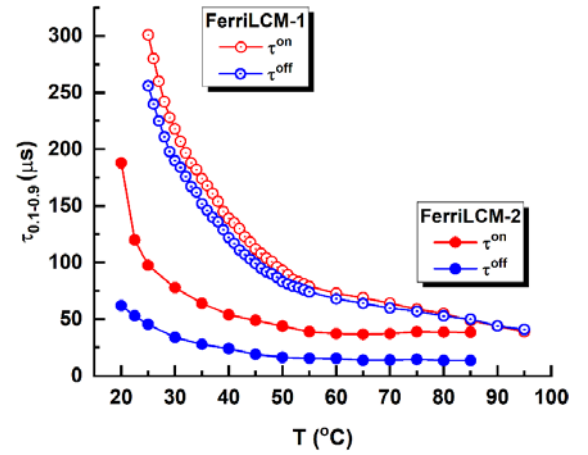


Fig. 4. Temperature dependencies of the electro-optical response time (on-time $\tau_{0.1-0.9}^{\text{on}}$ and off-time $\tau_{0.1-0.9}^{\text{off}}$) in DHF-mode for **FerriLCM-1** and **FerriLCM-2**

Excluding **SS-LACT-4** from **FerriLCM-1** mixture also results in a significant reduction in the helix pitch (see Fig. 5, a). Specifically, selective reflections from a periodic helical structure at a wavelength of $\lambda = 375$ nm, corresponding to the helix pitch $p_0 = 125$ nm, are observed at 65 °C and 39 °C for **FerriLCM-2** and **FerriLCM-1**, respectively. Additionally, the rotational viscosity calculated by eq. (3) is also lower at room temperature for **FerriLCM-2**. The temperature dependencies of rotational viscosity are illustrated in Fig. 5, b.

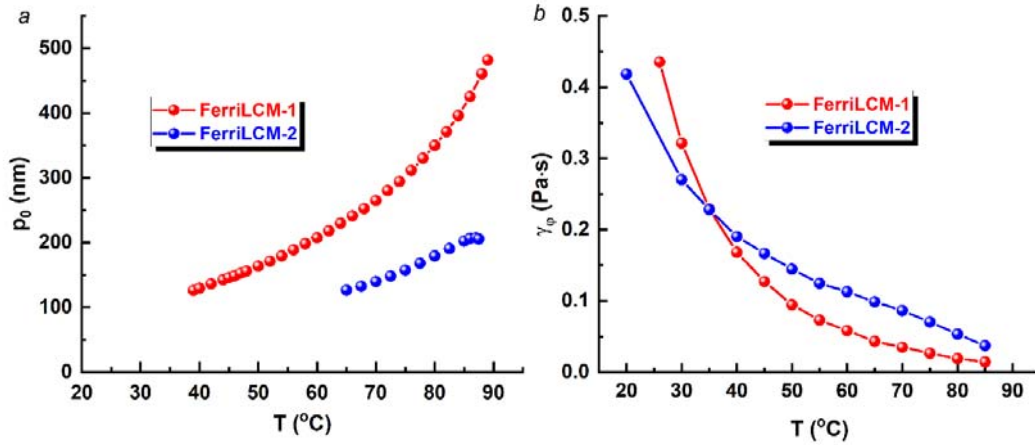


Fig. 5. Temperature dependencies of: *a* – the helix pitch p_0 ; *b* – rotational viscosity γ_ϕ for **FerriLCM-1** and **FerriLCM-2**

According to eq. (4), the electrically induced effective birefringence $\Delta n_{\text{eff}}^E(E, \lambda)$ is proportional to the square of the electric field E^2 . In paper [33], a formula has been derived that relates $\Delta n_{\text{eff}}^E(E, \lambda)$ to the main parameters of the polar liquid crystals: χ_G , P_s , θ and Δn_{mol} :

$$\Delta n_{\text{eff}}^E(E, \lambda) = \Delta n_{\text{mol}}(\lambda) \frac{\sin^2 2\theta}{1 - 3/2 \cdot \sin^2 \theta} \left(\frac{\varepsilon_0 \chi_G}{P_s} \right)^2 E^2. \quad (8)$$

Thus, from eqs. (4) and (8) we get:

$$K_{\text{kerr}} = \frac{\Delta n_{\text{mol}}(\lambda)}{\lambda} \frac{\sin^2 2\theta}{1 - 3/2 \cdot \sin^2 \theta} \left(\frac{\varepsilon_0 \chi_G}{P_s} \right)^2. \quad (9)$$

Using expressions for the dielectric susceptibility χ_G [34] according to eq. (10) and for the time τ^{off} of free relaxation of the helix structure according to eq. (11) [16], we can find a relationship between the orientational Kerr-effect coefficient K_{kerr} and τ^{off} , which is expressed by the equation (12), obtained by the authors:

$$\chi_G = \frac{P_s^2}{2\varepsilon_0 \sin^2 \theta q_0^2}, \quad (10)$$

$$\tau^{\text{off}} = \frac{\gamma_\phi}{K_\phi \sin^2 \theta q_0^2}, \quad (11)$$

$$K_{\text{kerr}} = \frac{\Delta n_{\text{mol}}(\lambda)}{\lambda} \frac{\sin^2 2\theta}{1 - 3/2 \cdot \sin^2 \theta} \left(\frac{P_s}{2\gamma_\phi} \right)^2 \tau_{0.1-0.9}^{\text{off}^2}. \quad (12)$$

So, the K_{kerr} value is proportional to $\tau_{0.1-0.9}^{\text{off}^2}$ as it fol-

lows from eq. (12). It is taken into account here that τ^{off} and $\tau_{0.1-0.9}^{\text{off}}$ are equal in order of magnitude. Here, in (10)–(12), K_ϕ is the helix elastic modulus in one constant approximation, and $q_0 = 2\pi/p_0$.

Figure 6 shows experimental dependencies $K_{\text{kerr}}(T)$ for **FerriLCM-1** and **FerriLCM-2**, and also theoretical dependencies $K_{\text{kerr}}(T)$, obtained using eqs. (9) and (12), for **FerriLCM-1**.

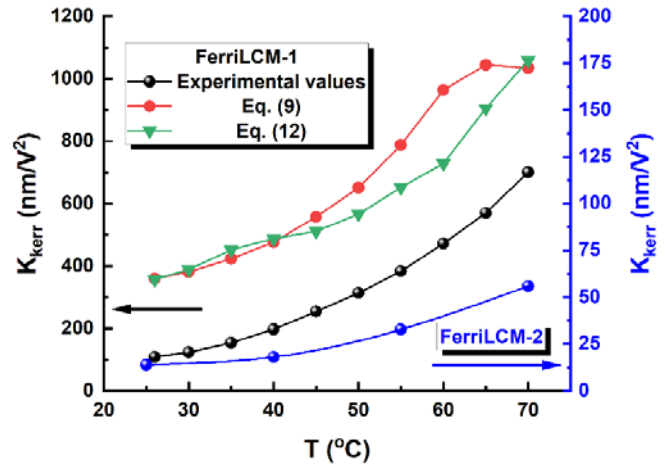


Fig. 6. Temperature dependencies of the Kerr orientational effect coefficient: left axis corresponds to experimentally determined and calculated according to equations (9), (12)

K_{kerr} for **FerriLCM-1**; right axis corresponds to experimentally determined K_{kerr} for **FerriLCM-2**,

$$\lambda = 532 \text{ nm}$$

The values of K_{kerr} calculated by eqs. (9) and (12) only qualitatively coincide with the experiment, but quantitatively there is a difference of about 3 times. This discrepancy can be explained by the fact that the phenomenological theory [33], which links χ_G with the electrically controlled effective birefringence Δn_{eff}^E , defined through the Bessel function in [35], assumes the presence of one critical electric field E_c in FLC, which is not true for the case of FiLC. Also, for FiLC, due to the much more complex molecular packaging [14], the one-constant approximation for elastic modulus, which is used in [33], may not be observed. By normalizing the experimental data for K_{kerr} to such strongly temperature-dependent parameters of the FiLC included in eq. (12) as spontaneous polarization P_s (Fig. 1, a) and rotational viscosity γ_ϕ (Fig. 5, b), it is possible to get the parameter G_{inv} :

$$G_{\text{inv}} = K_{\text{kerr}} \frac{\gamma_\phi^2}{P_s^2}. \quad (13)$$

The dependence of the parameter G_{inv} on the time $\tau_{0.1-0.9}^{\text{off}}$ of free relaxation of the helix structure is shown in Fig. 7. It is approximated with acceptable accuracy by an equation $G_{\text{inv}} = A \cdot \tau_{0.1-0.9}^{\text{off}^2}$, in agreement with eq. (12). Thus, the quadratic relationship between the orientational Kerr-effect coefficient and the time of free relaxation of the helical structure τ^{off} is observed.

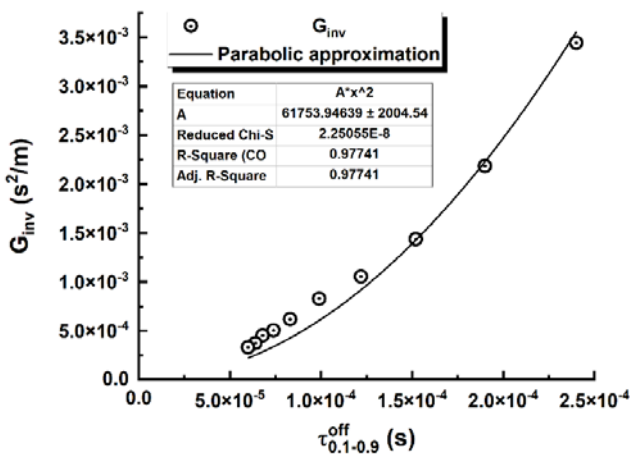


Fig. 7. The dependence of parameter G_{inv} on the time of free relaxation of the helix structure $\tau_{0.1-0.9}^{\text{off}}$ for **FerriLCM-1**

Conclusion

The effect of the molecular structure of two ferroelectric liquid crystal mixtures, **FerriLCM-1** and **FerriLCM-2**, on their properties has been investigated. It was found that the absence in **FerriLCM-2** of chiral dopant **SS-LACT-4**, with the same spontaneous polarization sign but an opposite handedness to the dopant **FODTA-6**, which present in both mixtures, resulted in a decrease in several parameters. Specifically, the spontaneous polarization P_s decreased by 33 %, electro-optical response times $\tau_{0.1-0.9}^{\text{on}}$ and $\tau_{0.1-0.9}^{\text{off}}$ decreased by 68 % and 82 % respectively, the rotational viscosity γ_ϕ decreased by 29 %, the electric susceptibility χ_G decreased by 82 % and the orientational Kerr-effect coefficient K_{kerr} decreased by 87 %. However, the tilt angle of the liquid crystal director in smectic layers slightly increased by 4 % and the critical electric fields E_{cp1} and E_{cp2} significantly increased by 3.8 times and 3.2 times, respectively. The quadratic relationship between the orientational Kerr-effect coefficient K_{kerr} and the free relaxation time of the helix structure $\tau_{0.1-0.9}^{\text{off}}$ in polar smectic phases was discovered. As the time for the relaxation process $\tau_{0.1-0.9}^{\text{off}}$ decreases, K_{kerr} also decreases. This observation implies that higher control voltages may be necessary to achieve the desired phase shift in the liquid crystals with microsecond response times.

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