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RESEARCH ARTICLE

Dielectric properties of the system: 4-*n*-pentyloxybenzoic acid–*N*-(4-*n*-butyloxybenzylidene)-4'-methylaniline

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Abstract

Objectives. *Our aim was to study the dielectric properties of the 4-n-pentyloxybenzoic acid–N-(4-n-butyloxybenzylidene)-4'-methylaniline system and reveal how different concentrations of N-(4-n-butyloxybenzylidene)-4'-methylaniline additives affect the dielectric properties of 4-n-pentyloxybenzoic acid.*
Methods. *System properties were investigated using polarization thermomicroscopy and dielcometry.*
Results. *We found that dielectric anisotropy changes its sign from positive to negative at the transition temperature of the high-temperature nematic subphase to the low-temperature one. The anisotropy of the dielectric constant of N-(4-n-butoxybenzylidene)-4'-methylaniline has a positive value and increases as to the system approaches the crystalline phase. The crystal structure of the 4-n-pentyloxybenzoic acid contains dimers formed by two independent molecules due to a pair of hydrogen bonds. The crystal structure of N-(4-n-butoxybenzylidene)-4'-methylaniline contains associates formed by orientational interactions of two independent molecules. 4-n-Pentyloxybenzoic acid dimers (270 nm) and associates of N-(4-n-butoxybenzylidene)-4'-methylaniline (250 nm) proved to have approximately the identical length. Considering the close length values of the structural units of both compounds and the dielectric anisotropy sign, we assume that the N-(4-n-butoxybenzylidene)-4'-methylaniline associates are incorporated into the supramolecular structure of the 4-n-pentyloxybenzoic acid. The specific electrical conductivity of the compounds under study lies between* 10^{-7} *and* 10^{-12} $S \cdot cm^{-1}$*. The relationship between the specific electrical conductivity anisotropy and the system composition in the nematic phase at the identical reduced temperature, obtained between 100 and 1000 Hz is symbatic. However, the electrical conductivity anisotropy values of the system obtained at 1000 Hz are lower compared to those obtained at 100 Hz. At N-(4-n-butoxybenzylidene)-4'-methylaniline concentrations between 30 and 60 mol %, the electrical conductivity anisotropy values are higher than those of the individual component.*

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Conclusions. *A change in the sign of the dielectric constant anisotropy of the 4-n-pentyloxybenzoic acid during nematic subphase transitions was established. We showed that the system has the highest dielectric constant anisotropy value when components have an equal number of moles. Highest electrical conductivity anisotropy values are observed when the concentration of the N-(4-n-butoxybenzylidene)-4'-methylaniline system lies between 30 and 60 mol %.*

Keywords: *liquid crystals, mixtures of nematogens, dielcometry, anisotropy of dielectric constant, anisotropy of electrical conductivity*

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НАУЧНАЯ СТАТЬЯ

Диэлектрические свойства системы: 4-*н*-пентилоксибензойная кислота–*N*-(4-*н*-бутилоксибензилиден)-4’-метиланилин

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Аннотация

Цель. *Изучить диэлектрические свойства системы: 4-н-пентилоксибензойная кислота–N-(4-н-бутилоксибензилиден)-4’-метиланилин. Выявить влияние добавок N-(4-н-бутилоксибензилиден)-4’-метиланилина различной концентрации на диэлектрические свойства 4-н-пентилоксибензойной кислоты.*
Методы. *Свойства системы исследовались методами поляризационной термомикроскопии и диэлькометрии.*
Результаты. *Установлено, что при температуре перехода высокотемпературной нематической субфазы в низкотемпературную диэлектрическая анизотропия меняет свой знак с положительного на отрицательный. Анизотропия диэлектрической проницаемости N-(4-н-бутоксибензилиден)-4’-метиланилина имеет положительные значения и увеличивается по мере приближения к фазовому переходу в кристаллическую фазу. В кристаллической структуре 4-н-пентилоксибензойной кислоты присутствуют димеры, образованные двумя независимыми молекулами за счет пары H-связей. В кристаллической структуре N-(4-н-бутоксибензилиден)-4’-метиланилина присутствуют ассоциаты, образованные за счет ориентационных взаимодействий двух независимых молекул. Отмечена близость длин димеров 4-н-пентилоксибензойной кислоты (270 нм) и ассоциатов N-(4-н-бутоксибензилиден)-4’-метиланилина (250 нм). Учитывая близость длин структурных единиц обоих соединений и знак диэлектрической анизотропии, можно предположить, что ассоциаты N-(4-н-бутоксибензилиден)-4’-метиланилина встраиваются в надмолекулярную структуру 4-н-пентилоксибензойной кислоты. Удельная электропроводность исследуемых соединений лежит в диапазоне 10^{-7}–10^{-12} См·см$^{-1}$. Зависимости анизотропии удельной электропроводности от состава системы для нематической фазы при одинаковой приведенной температуре, полученные на частотах 100 и 1000 Гц, имеют симбатный характер. Однако величины анизотропии удельной электропроводности системы, определенные на частоте 1000 Гц, ниже, чем на частоте 100 Гц. При концентрации N-(4-н-бутоксибензилиден)-4’-метиланилина от 30 до 60 мол. % значения анизотропии удельной электропроводности системы выше, чем для индивидуального компонента.*

Выводы. *Установлена смена знака анизотропии диэлектрической проницаемости 4-н-пентилоксибензойной кислоты при переходе между нематическими субфазами. Показано, что самое высокое значение анизотропии диэлектрической проницаемости система имеет при эквимолярном соотношении компонентов. Наибольшие значения анизотропии удельной электропроводности наблюдаются при содержании в системе от 30 до 60 мол. % N-(4-н-бутоксибензилиден)-4'-метиланилина.*

Ключевые слова: *жидкие кристаллы, смеси нематогенов, диэлькометрия, анизотропия диэлектрической проницаемости, анизотропия удельной электропроводности*

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INTRODUCTION

In addition to individual supramolecular mesogens, mixtures based on classical calamite liquid crystals have attracted a great deal of interest. Doping them with non-mesogenic and liquid crystal substances offers a route to developing new liquid crystal materials. Specific nematogen interactions in nematogens can considerably affect the component orientational ordering, associative state, and other mesophase properties [1–5]. Lack of systematic studies into these interactions inhibits the development of new functional liquid crystal materials with tailored properties that can find use in various areas.

A characteristic feature of materials in a liquid-crystalline state is their anisotropic properties. These include dielectric constant, magnetic susceptibility, and refractive index, among others. Moreover, a detailed study of these characteristics could boost practical applications of certain mesomorphic functional materials. Dielectric constant data and its relationship with the temperature and composition of the system under study are necessary in the development of components for electro-optical devices [6–9].

In most cases, the nematic mesophase of 4-*n*-alkyloxybenzoic acids, which are components of liquid crystal materials used in information displays, has a positive dielectric constant anisotropy [10–13].

The anisotropy value of the dielectric constant could be affected during the molecular design stage of the mesogen due to the introduction of various functional groups. Furthermore, the anisotropy of the dielectric constant can significantly change in liquid crystal compositions and mixtures, depending on the type of intermolecular interactions and the compositions of the mixtures under study [13–19].

In this work, we studied how the composition of the 4-*n*-pentyloxybenzoic acid–*N*-(4-*n*-butyloxybenzylidene)-4'-methylaniline system affects its dielectric properties (Fig. 1).

The system mesomorphic and volumetric properties were studied earlier [20]. The resulting phase diagram of the specified system with a continuous nematic phase has a eutectic equilibrium point at 40.00 mol % 4-*n*-pentyloxybenzoic acid. In this case, for a given ratio of the mixture components, the maximum temperature range of the nematic phase is observed. It should be noted that the phase transition temperature values of nematogens and their mixtures are determined by polarization thermomicroscopy, dilatometry, and dielcometry.

N-(4-*n*-butoxybenzylidene)-4'-methylaniline
(4-AOBMA)

4-*n*-pentyloxybenzoic acid
(5-AOBA)

Fig. 1. Structural formulas of the studied compounds.

EXPERIMENTAL

The individual components of the investigated mixtures **4-AOBMA** and **5-AOBA** of analytical grade were purified by double recrystallization from ethanol followed by evacuation under 1.3 Pa from the isotropic phase to constant weight.

Evacuation helped remove volatile impurities and solvent residues after recrystallization. The purification quality was controlled by the clearing point (temperature of the nematic-isotropic phase transition) (see the table) [21–23], the absence of delamination during the nematic-isotropic phase transition, and the electrical conductivity values. The table introduces the following definitions: Cr–crystalline phase, N–nematic phase, I–isotropic liquid phase.

The phase transition temperatures of the studied compounds (°C)

Compounds	Cr		N		I
4-AOBMA	•	65.0	•	70.0	•
5-AOBA	•	124.0	•	151.0	•

Substance mixtures were prepared using the gravimetric method, homogenized at a temperature above the clearing point, and slowly cooled to complete crystallization, and then grounded in an agate mortar. The studied mixtures had the following 4-*n*-pentyloxybenzoic acid concentrations: 10.00, 20.00, 30.00, 40.00, 50.00, 60.00, 70.00, 80.00, and 90.00 mol %.

The dielectric constants of individual substances and their mixtures were measured using the method of dielcometry (immittance meter E7-15 (LCR meter), *MERATEST*, Russia). A constant magnetic field of 5000 Hz was used to determine the liquid crystal orientation. The cell for measuring the dielectric constant of the author's design, which is a flat capacitor with an area of 0.44 cm^2 and a thickness of 0.25 mm, was thermostated with an accuracy of ±0.1°C. The dielectric constant was determined at a voltage per cell of 1.2 V. The cell was calibrated against toluene, carbon tetrachloride, and benzene of chemically pure grade for spectroscopy. The error in determining the dielectric constant did not exceed 0.7%.

The electrical conductivities of individual substances and their mixtures were measured on an E7-15 immittance meter at 1 kHz and 100 Hz.

The dielectric constant anisotropy (Δε) was calculated as the difference between the values of the dielectric constant measured along the long axes of the molecules (parallel to the director) and the dielectric constant values measured across the long axes of the molecules (perpendicular to the director):

$$\Delta\varepsilon = \varepsilon_{\parallel} - \varepsilon_{\perp}$$

The electrical conductivity anisotropy was calculated as the ratio of the electrical conductivity values measured along the long axes of the molecules (parallel to the director) and the electrical conductivity values measured across the long axes of the molecules (perpendicular to the director):

$$\Delta\sigma = \sigma_{\parallel}/\sigma_{\perp}$$

RESULTS AND DISCUSSION

Since electro-optical effects expose the liquid crystal layer to an electric field, the dielectric constant and electrical conductivity are deemed extremely important parameters. The indicated values were measured for the individual components and their mixtures.

As Fig. 2 demonstrates, the compound **5-AOBA** (4-*n*-pentyloxybenzoic acid) in the nematic phase changes the sign of the dielectric constant anisotropy. The dielectric anisotropy changes its sign from positive to negative when transiting from the high-temperature nematic subphase to the low-temperature subphase. We deem that this experimental result can be explained by the "crystal structure memory" of the compound in the mesophase, which manifests when different nematic subphases are formed from different crystalline modifications of **5-AOBA**.

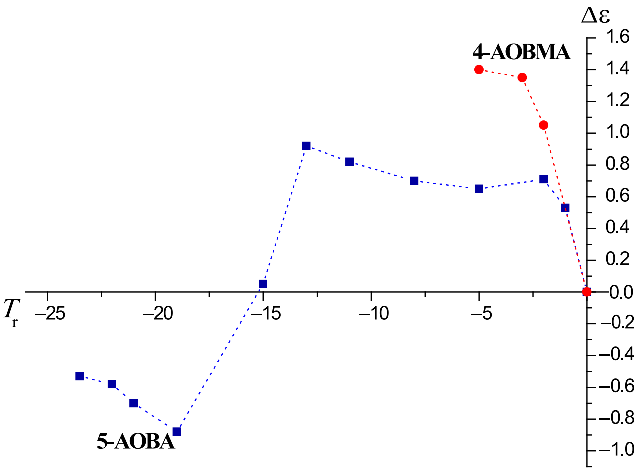

Fig. 2. Dependence of the dielectric constant anisotropy of the individual system components in the nematic phase on the reduced temperature $T_r = T - T_{N\text{–}I}$ (1 kHz frequency). $T_{N\text{–}I}$ – transition temperature from nematic (N) to isotropic liquid (I).

4-*n*-Pentyloxybenzoic acid has two crystalline modifications: triclinic and monoclinic [24]. The triclinic modification has a stacked structure that reminisces the smectogen structure. However, the interplanar distance in the stacks is much larger than that in the smectic phase. This indicates considerably weaker stacking interactions in such packing.

In the aromatic regions of the monoclinic modification, there are no stacked structural elements, but T-shaped ones, the combination of which yields a parquet packing. Such a packing causes weaker interactions among structural elements compared to those of a stacked structure. Another noteworthy observation is the disordering of the carboxyl hydrogen atom by two positions at the oxygen atoms [24, 25].

The crystal structure of 4-*n*-pentyloxybenzoic acid contains dimers formed by two independent molecules due to a pair of hydrogen bonds.

We deem that these crystalline modifications are transformed into nematic subphases that differ in both dielectric and volumetric properties [20]. The mechanism of this phase transition is still elusive. It can only be assumed that the triclinic modification turns into a low-temperature nematic subphase, while the monoclinic modification becomes a high-temperature nematic subphase. This assumption is based on the fact that the low-temperature nematic subphase has a texture that resembles that of smectogens, while the high-temperature nematic subphase has a classic schlieren texture.

The decreased anisotropy value of the dielectric constant when the system approaches the nematic-isotropic phase transition is associated with the loss of orientational ordering when the temperature increases.

The dielectric constant anisotropy of *N*-(4-*n*-butyloxybenzylidene)-4'-methylaniline has positive values and increases as the system phase approaches the crystalline phase. Associates formed due to orientational interactions of two independent molecules are also present in the crystal structure of *N*-(4-*n*-butyloxybenzylidene)-4'-methylaniline [26]. It is worth noting that the lengths of **5-AOBA** dimers and **4-AOBMA** associates are close: 270 nm for **5-AOBA** and 250 nm for **4-AOBMA** (Figs. 3 and 4). This may be due to the fact that the packing of 4-*n*-pentyloxybenzoic acid dimers [20] is denser, which is due to the acoplanarity of *N*-(4-*n*-butoxybenzylidene)-4'-methylaniline molecules.

The greatest value of the dielectric constant anisotropy in the nematic phase is observed when the components have an equal number of moles. Considering the close length values of the structural units of both compounds and the dielectric anisotropy sign, we assume that the **4-AOBMA** associates are incorporated into the supramolecular structure of **5-AOBA**. In the mixed two-phase nematic-isotropic region, the system dielectric anisotropy values do not practically change as the **4-AOBMA** concentration increases and approaches 0.1. The dielectric constant anisotropy of the system increases up to a value of 0.2 at a content of *N*-(4-*n*-butyloxybenzylidene)-4'-methylaniline of 90 mol % (Fig. 5).

In addition to dielectric anisotropy, electrical conductivity is an important property of liquid crystal materials. Thoroughly purified liquid crystals must have

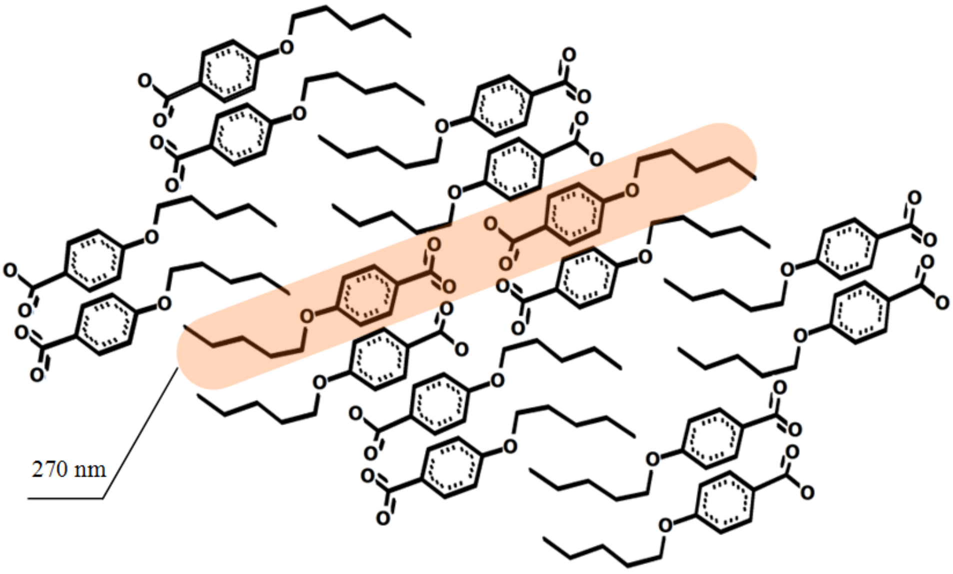

Fig. 3. The crystal packing fragment of 4-*n*-pentyloxybenzoic acid molecules [24] (the molecule long axes are in the plane of the figure).

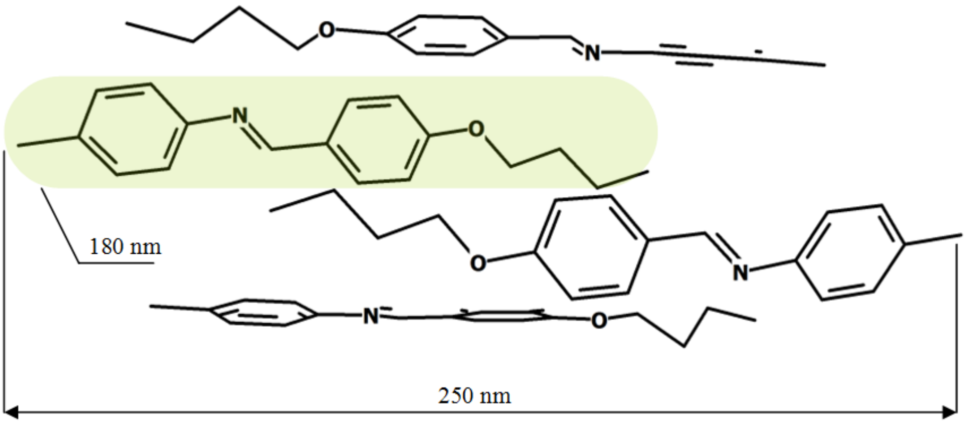

Fig. 4. The crystal packing fragment of *N*-(4-*n*-butyloxybenzylidene)-4'-methylaniline molecules [26] (the molecule long axes are in the plane of the figure).

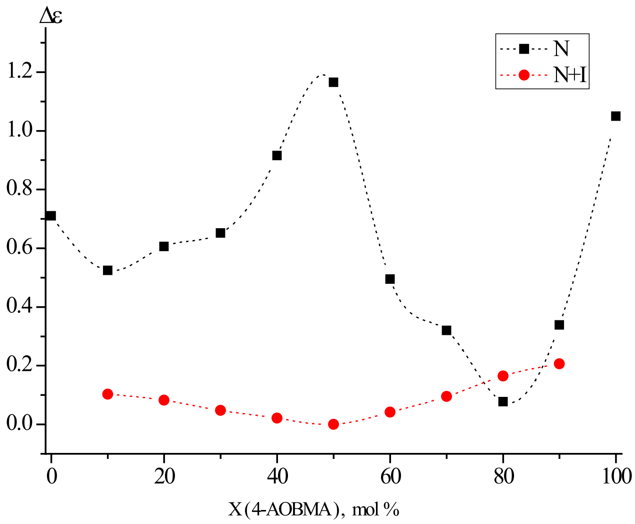

Fig. 5. Dependence of the dielectric constant anisotropy on the mixture composition for the nematic phase (N) at $T_r = -2$°C and nematic-isotropic region (N+I) $T_r = -2$°C ($T_r = T - T_{N+I-I}$), 1 kHz frequency.

extremely low intrinsic electrical conductivities. The specific electrical conductivity of liquid crystals usually ranges between 10^{-7} and 10^{-12} S·cm^{-1}. The investigated nematogens **5-AOBA** and **4-AOBMA** satisfy this requirement. For example, the specific conductivity of **5-AOBA** at 102°C is $3.9 \cdot 10^{-12}$ S·cm^{-1}, whereas the specific conductivity of **4-AOBMA** at 58°C is $1.7 \cdot 10^{-12}$ S·cm^{-1} (both conductivity values are given for the crystalline phase of the compounds).

The electrical conductivity mechanism in liquid crystals is ionic. In this case, the nature of charge carriers can be both intrinsic and impure. The electrical conductivity of mesogens is anisotropic.

Between 100 and 1000 Hz, the electrical conductivity anisotropy of *N*-(4-*n*-butyloxybenzylidene)-4'-methylaniline does not depend on the temperature in the mesophase (Fig. 6). In the 4-*n*-pentyloxybenzoic acid, the anisotropy of specific electrical conductivity sharply increases at the temperature of the phase transition between one nematic subphase to another. In the low-temperature nematic subphase, the electrical conductivity values are slightly higher than those in the high-temperature nematic subphase (Fig. 7). It should be noted that for both the **5-AOBA** acid and the **4-AOBMA** Schiff base, the anisotropy values of the electrical conductivity obtained at 100 and 1000 Hz are quite close.

Analysis results regarding the relationship between temperature and anisotropic electrical conductivity values show that the latter values exceed 1 of **4-AOBMA**. This indicates the presence of regions with a short-range smectic order in the nematic phase of **4-AOBMA**.

Let us consider how the concentration additives of

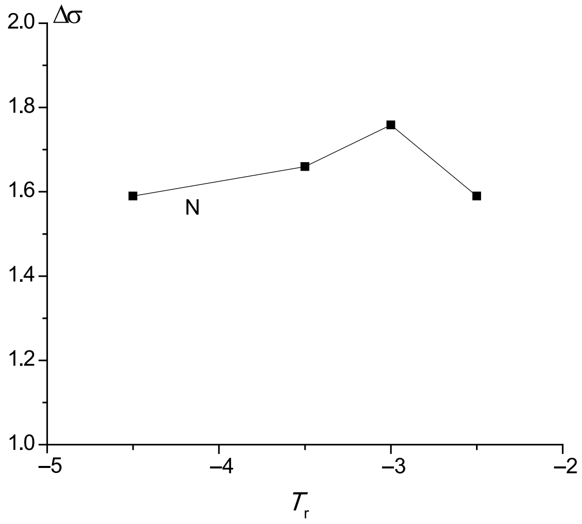

Fig. 6. Dependence of the anisotropy of specific electrical conductivity (Δσ) on the reduced temperature in the nematic phase of *N*-(4-*n*-butyloxybenzylidene)-4'-methylaniline, 1 kHz frequency.

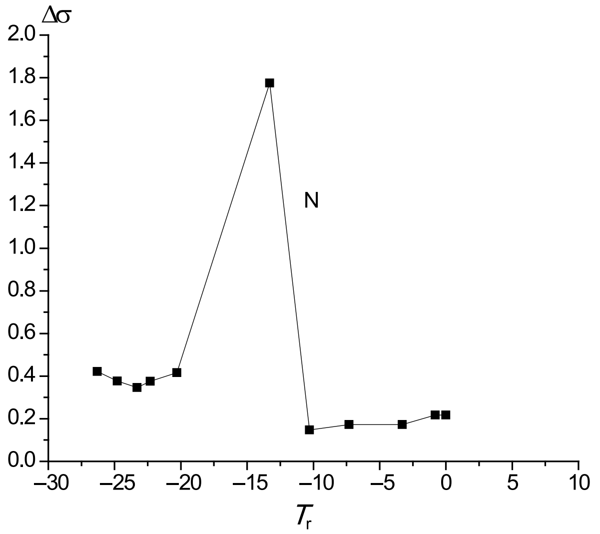

Fig. 7. Dependence of the anisotropy of specific electrical conductivity (Δσ) on the reduced temperature in the nematic phase of 4-*n*-pentyloxybenzoic acid, 1 kHz frequency.

4-AOBMA affect the electrical conductivity anisotropy of **5-AOBA** in the nematic and mixed two-phase nematic-isotropic region.

The relationship between the anisotropy of specific electrical conductivity and the system composition in the nematic phase N at T_r = −2°C, obtained at 100 and 1000 Hz, demonstrate a symbatic character. However, the anisotropy values of the system electrical conductivity at 1000 Hz are lower. The highest anisotropy values of specific electrical conductivity are observed when the system (**4-AOBMA**) concentration ranges between 30 and 60 mol %. It should be noted that, with the indicated compositions, the anisotropy values of the system electrical conductivity exceed those of **4-AOBMA** itself (Fig. 8).

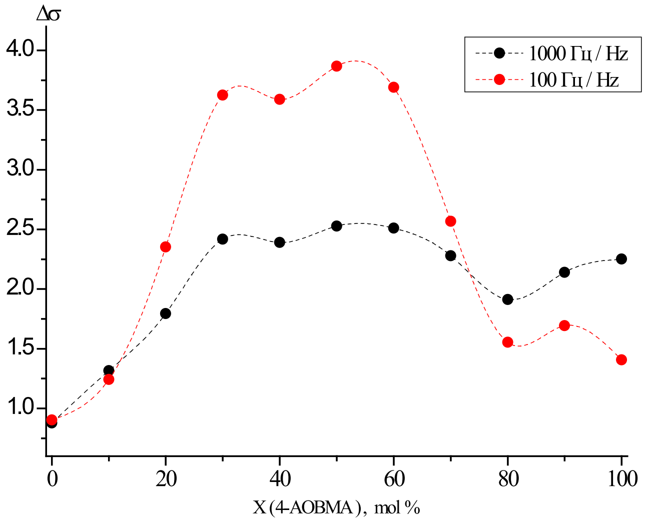

Fig. 8. Dependence of the anisotropy of specific electrical conductivity on the system composition in the nematic phase N at T_r = −2ºC, 100 and 1000 Hz.

A different picture is observed for a mixed, two-phase nematic-isotropic region of the system at 100 and 1000 Hz (Fig. 9), although both dependencies have a symbatic character as in the previous case.

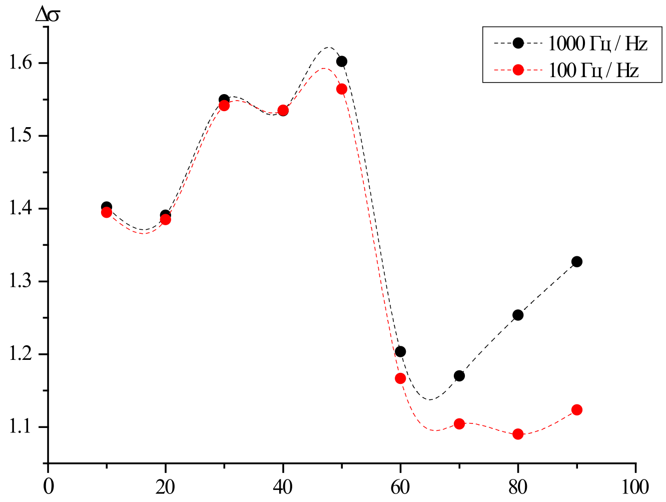

Fig. 9. Dependence of the anisotropy of specific electrical conductivity on the composition of the system in the nematic-isotropic region N+I at T_r = −2ºC, 100 and 1000 Hz.

The anisotropy values of the system electrical conductivity at both frequencies are close compared to those of the previous case. Increasing the concentration of the **4-AOBMA** compound up to 50 mol % and from 70 to 90 mol % causes the anisotropic electrical conductivity of the system to increase. When the system concentration ranges between 50 and 70 mol % **4-AOBMA**, a decrease in the anisotropy of the electrical conductivity of the system is observed.

In conclusion, we note that for concentrations of *N*-(4-*n*-butyloxybenzylidene)-4'-methylaniline ranging between 30 and 80 mol % both in the nematic phase and in the mixed two-phase nematic-isotropic region, the anisotropy values of the specific electrical conductivity increase as the temperature decreases. This result indicates that the degree of orientational ordering increases.

CONCLUSIONS

Using the method of dielcometry, we studied the dielectric properties of the 4-*n*-pentyloxybenzoic acid–*N*-(4-*n*-butyloxybenzylidene)-4'-methylaniline system with a step of 10 mol % in component concentration.

Results revealed that unlike the 4-*n*-pentyloxybenzoic acid–*N*-(4-*n*-butyloxybenzylidene)-4'-methylaniline has higher dielectric constant anisotropy values.

A change in the anisotropy sign of the dielectric constant of 4-*n*-pentyloxybenzoic acid during nematic subphase transitions from negative (for a low-temperature nematic subphase) to positive (for a high-temperature nematic subphase) was established.

The system has the highest dielectric constant anisotropy when components have an equal number of moles, and the highest anisotropy values of specific electrical conductivity at concentrations of *N*-(4-*n*-butyloxybenzylidene)-4'-methylaniline ranging between 30 and 60 mol %.

Acknowledgments

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Authors' contribution

S.A. Syrbu – development of the concept of scientific work, consultation on planning, methodology and implementation of the study, writing the text of the article;

M.S. Fedorov – conducting research, reviewing publications on the topic of the article, processing the experimental data, preparing illustrations;

E.A. Lapykina – conducting research, analysis of literature data;

V.V. Novikov – conducting research, collecting and processing the material.

The authors declare no conflicts of interest.

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