

Dielectric Properties of Liquid Crystals in Polycapillary Matrices

B. A. Belyaev^{a,*}, N. A. Drokin^a, M. A. Kumakhov^b, and V. F. Shabanov^a

^a *Kirensky Institute of Physics, Siberian Branch, Russian Academy of Sciences,
Akademgorodok 50, Krasnoyarsk, 660036 Russia*

* *e-mail: belyaev@iph.krasn.ru*

^b *Institute of X-Ray Optics, ul. Chasovaya 28, Moscow, 125315 Russia*

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Abstract—This paper reports on the results of investigations into the dielectric properties of liquid crystals embedded in polycapillary matrices and describes a technique for their measurement. It has been revealed that the chemical structure of the rigid core and the length of mobile alkyl groups of liquid-crystal molecules of the alkylnobiphenyl group substantially affect the equilibrium configuration of the liquid-crystal director in capillaries. The reorientation of liquid-crystal molecules embedded in capillaries in the nematic phase under the influence of an external magnetic field has been investigated.

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1. INTRODUCTION

It is well known that liquid crystals have been widely used in various display systems. However, at present, the possibility of using liquid crystals as active media in various constructions of controllable microwave microelectronic devices has been studied intensively. In particular, electrically tunable liquid-crystal phase shifters, which exhibit good characteristics in the centimeter and millimeter wavelength ranges, have been developed in recent years [1, 2]. It is important to note that, in the millimeter range, liquid-crystal tunable devices compare favorably not only with semiconductor devices but also with those based on ferromagnets which operate in unacceptably strong magnetic fields [3].

The ability of liquid-crystal molecules to orientate in contact with any surface is the well-known fact. It is also known that the character of the orientation depends not only on the particular liquid crystal and the material of the medium in contact with it but also, to a large extent, on the quality of the treatment of the contact surface, i.e., the presence of microscopic irregularities, scratches, and pores on the surface. In this case, the molecules under the action of external magnetic and electric fields can be reoriented and then, after the removal of the external action, recover their initial state. It should be noted that the polarization of liquid-crystal molecules is substantially affected by the volume and shape of the cell containing the liquid crystal, as well as by the temperature, because it has a significant effect on the degree of anchoring of molecules.

Taking into account the fact that, in any device, the liquid crystal is always located in a cell that is prepared from a particular material and, as a rule, has a small volume, the determination of the initial orientation of molecules in the cell and the study of specific features of their polarization under the action of external fields is an important problem of modern physics. One of these complex problems, which have attracted the attention of many researchers, is associated with the investigation of the mechanism of polarization of liquid crystals embedded in porous matrices and various composite structures. In this case, the objects of investigations include, in particular, porous glassy media based on borosilicate glasses with interconnected and randomly oriented pores [4–7], zeolites [8, 9], compounds of the aerogel type [10, 11], and liquid-crystal droplets encapsulated into polymers [12, 13].

Investigations of these complex media containing liquid crystals have revealed that, as a rule, the rate of polarization of liquid crystals decreases as a result of the strong anchoring of molecules to walls and, what is more important because of the smallness of the liquid-crystal volume in the droplet or pore. It should be noted that the main problem in experimental investigations of disordered porous media is the determination of the equilibrium configuration of the “director” of liquid-crystal molecules in pores and the recording of the character of its motion under the action of external electric or magnetic fields. As is known, this problem is difficult to solve with the use of optical methods of investigation; however, such problems can be rather reliably solved using electrical measurements at low, high, and ultrahigh frequencies.

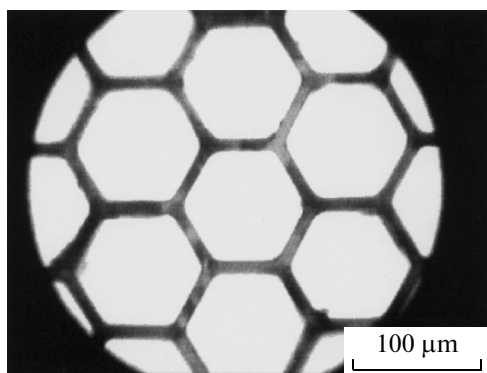


Fig. 1. Photograph of the region of the polycapillary matrix with a capillary size of $\sim 100 \mu\text{m}$.

In particular, the dielectric characteristics and anisotropic properties were studied for liquid crystals embedded in matrices with parallel cylindrical pores, for example, in Anapore aluminum oxide membranes [14–17] and polymer films in which micron and sub-micron pores were produced under irradiation with heavy ions [17, 18]. It was demonstrated that, usually, in untreated Anapore membranes, the liquid-crystal molecules are ordered parallel to the pore walls. However, the chemical modification of the pore surface, for example, with aliphatic acids ($\text{C}_n\text{H}_{2n+1}\text{COOH}$), makes it possible to provide a radial ordering of molecules. A result, investigations of untreated and chemically treated pores allow one to measure permittivities for the orientation of liquid-crystal molecules along the high-frequency electric field (ϵ_{\parallel}) and in the orthogonal direction (ϵ_{\perp}). This provides means for determining the dielectric anisotropy $\Delta\epsilon = \epsilon_{\parallel} - \epsilon_{\perp}$ at the measurement frequency. These investigations, in particular, have demonstrated that, in the low-frequency range for both permittivity components, an additional dispersion region arises, which leads to an increase in the anisotropy, and that an increase in the frequency results in an inversion of the sign of the dielectric anisotropy, which is characteristic of liquid crystals [16].

It is obvious that matrices with parallel micropores are objects convenient for studying the mechanism of anchoring and ordering of liquid-crystal molecules in the bulk of pores and on their surface. However, the possibilities for using these media in practice, for example, for the design of controllable microwave devices are limited because the processes of reorientation of liquid-crystal molecules in response to external electric or magnetic fields are hindered in pores with so small sizes. In this respect, the study of the dielectric properties of liquid crystals embedded in regular polycapillary matrices so that the technology of their manufacturing makes it possible to vary the capillary

diameter over a very wide range is of considerable interest. It is clear that an increase in the capillary diameter allows one to trace the corresponding decrease in the critical magnetic and electric fields of reorientation of molecules (Fréedericksz fields) due to the increase in the “bulk” liquid-crystal phase as compared to the surface phase. The present work is devoted to these investigations. The main purpose of this work is to determine the equilibrium configuration of the liquid-crystal director and to study the processes of dipole reorientation of liquid-crystal molecules embedded in polycapillary matrices.

2. SAMPLE PREPARATION AND EXPERIMENTAL TECHNIQUE

Samples of porous matrices used for embedding liquid crystals were prepared by transverse cutting of glass tubes filled with thin-film glass capillaries. The technology for manufacturing these polycapillary systems was developed for producing original lenses that makes it possible to effectively focus X-ray radiation [19]. The developed technology allows one to prepare regular polycapillary systems with capillary diameters varying from several fractions of a millimeter to sub-micron sizes; in this case, the cross section of capillaries, as a rule, has the form of a regular hexahedron.

In our work, we used porous matrices in the form of disks $\sim 10 \text{ mm}$ in diameter and $\sim 1 \text{ mm}$ in thickness, in which the diameters of through pores were equal to 100, 10, and 5 μm . As an illustration, the photograph of the region of the porous matrix with capillary sizes of 100 μm is displayed in Fig. 1. It can be seen from this figure that the matrix has a regular porous structure and that the capillaries have the form of a regular hexahedron. The relative pore concentration, which evidently characterizes the coefficient of filling of the porous matrix with a liquid crystal, was determined by the direct calculation of the ratio between the area of capillary holes and the total area of the matrix and was $v = 0.8$ for the matrices with pore sizes of 100 and 10 μm and $v = 0.45$ for the matrices with a pore size of 5 μm .

In this work, we measured the temperature dependences of the permittivity of nematic liquid crystals embedded in the porous matrices. The liquid crystals belonged to the series of alkylcyanobiphenyls $n\text{CB}$ ($n = 5\text{--}8$). In this series, the 8CB liquid crystal with the longest alkyl tail for which there exists a smectic liquid-crystal phase in the temperature range 10.9–33.5°C is worthy of notice. Moreover, we investigated the following liquid crystals: *trans*-4-pentyl-1-(4-cyanophenyl)cyclohexane (5PCH), 4-(4'-pentyl-bicyclo[2.2.2]octane)phenyl (5BCO), and *trans*-4-propyl-(4-cyanophenyl)cyclohexane (3PCH). The

dipole moment of all the aforementioned compounds is determined by the cyano group ($C\equiv N$) located on the one side of the rigid molecular core, whereas the mobile methylene fragments (alkyl groups) are located on the other side of the core. It should be noted that the rigid core of the liquid-crystal molecules of the nCB series consists of two phenyl rings, which are linked together along one axis and whose planes are rotated by some angle with respect to each other. The molecular cores in the 3PCH and 5BCO liquid crystals differ from those in the nCB compounds only by replacement of one of the phenyl rings by the cyclohexane and bicyclooctane fragments, respectively. The 3PCH liquid-crystal compound belongs to monotropic nematic liquid crystals. This compound is monotropic because the transition to the ordered nematic phase occurs at the temperature $T = 46^\circ\text{C}$ only from the isotropic state preliminarily prepared through heating of the compound to higher temperatures. The study of this compound is of importance, because its alkyl tail is ultimately short.

Before filling pores with liquid crystals, the polycapillary matrices were heated under vacuum for 2 h at a temperature of 90°C . The pores were filled with the liquid crystal in the isotropic phase, i.e. at temperatures higher than the “nematic–isotropic liquid” transition temperatures T_{NI} . After the filling of pores, the residues of the liquid crystals were removed from the surface of the matrices with the use of paper filters and the prepared samples were held at temperatures $T > T_{NI}$ for 12 h. A thin indium foil playing the role of plates of a parallel-plate capacitor was attached to the end surfaces of matrices. The sample thus prepared was fixed between spring contacts located on a miniature holder of a sensor, which was placed in a thermostat positioned between poles of an electromagnet, which made it possible to produce the magnetic field $H = 16$ kOe. The orientation of the external magnetic field with respect to the direction of the axes of cylindrical pores was changed by rotating the holder of the sensor with the sample under investigation. The field was parallel (H_{\parallel}) in one case and perpendicular (H_{\perp}) in the other case with respect to the direction of the pore axes.

The rate of change in the temperature in the course of experimental investigations was equal to $8\text{--}10^\circ\text{C/h}$. In the thermostating regime, the temperature was maintained accurate to within $\sim 0.3^\circ\text{C}$. The temperature control or a specified rate of change in the temperature was provided using a program-controlled thermal unit, which automatically regulated the temperature and feed of air used for blowing the measuring sensor with the sample.

The dielectric measurements were performed at a frequency of 1 kHz on a specially designed automatic

setup based on a digital E7-8 LCR meter [20]. In this case, the capacitive sensor was connected according to a four-wire circuit, which excluded the influence of connecting conductors on the results of the measurements. In the course of experiments, we investigated the temperature dependences of the capacitance of the sample C_x and its loss tangent $\tan\delta$. As is known, the capacitance C_x is proportional to the real component of the permittivity of the sample under investigation, and the loss tangent $\tan\delta$ is proportional to the imaginary component of the permittivity. It should be noted that the capacitance C_x was measured initially for the empty porous matrix and then for the matrix filled with the liquid crystal. In this case, the absolute accuracy in the determination of the capacitance was no worse than ~ 0.05 pF and the absolute accuracy in the determination of the loss tangent $\tan\delta$ was no worse than ~ 0.001 .

The technique for determining the equilibrium orientation of the director of the liquid crystal embedded in the pores consists in comparing the behavior of the observed temperature dependences of the quantities C_x and $\tan\delta$ in the region of the phase transition from the isotropic state to the nematic state of the samples under investigation with the corresponding dependences obtained for the homogeneously ordered liquid crystals in a continuous volume. This comparison allows one to qualitatively determine the equilibrium orientation of the molecules with respect to the pore walls and to evaluate the degree and character of reorientation of the director of molecules under the action of external electric or magnetic fields.

3. RESULTS AND DISCUSSION

The performed experiments have revealed that the loss tangents $\tan\delta$ for all samples of the empty porous matrices under investigation did not exceed 0.001 and almost did not increase after the filling of the matrices with the liquid crystals. In this respect, much attention in our work was focused on the temperature measurements and the analysis of the dependences of the capacitance $C_x(T)$, which, as was noted above, reflects the behavior of the real component of the complex permittivity of the samples. It is important to note that the direction of the polarization of the high-frequency electric field in the samples coincides with the direction of the capillary axes. Therefore, the capacitance of the measured sample is maximum for the orientation of the director of the liquid-crystal molecules along the capillaries and minimum for the orthogonal orientation of the director (according to the permittivities ϵ_{\parallel} and ϵ_{\perp}).

Let us first consider the dependences $C_x(T)$ for the polycapillary matrices filled with the 8CB liquid crys-

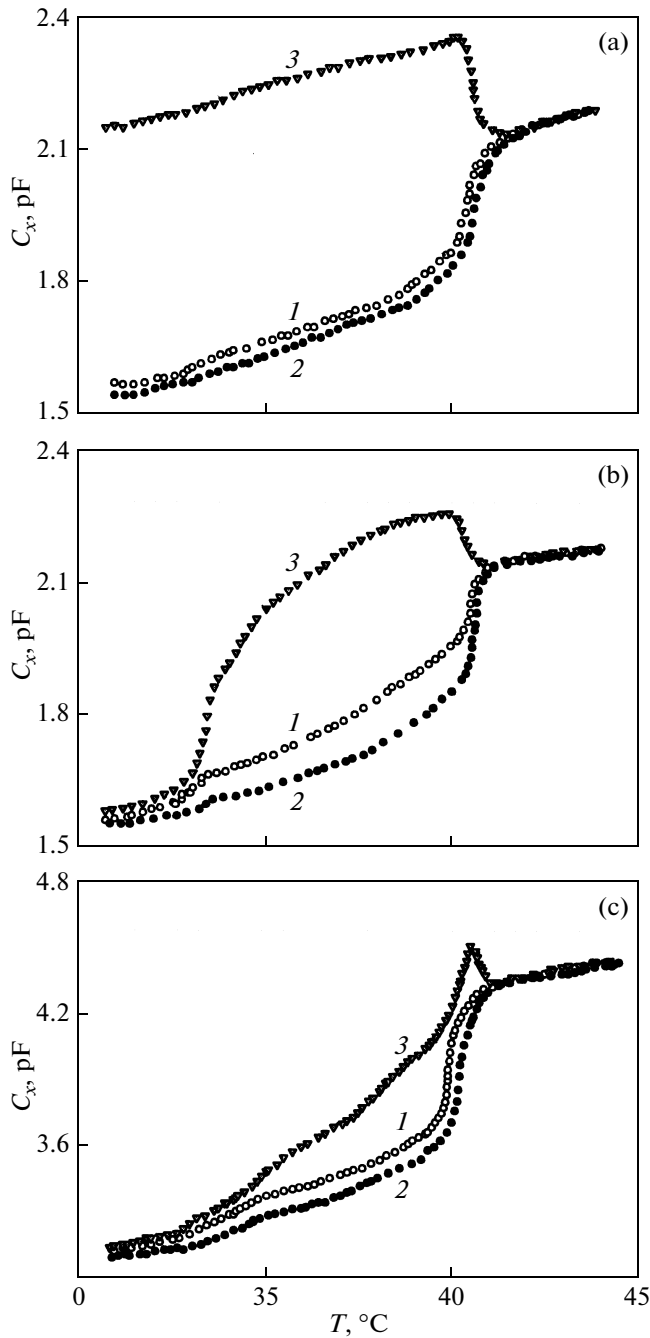


Fig. 2. Temperature dependences of the capacitance of the measuring capacitors based on the matrices with pore diameters of (a) 100, (b) 10, and (c) 5 μm filled with the 8CB liquid crystal (1) in the absence of the magnetic field and in the magnetic fields (2) H_{\perp} and (3) H_{\parallel} .

tal (Fig. 2), for which, as was noted above, the molecules have the longest alkyl tail. It can be seen that, in the capillaries 100 μm in size (Fig. 2a), an increase in the temperature to the temperature of the phase transition from the nematic liquid-crystal state to the isotropic liquid $T_{NI} = 40.5^{\circ}\text{C}$ leads to a monotonic increase in the capacitance of the sample in the pres-

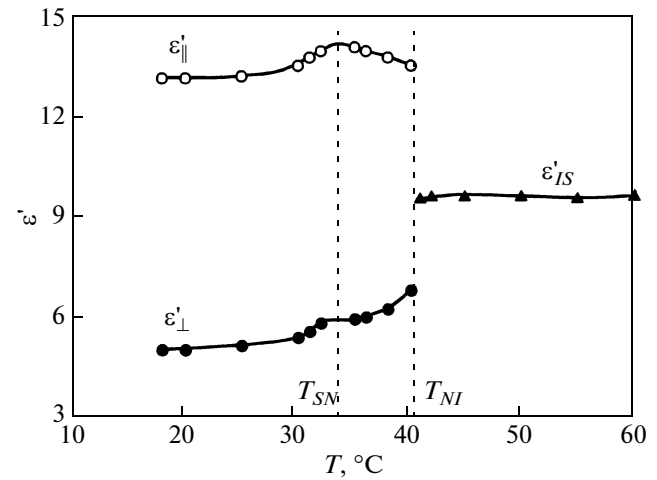


Fig. 3. Temperature dependences of the longitudinal ε_{\parallel} and transverse ε_{\perp} components of the static permittivity for the 8CB liquid crystal in the smectic ($T < T_{SN}$), nematic ($T_{SN} < T < T_{NI}$), and isotropic ($T > T_{NI}$) phases.

ence of the orienting magnetic field applied parallel H_{\parallel} and perpendicular H_{\perp} to the capillary axes (curves 2, 3) and in the absence of the field (curve 1). In this temperature range, the capacitance $C_x(T)$ of the sample is higher for the magnetic field H_{\parallel} orienting the director of the liquid-crystal molecules in the direction of the polarization of the high-frequency electric field and lower for the magnetic field H_{\perp} orienting the director of the liquid-crystal molecules in orthogonal direction with respect to the polarization of the electric field. However, the fact that the capacitance of the sample is low in the absence of the field and insignificantly decreases in the orthogonal field H_{\perp} indicates that, in the initial state ($H = 0$), the director of the liquid-crystal molecules is oriented almost perpendicular to the capillary axes.

It should be noted that the behavior of the curves $C_x(T)$ is in rather good agreement with the known temperature dependences of the permittivities $\varepsilon_{\parallel}(T)$ and $\varepsilon_{\perp}(T)$ [21] for the oriented bulk sample of the 8CB liquid crystal (Fig. 3). In Fig. 3, the dashed lines indicate the temperatures of the phase transitions of the liquid crystal from the smectic state to the nematic state T_{SN} and from the nematic state to the isotropic state T_{NI} . It can be seen that the permittivities ε_{\parallel} and ε_{\perp} in the range of the temperature T_{SN} remain almost unchanged, as well as the capacitances of the sample under investigation in the orienting fields H_{\parallel} and H_{\perp} (Fig. 2a). However, upon the transition of the liquid crystal to the isotropic state, the dependences $\varepsilon_{\parallel}(T)$ and $\varepsilon_{\perp}(T)$ exhibit pronounced jumps, which for the liquid crystal in the porous matrices are also observed

in the dependences $C_x(T)$, but these jumps are slightly smeared.

It is important to note that an increase in the temperature is accompanied by an increase in the capacitance C_x of the sample of the porous matrix (with a capillary size of 100 μm) containing the 8CB liquid crystal (Fig. 2a) both below and above the transition temperature T_{NI} . This can be explained by the strong temperature dependence of the energy of anchoring of the liquid-crystal molecules (to the pore walls), in which, as was noted above, the long axes are predominantly oriented perpendicular to the capillary axes; i.e., they are ordered either radially or in a planar polar manner [22]. The results of the performed investigations, including those of the samples with smaller capillary sizes (Figs. 2b, 2c), allow us to make the inference that the increase in the temperature leads to a decrease in the energy of anchoring of the liquid-crystal molecules to the pore walls.

Actually, as the temperature increases to approximately 36.5°C, the initial state of the liquid-crystal molecules in the sample with a pore diameter of 10 μm (Fig. 2b) remains almost unchanged when the magnetic field is applied in any direction. This means that the strength of the magnetic field used in the experiment ($H = 16$ kOe) is insufficient for overcoming the energy of anchoring of the liquid-crystal molecules to the capillary walls. However, with an increase in the temperature above 36.5°C, the capacitance C_x in the field H_{\parallel} increases rapidly (curve 3) most likely due to the reorientation of the molecular axes along the field, i.e., in the direction of the capillary axes. The process of reorientation of the liquid-crystal molecules with an increase in the temperature is similar to the Fréedericksz transitions observed in electric or magnetic fields.

It is obvious that, with a decrease in the capillary diameter, the volume of the liquid-crystal molecules located in the “near-surface” layer increases with respect to the total volume of the liquid crystal. This results in a proportional increase in the fraction of the energy responsible for the anchoring of molecules to the capillary surface. Therefore, in the sample with a pore diameter of 5 μm (Fig. 2c, curve 3), the reorientation of the molecules is observed at a higher temperature of approximately 40°C (in the vicinity of the temperature of the phase transition of the liquid crystal to the isotropic liquid). Similar dependences $C_x(T)$ and the revealed (in our work) regularities of their change with a decrease in the capillary diameter in the samples occur for the entire series of alkylcyanobiphenyls with shorter alkyl tails $n = 7$ and 6 under investigation.

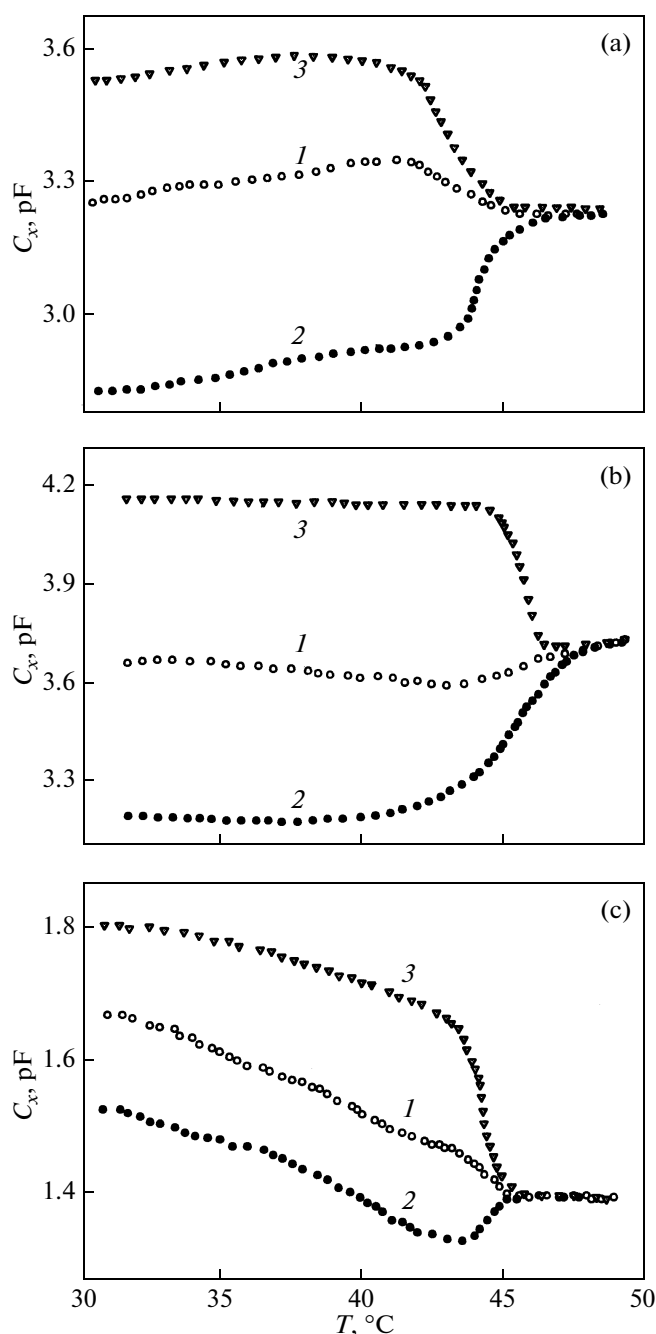


Fig. 4. Temperature dependences of the capacitance of the measuring capacitors based on the matrices with pore diameters of (a) 100, (b) 10, and (c) 5 μm filled with the 3PCH liquid crystal (1) in the absence of the magnetic field and in the magnetic fields (2) H_{\perp} and (3) H_{\parallel} .

However, for the 3PCH liquid crystal with the shortest alkyl tail ($n = 3$), the measured dependences $C_x(T)$ (Fig. 4) are characterized by substantial differences. In particular, relatively insignificant changes in the capacitance with an increase in the temperature are observed in the absence of the constant magnetic field (curves 1). In this case, for the sample with a cap-

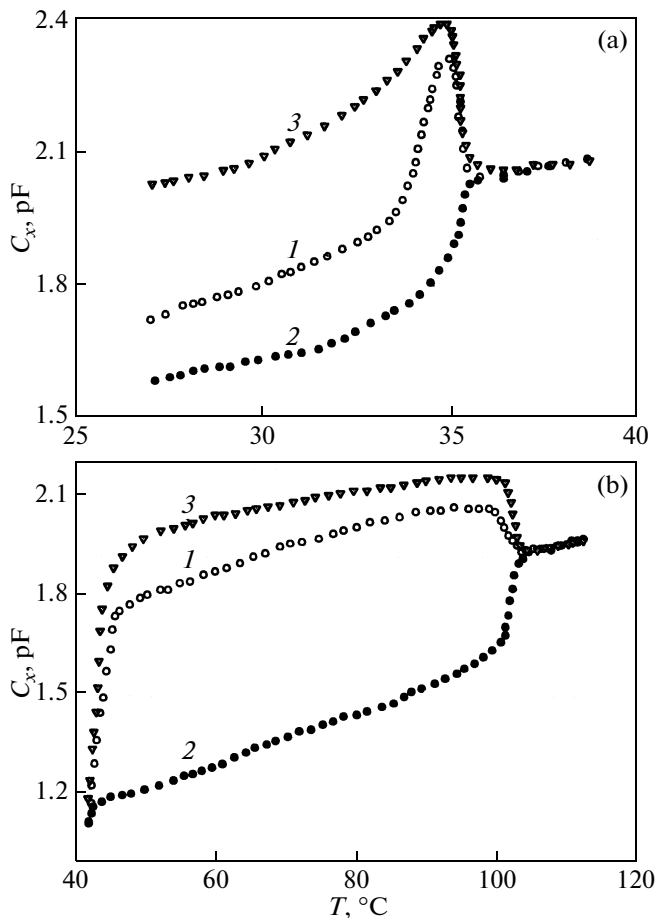


Fig. 5. Temperature dependences of the capacitance of the measuring capacitors based on the matrix with a pore diameter of 100 μm filled with the (a) 5CB and (b) 5BCO liquid crystals (1) in the absence of the magnetic field and in the magnetic fields (2) H_{\perp} and (3) H_{\parallel} .

illary diameter of 100 μm (Fig. 4a), the measured capacitance first increases monotonically and then decreases in the region of the transition of the liquid crystal to the isotropic liquid. By contrast, for the sample with a capillary diameter of 10 μm (Fig. 4b), the capacitance $C_x(T)$ first decreases and then increases. Finally, for the sample with a capillary diameter of 5 μm (Fig. 4c), the capacitance $C_x(T)$ decreases monotonically to the temperature of the transition of the liquid crystal to the isotropic liquid.

It should be noted that, in the nematic liquid-crystal state, all capacitances of the sample with a pore diameter of 10 μm (Fig. 4b) in the absence of the constant magnetic field (curve 1) lie approximately at the center of the range between the values measured for the perpendicular (curve 2) and parallel (curve 3) directions of the field with respect to the sample axis. This can indicate either the absence of the spontaneous ordering of molecules or the orientation of their

director in the initial state at an angle of approximately 45° with respect to the capillary axis. The fact that, in the absence of the magnetic field, the capacitance of the samples with an increase in the capillary diameter approaches the values of $C_x(T)$ observed in the parallel magnetic field (Fig. 3a) and, vice versa, with a decrease in the capillary diameter (Fig. 3c), in principle, does not contradict the assumption regarding a disordered state of the mesophase. In this case, there should arise regions with a spontaneous orientation of dipole moments of the molecules with directions either parallel or perpendicular to the pore walls, which should occupy some small volume of the liquid crystal in the vicinity of the pore walls. However, in our opinion, the second assumption that the liquid-crystal director in the initial state is oriented at an angle of 45° with respect to the capillary axis is most probable. In this case, a change in the capillary diameter leads either to a decrease or to an increase in this angle.

It could be expected that this behavior of the 3PCH liquid crystal in the pores is a consequence of the anomalously short alkyl tail of its molecules. However, taking into account that, in this compound, the rigid core of the molecule contains the cyclohexane fragment in contrast to the phenyl rings in the liquid crystals of the $n\text{CB}$ series, it is of interest to compare the dielectric properties of the 5CB and 5BCO liquid crystals, which have alkyl chains with the same length $n = 5$ but different molecular cores (the phenyl rings in the 5CB liquid crystal and the bicyclooctane fragment in the 5BCO liquid crystal). The results of the measurements of these compounds embedded in pores with a diameter of 100 μm are presented in Fig. 5.

It can be seen from Fig. 5a (curve 1) that, in the absence of the field H , the capacitance $C_x(T)$ of the 5CB liquid crystal in the pores at $T < T_{NI}$ initially increases and then decreases. Therefore, in the vicinity of the phase transition point the long axes of molecules oriented parallel to the pore walls cannot be fixed in this position and tend to be reoriented in a partially planar polar or radial state. A similar tendency is also observed under the action of the magnetic field applied along the pore axis (H_{\parallel}) (Fig. 5a, curve 2). As can be seen from Fig. 5a (curve 3), the external field H_{\perp} at $T < T_{NI}$ results in a decrease in the capacitance $C_x(T)$ or, in other words, additionally orients the long molecular axes perpendicular to the pore walls.

The corresponding results of the measurements of the dielectric properties of the 5BCO liquid crystal are presented in Fig. 5b. It can be seen from this figure that, even in the absence of the external magnetic field, the capacitance $C_x(T)$ upon the transition from the isotropic state to the nematic state increases sharply, which suggests a predominant longitudinal

spontaneous orientation of the molecules. Under the action of the external field H_{\parallel} on the sample, the capacitance $C_x(T)$ somewhat increases as a result of the more homogeneous ordering of the molecules along the pore walls. As can be seen, the application of the external field H_{\perp} at $T < T_{NI}$ leads to a considerable decrease in the capacitance $C_x(T)$, which suggests that the liquid-crystal molecules are oriented perpendicular to the pore walls. However, a further decrease in the temperature is accompanied by a drastic decrease in the capacitance $C_x(T)$ both in the absence of the external magnetic field and in the case of its direction along the capillary axes. This implies that, at low temperatures, the liquid-crystal molecules are spontaneously oriented predominantly in an axial manner and the magnetic field used in the experiment with the strength $H = 16$ kOe is insufficient for overcoming the anchoring energy of the molecules. It should be noted that the energy barrier W_H , which is overcome by the magnetic field H , can be easily estimated from the expression [23]

$$W_H = \frac{1}{2\pi} \gamma h H,$$

where γ is the gyromagnetic ratio and h is the Planck constant. This results in $W_H \approx 2.97 \times 10^{-16}$ erg.

4. CONCLUSIONS

Thus, the analysis of the measured temperature dependences $C_x(T)$ for samples of polycapillary glass matrices filled with liquid crystals demonstrated that the character of the molecular ordering depends not only on the pore sizes but also on the molecular structure of the liquid crystals under investigation. In particular, it was established that the liquid crystals of the alkylcyanobiphenyl series with $n = 5-8$ are predominantly ordered in a homeotropic manner with respect to the pore walls. Possibly, the arising planar polar or radial orientations of the director field are associated with the electrostatic or molecular mechanisms of the interaction of the $-C\equiv N-$ terminal fragments of the molecules with the $=Si=O$ groups forming the surface of the glass capillary. The homeotropic ordering of the molecules is also favored by the steric interactions of fairly long alkyl chains [24].

Moreover, the experiments showed that, most likely, the liquid-crystal molecules with large cyclohexane or bicyclooctane fragments of the cores of the 3PCH and 5BCO liquid crystals are located in the pores either in the disordered state or with the orientation of the director of the molecules at an angle of approximately 45° with respect to the capillary axis; however, in the magnetic fields they can be easily oriented parallel and perpendicular to the pore walls.

This fact undeniably indicates the molecular interaction between the cores of the molecules and the pore walls. It seems likely that the revealed processes of orientation of the liquid-crystal molecules in the polycapillary matrices are associated with the competition of the molecular, electrostatic, and steric interactions of the cores, alkyl chains, and dipole moments with the pore walls.

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