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Refractive and birefringent properties of 4-alkyl-4'-oxycyanobiphenyls at direct and reverse phase transitions

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Refractive and birefringent properties of 4-alkyl-4'-oxycyanobiphenyls at direct and reverse phase transitions

Atila Eren Mamuk^a, Arif Nesrullajev^a, and Prabir K. Mukherjee^b

^aMugla Sıtkı Koçman University, Department of Physics, Laboratory of Liquid and Solid Crystals, Mugla, Turkey;

^bDepartment of Physics, Government College of Engineering and Textile Technology, 12 William Carey Road, Serampore, Hooghly, India

ABSTRACT

Studies of temperature behavior of the mean refractive index, ordinary and extraordinary refractive indices, and birefringence have been carried out for three nematogens. The thermo-morphologic properties of the biphasic regions of the direct *nematic-isotropic liquid* and reverse *isotropic liquid-nematic* phase transitions have been investigated. The 4-n-alkyl-4'-oxycyanobiphenyls ($n = 5, 6, 7$) were objects of our study. Studies have been carried out for the circular cycle as the heating-cooling. Temperature behavior of the order parameter in nematic mesophase and at the *nematic-isotropic liquid* and *isotropic liquid-nematic* phase transition is discussed.

KEYWORDS

Liquid crystals; Refractive properties; Optical birefringence; Order parameter; Phase transitions

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Introduction

Liquid crystals are materials, which exhibit simultaneously partially ordering properties as solid crystals and rheological properties as fluids. Important peculiarity of these materials is large variety of the optical properties [1–7]. Liquid crystals exhibit optical anisotropy and display the mean refractive index, refractive index of the ordinary ray, refractive index of extraordinary ray, birefringence, dichroism, optical activity, etc. Besides, liquid crystalline mesophases can be optically uniaxial or optically biaxial and can display positive optical anisotropy or negative optical anisotropy. Important peculiarity of liquid crystals is also large variety of phase transitions [8–16]. These transitions can be of the first order or second order, characterize various behavior of the order parameter and display various critical effects. Besides, different types of liquid crystalline materials can exhibit various types of enantiotropic or monotropic phase transitions. Such peculiarities of liquid crystals make these materials sufficiently important media for fundamental investigations and technical and technological applications.

Although the nematic-isotropic (N-I) phase transition is one of the most studied phase transition in thermotropic liquid crystals, it is still in considerably current interest. Further there are still no clear answers to some key questions concerning the nature of this phenomenon. One of the key questions is that what makes the N-I transition so weakly first order

CONTACT Arif Nesrullajev ✉ arifnesr@mu.edu.tr ☎ Mugla Sıtkı Koçman University, Department of Physics, Laboratory of Liquid and Solid Crystals, Mugla 48000, Turkey.

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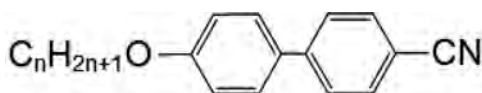


Figure 1. The molecular structure of liquid crystals, examined in this study. 5OCB: $n = 5$; 6OCB: $n = 6$; 7OCB: $n = 7$.

[14]. While research of the optical properties and temperature behavior of the optical parameters for the heating processes of nematic liquid crystals has been going on over the few decades [17–30], information about temperature behavior of the above mentioned optical properties for the cooling processes and for the circular cycle as the *heating-cooling* processes is absent in scientific literature.

In this work, temperature dependences of the refractive indices (n , n_o , n_e) and birefringence (Δn) for the *heating-cooling* circular cycle have been studied. Temperature dependences of the order parameter in large temperature interval and at the direct and reverse phase transitions have been estimated. Three mesogens of the 4- n -alkyl-4'-oxycyanobiphenyls series have been used. We would like to note that such materials, also as the 4- n -alkyl-4'-cyanobiphenyls, have the positive dielectric anisotropy, exhibit liquid crystalline mesophase in large temperature interval and are perspective materials for the twist and supertwist displays, and multimatrix elements.

2. Experimental

2.1. Materials

Three homologues of the 4- n -alkyl-4'-oxycyanobiphenyls series were objects of our investigations. The homologues investigated are, 4- n -pentyl-4'-oxycyanobiphenyls (5OCB), 4- n -heptyl-4'-oxycyanobiphenyls (6OCB), and 4- n -hexyl-4'-oxycyanobiphenyls (7OCB). Molecular structure of the 4- n -alkyl-4'-oxycyanobiphenyls series is presented in Figure 1. 4- n -alkyl-4'-cyanobiphenyls were purchased from Merck and used without further purification.

These liquid crystalline materials are thermally stable and have low viscosity. Besides, these materials exhibit low-temperature nematic mesophase, display enantiotropic phase transitions and have high positive optical anisotropy. The 5OCB, 6OCB, and 7OCB exhibit the following sequence of the phase transitions: *solid crystal* (Cr) \rightarrow *nematic* (N) \rightarrow *isotropic liquid* (I) \rightarrow N \rightarrow Cr. Temperatures of the direct and reverse phase transitions are presented in Table 1.

2.2. Methods and samples

For measurements of the optical refractive and birefringent properties of 5OCB, 6OCB, and 7OCB, the polythermic refractometry setup with Atago 4T Abbe refractometer, Atago digital

Table 1. Temperatures of phase transitions in 5OCB, 6OCB, and 7OCB.

Liquid crystal	Temperatures of phase transitions (K)		
	Cr–N	N–I	I–N
5OCB	321.3	335.9	334.2
6OCB	330.8	343.5	341.4
7OCB	327.0	342.5	340.6

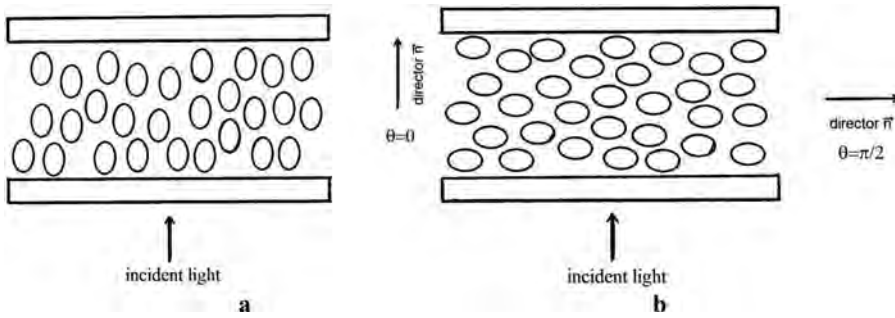


Figure 2. Schematic representation of the homeotropic (a) and planar (b) alignment of N mesophase.

temperature system and thermostat unit has been used. Accuracy for the refractive indices measurements was as 0.1%. Temperature of liquid crystals under investigation was controlled by digital temperature controller with accuracy as ± 0.1 K.

Textures of 5OCB, 6OCB, and 7OCB were studied using the polarizing optical microscopy technique (POM). The thermo-morphologic properties of the biphasic regions of phase transitions in liquid crystals under investigations have been studied by method of the temperature wedge [31], which was modified as the capillary temperature wedge (CTW) device [32, 33]. This device provides the observations of all of the thermic states of liquid crystalline materials in the real scale of time and in a wide temperature range. This device also provides the calculation of the phase transition temperatures and the temperature and linear widths of the biphasic regions with accuracy not less than 10^{-2} K [32–35]. Temperature gradient along the temperature wedge was kept during experiment as 9.30×10^{-4} K $\cdot\mu\text{m}^{-1}$.

For determination of the refractive indexes n_e and n_o , peculiarities of polarizers, and the homeotropic and planar alignment of liquid crystalline materials have been used. In the homeotropic texture, the director of mesophase is oriented perpendicularly to the reference surfaces of the sandwich-cell (Figure 2a). This texture is optically isotropic for light falling perpendicularly to the reference surfaces and is optically anisotropic for light falling parallel to the reference surfaces. In the planar texture, the director of mesophase is oriented parallel to the reference surfaces of the sandwich-cell (Figure 2b). This texture is optically anisotropic for light falling perpendicularly to the reference surfaces and is optically isotropic for light falling parallel to the reference surfaces. Degree of the planar alignment has been examined by the POM and estimated by the optical polarization (OP) degree. The value of the OP degree has been determined as

$$P = \frac{I_{\max} - I_{\min}}{I_{\max} + I_{\min}} \quad (1)$$

Here the I_{\min} is the intensity of light, which is transmitted from sample, placed parallel to the polarizer (or analyzer); the I_{\max} is the intensity of light, which is transmitted from the sandwich-cell, placed under 45° to the polarizer (or analyzer). The degree of the planar alignment of 5OCB, 6OCB, and 7OCB was estimated as $P \approx 0.92$. Degree of the homeotropic alignment was checked on control samples by the POM and estimations of the conoscopic images. In Figure 3, the conoscopic image of the homeotropic alignment in 5OCB, 6OCB, and 7OCB is presented. This conoscopic image characterizes the uniform homeotropic alignment of uniaxial liquid crystalline mesophase.

For investigations of textures of 5OCB, 6OCB, and 7OCB and studies of morphologic properties of the biphasic regions of the direct *nematic-isotropic liquid* and reverse *isotropic liquid-nematic* phase transitions, samples as the sandwich-cells were used. The thickness of liquid

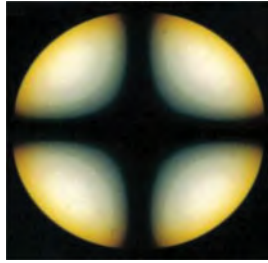


Figure 3. Conoscopic image of the homeotropic alignment in 5OCB, 6OCB, and 7OCB.

crystalline layer placed between the reference surfaces of the sandwich-cell was as $40.0 \pm 0.1 \mu\text{m}$.

3. Results and discussion

In [Figure 4](#), textures of nematic phase, which have been observed in 5OCB, 6OCB, and 7OCB are presented. These textures are the marble type. The boundaries between different uniform regions on these textures are the disclinations. In these textures number singularities also take place. These textures are classic textures for nematic phase in thermotropic liquid crystal [36–38]. As typical peculiarity of nematic texture, in [Figure 4](#) the thread-like formations are observed.

Investigations showed that in 5OCB, 6OCB, and 7OCB linear dependences of the refractive index n vs. temperature observe ([Figure 5](#)). These dependences were repeatable for both the heating and cooling processes. As seen in [Figure 4](#), the $n = n(T)$ dependences for liquid crystals under investigations monotonously decrease with an increase of temperature and monotonously increase with a decrease of temperature. Besides, as seen in this figure, an

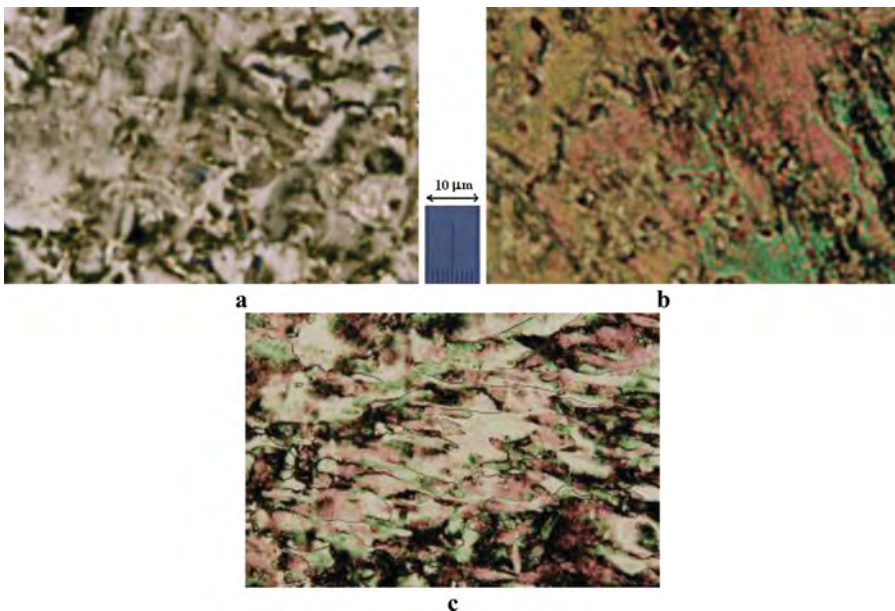


Figure 4. Characteristic textures of nematic mesophase in 5OCB (a), 6OCB, (b) and 7OCB (c).

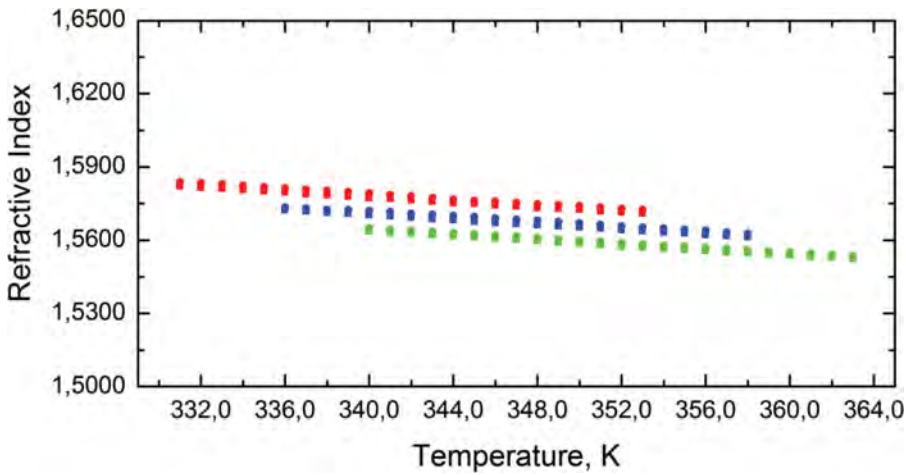


Figure 5. Temperature dependences of the n for 5OCB (in red), 6OCB (in blue), and 7OCB (in green). • – the heating process; ■ – the cooling process.

increase of the alkyl chain in the 4-*n*-alkyl-4'-oxycyanobiphenyls leads to a decrease of values of the refractive index. We would like to note that for $n = n(T)$ dependences, any critical changes in value of the n at the N–I or I–N phase transitions have not been observed. Taking into considerations that n characterizes isotropic optical properties of the media, it is clear that the isotropic refractive properties in 5OCB, 6OCB, and 7OCB are stable and repeatable.

We have investigated the anisotropic refractive properties of 5OCB, 6OCB, and 7OCB and temperature dependences of the ordinary n_o and extraordinary n_e refractive indices for nematic phase, and the n_{iso} refractive index for isotropic phase. As is known, the refractive index n of isotropic optical properties is connected with the refractive indices of anisotropic optical properties as [39–41]

$$n^2 = \frac{n_e^2 + 2n_o^2}{3} \quad (2)$$

In **Figure 6**, the temperature dependences of n_o , n_e , and n_{iso} for 5OCB, 6OCB, and 7OCB during the *heating process* are presented. As seen in this figure, n_o , n_e , and n_{iso} show different behavior. The variation of n_o shows the weak temperature dependence in the region of nematic phase and some increase near the transition to the isotropic liquid. The n_e exhibits strong temperature dependence and a decrease in region of nematic phase. The values of n_o and n_e disappear at the N–I phase transition temperature. After the clearing temperature, 5OCB, 6OCB, and 7OCB exhibit linear temperature behavior of the refractive index n_{iso} in the isotropic liquid state. This effect is connected with a disappearance of the optical anisotropic properties 5OCB, 6OCB, and 7OCB and appearance of the optical isotropic properties in liquid crystalline materials after the clearing temperature.

Temperature dependences of the n_e , n_o , and n_{iso} for 5OCB, 6OCB, and 7OCB during the *cooling process* are also presented in **Figure 6**. As seen in this figure, at the I–N phase transition the optical isotropic properties of liquid crystals under investigations disappeared and the optical anisotropic properties are appeared. But, as seen in **Figure 6**, these transformations for the cooling process observe at lower temperatures, i.e., in 5OCB, 6OCB, and 7OCB for $n_o = n_o(T)$, $n_e = n_e(T)$ and $n_{iso} = n_{iso}(T)$ dependences, the thermal hysteresis takes place. Such thermal hysteresis has been observed for number of physical parameters in various liquid crystalline materials [11, 12, 42–56].

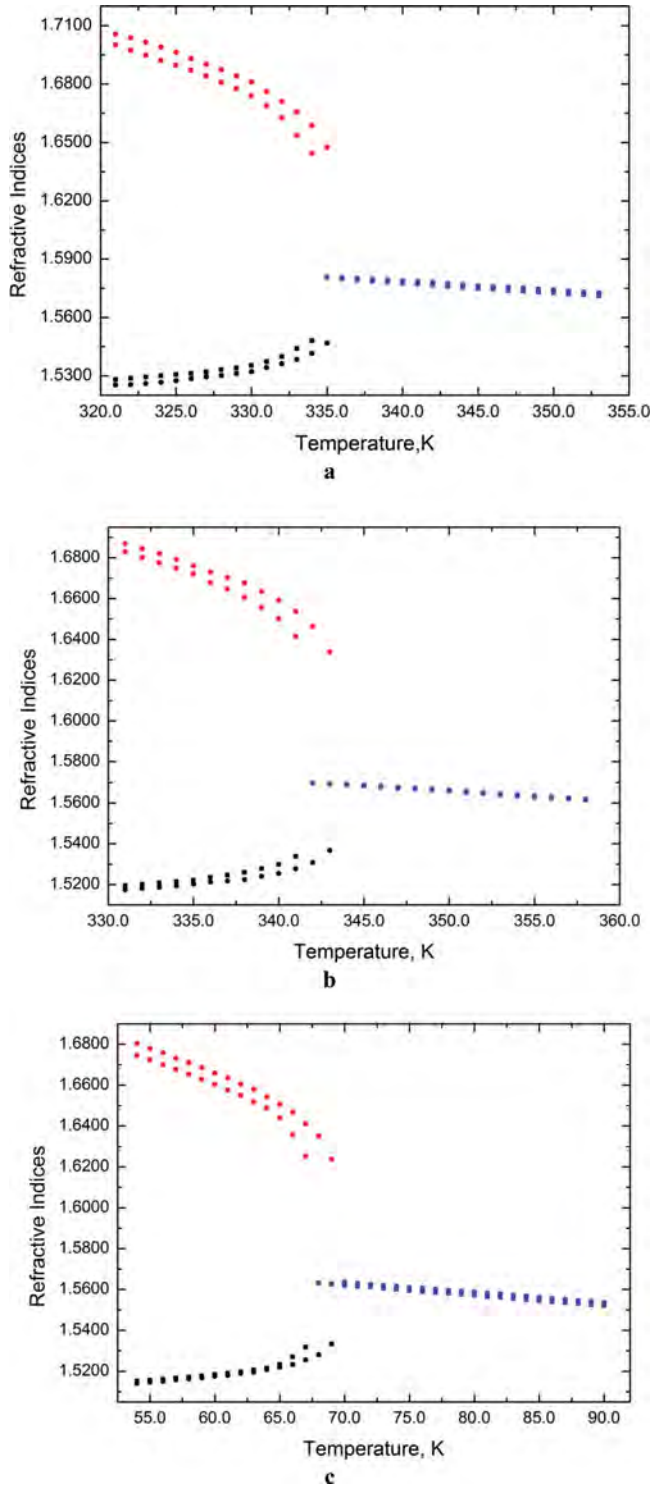


Figure 6. Temperature dependences of the n_e (in red), n_o (in black), and n_{iso} (in blue) for 5OCB (a), 6OCB (b), and 7OCB (c). • – the heating process; ■ – the cooling process.

In terms of the Landau-de Gennes model [1], the thermal hysteresis and T^{**} and T^* temperatures are predicted. The T^{**} and T^* temperatures are the absolute stability of the isotropic liquid and N mesophase, accordingly. The free energy of such model for above mentioned phase transition can be written in the form of

$$F = F_0 + \frac{1}{2}AQ^2 - \frac{1}{3}BQ^3 + \frac{1}{4}CQ^4 \quad (3)$$

Here F_0 is the free energy of isotropic liquid; the Q is the orientational order parameter. For N mesophase $Q \neq 0$ and for isotropic liquid $Q = 0$ takes place. The presence of a cubic term in Eq. (3) makes the transition a first order one. Here A parameter is determined as $A = a(T - T^*)$. The T^* and T^{**} temperatures can be calculated from the form of the free energy given by Eq. 3 as [9, 10, 57, 58]

$$T^* = T_{NI} - \frac{2B^2}{9aC} \quad (4)$$

$$T^{**} = T_{NI} + \frac{B^2}{36aC} \quad (5)$$

Accordingly, temperature widths of the biphasic region can be calculated from Eqs. (4) and (5) as

$$\Delta T = T - T = \frac{B^2}{4aC} \quad (6)$$

Here the T_{NI} is temperature of phase transition between N mesophase and isotropic liquid. At this temperature the free energy of N mesophase and isotropic liquid is zero. The A , a , B , and C are material parameters. Therefore, is clear that the Eqs. (3)–(6) shows that I-N phase transition always exhibits thermal hysteresis and indicates the first order character of the transition.

We would like to note that the extrapolation of the refractive index n_{iso} of 5OCB, 6OCB, and 7OCB into the nematic phase has been made both for heating and cooling cases. The results, which have been obtained by extrapolating, entirely coincide with results for the $n = n(T)$ dependences, which were obtained by the refractometric method (Figure 5). I.e. full coincidence of the temperature dependences for the n and n_{iso} has been obtained.

We have also measured the temperature behavior of the birefringence (Δn) of 5OCB, 6OCB, and 7OCB. The birefringence is an important optical parameter and characterizes optical anisotropic properties of media. This parameter is determined as

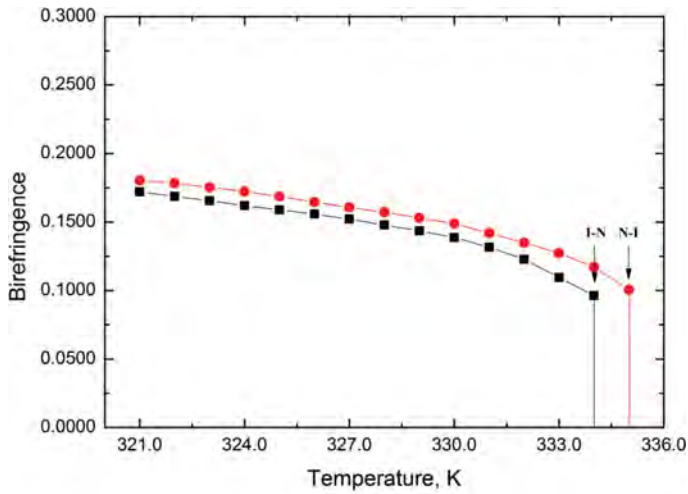
$$\Delta n = n_e - n_o = n_{||} - n_{\perp} \quad (7)$$

Besides, Δn , n , n_e , and n_o are connected with one another as [59–61]

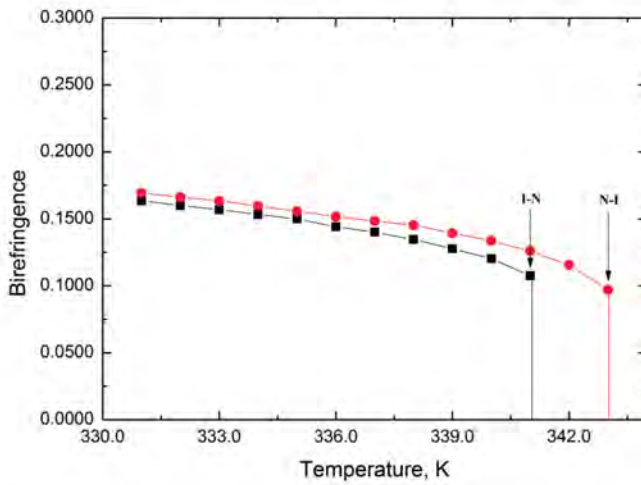
$$n_o = n - \frac{1}{3}\Delta n \quad (8a)$$

$$n_e = n + \frac{2}{3}\Delta n \quad (8b)$$

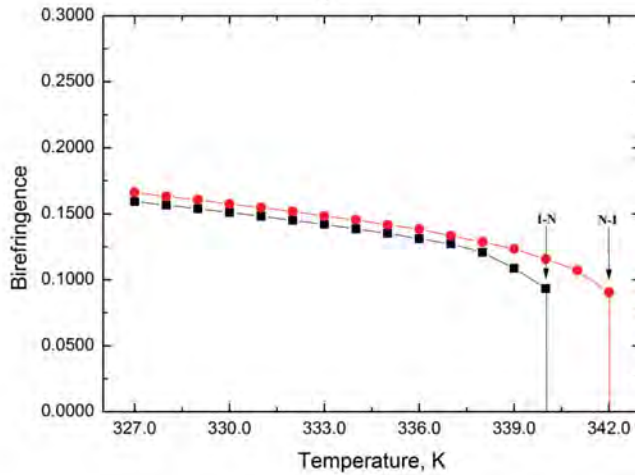
In Figure 7, $\Delta n = \Delta n(T)$ dependences of 5OCB, 6OCB, and 7OCB for the heating process are presented. As seen in this figure, clear decrease of Δn with an increase of temperature in the region of nematic phase takes place for 5OCB, 6OCB, and 7OCB. This result indicates the clear decrease of the optical anisotropic properties with an increase of temperature. At the clearing temperature $\Delta n = 0$. This result is connected with disappearance of the optical anisotropic properties at the N–I phase transition temperatures in 5OCB, 6OCB, and



a



b



c

Figure 7. Temperature dependences of the Δn for 50CB (a), 60CB (b), and 70CB (c). ● – the heating process; ■ – the cooling process.

7OCB, and with the fact that at the N–I phase transition $n_e = n_o = n$ equation takes place. It is interesting to note that value of Δn in 5OCB is bigger than that in 7OCB. i.e., an increase of length of the alkyl chain in 4-n-alkyl-4'-oxycyanobiphenyls leads to a decrease of the optical anisotropic properties of these materials. Taking into consideration that an increase of the alkyl chain in the 4-n-alkyl-4'-oxycyanobiphenyls leads also to a decrease of values of the refractive index, one can conclude that change of the length leads to change of the optical isotropic and the optical anisotropic properties of these materials.

In Figure 7, $\Delta n = \Delta n(T)$ dependences of 5OCB, 6OCB, and 7OCB for the cooling process are also presented. As can be seen in this figure, the Δn values fluently increase with a decrease of temperature. As also seen in Figure 7, the birefringence in 5OCB, 6OCB, and 7OCB by the *cooling process* appears at lower temperature, than this parameter disappears by the *heating process*. The shift of the $\Delta n = \Delta n(T)$ dependences to the lower temperature for the *cooling process* is connected with the temperature shift of the reverse N–I phase transition temperatures. The reason is that the nematic and isotropic states are alternatively metastable for $T > T_{NI}$ and $T < T_{NI}$. Although the transition may, in reality, take place at any temperature within this interval, the most probable transition is T_{NI} , which corresponds to an equal stability for the two phases. As a consequence of the region of metastability for the two phases, the measured transition temperature will not be the same on heating and cooling. So the temperature shift would be different during heating and cooling process.

Temperature behavior of Δn , the dielectric anisotropy, diamagnetic anisotropy, anisotropy of viscosity etc. corresponds to temperature behavior of the order parameter Q in liquid crystalline mesophases [18–20, 60, 61]. The Q and Δn are connected as [18, 26, 62, 63]

$$Q(T) = \frac{\Delta n(T)}{\Delta n_0} \quad (9)$$

Here Δn_0 is constant value and is the birefringence of liquid crystalline material in the crystalline state at the $T = 0$. This can be obtained by the fitting the value of Δn to the $T = 0$ state. Therefore is clear that the $Q \sim \Delta n$ correlation fulfils. This fact indicates that the temperature behavior of Δn has been used for the determination of the temperature behavior of the order parameter.

In Figure 8, the temperature dependences of the order parameter Q for 5OCB, 6OCB, and 8OCB are presented. As seen in this figure, by an increase of temperature unremitting and fluent decrease of the Q takes place in the region of nematic phase. Such behavior of $Q(T)$ indicates the unremitting and fluent decrease of long range of orientational ordering and on fluent change of the $D_{\infty h}$ symmetry. As seen in Figure 8, at the N–I phase transition the liquid crystalline ordering disappears and the $Q = 0$ equation takes place at the clearing point. Similar behavior of the $Q(T)$ was also observed for the heating process for large number of nematic mesophase in various liquid crystals [59, 63–66].

By a decrease of temperature, unremitting and fluent increase of the Q takes place in region of nematic mesophase (Figure 8). Such behavior of the $Q(T)$ indicates on gradual increase of the nematic ordering. As can be seen in Figure 8, appearance of the Q at the I–N phase transition is observed at lower temperature than disappearance of the Q at the N–I phase transition. Such interesting thermo-physical behavior of the Q is sufficiently important peculiarity for the heating-cooling processes in liquid crystals. Availability of the thermal hysteresis indicates the differences between transformations of a nematic ordering with symmetry as $D_{\infty h}$ to a isotropic state with symmetry as K_{∞} . We would like to note that the thermal hysteresis between the direct and reverse phase transition temperatures has been observed by various

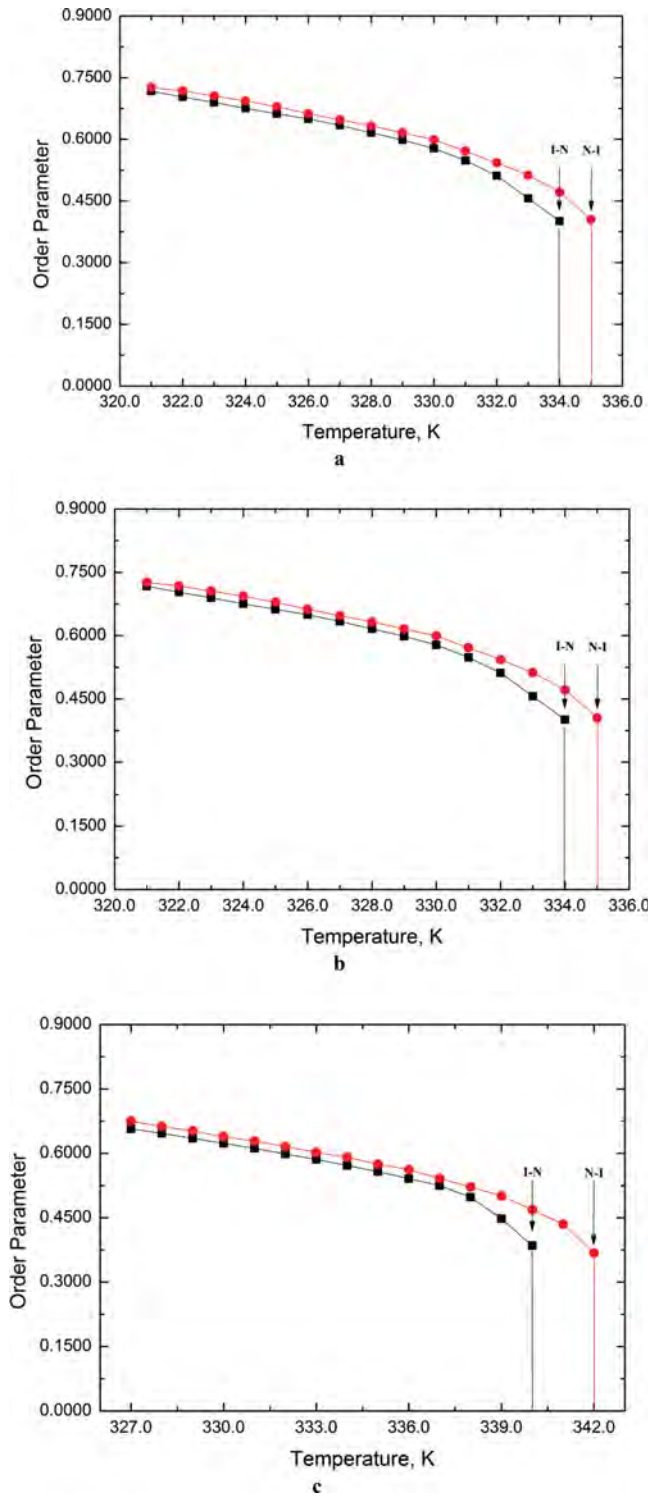


Figure 8. Temperature dependences of the Q for 50CB (in red), 60CB (in blue), and 70CB (in green). ● – the heating process; ■ – the cooling process.

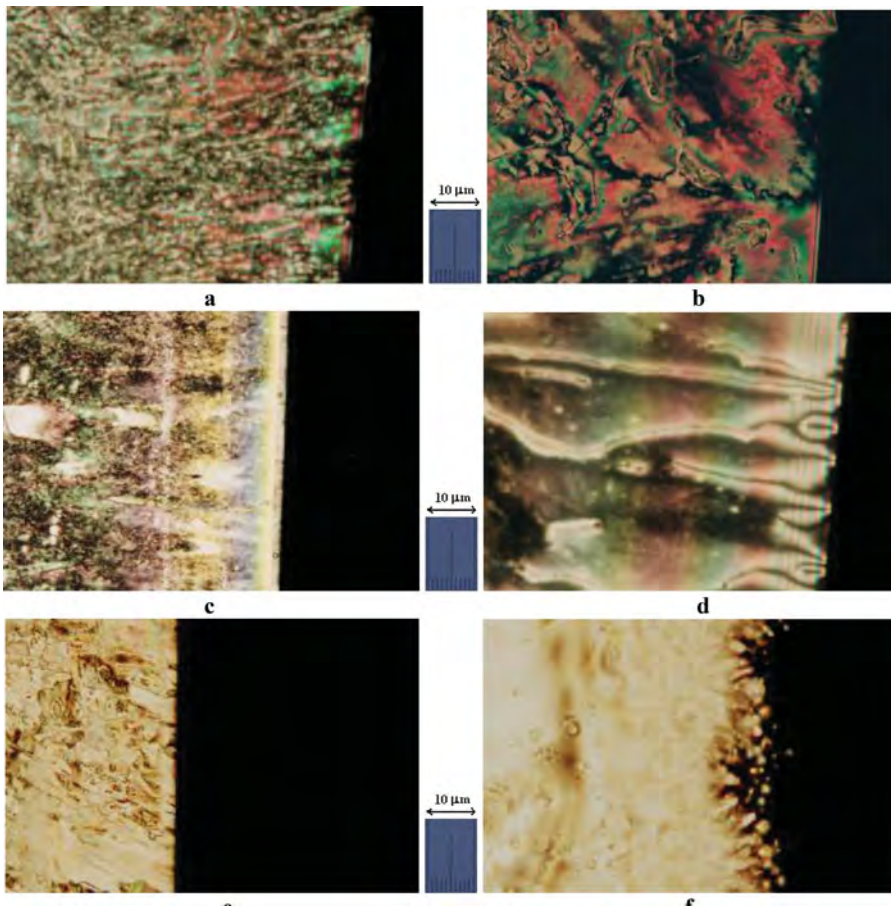


Figure 9. Regions of phase transitions between nematic mesophase and isotropic liquid. (a) N–I (5OCB), (b) I–N (5OCB); (c) N–I (6OCB); (d) I–N (6OCB); (e) N–I (7OCB); and (f) I–N (7OCB).

researchers for different physical parameters in large number of liquid crystalline materials [11, 12, 42–46, 48–51, 53].

We have also investigated the thermo-morphologic properties of the biphasic regions of the direct N–I and reverse I–N phase transitions. In [Figure 9](#), the biphasic regions of the above mention phase transitions for 5OCB, 6OCB, and 7OCB are presented. We would like to note that the biphasic regions of the N–I and I–N represent the thermal interval where the low temperature phase (i.e., nematic phase) and the high temperature phase (i.e., isotropic liquid) simultaneously coexist. As is seen in [Figure 9](#), in the biphasic regions of both direct N–I and reverse I–N phase transitions a lot of disclinations and texture defects take place. Appearance of such defective formations is connected with temperature gradient along the long axis of the sandwich-cell in the CTW device. Investigations showed that the temperature and linear widths of the biphasic regions for the reverse I–N phase transitions is larger than that for the direct N–I phase transitions in 5OCB, 6OCB, and 7OCB ([Table 2](#)). This fact indicates the thermal hysteresis for the biphasic region of the N–I phase transition.

Existence of the biphasic regions and availability of the thermal hysteresis have been theoretically predicted for the phase transitions between nematic phase and isotropic liquid in [1, 9, 11, 43, 67, 68] and was experimentally observed in [45, 46, 48, 69]. As is known, existence

Table 2. Temperature and linear widths of the biphasic regions of the direct and reverse phase transitions in 5OCB, 6OCB, and 7OCB.

Liquid crystal	Temperature widths of the biphasic regions (K)		Linear widths of the biphasic regions (μm)	
	N-Is	Is-N	N-Is	Is-N
5OCB	1.02×10^{-2}	1.53×10^{-2}	11.0	16.5
6OCB	0.50×10^{-2}	1.20×10^{-2}	5.3	12.9
7OCB	0.52×10^{-2}	1.81×10^{-2}	5.8	19.0

of the biphasic regions and availability of the thermic hysteresis are connected with superheating and supercooling of liquid crystalline material and are typical display for the first order *liquid crystalline mesophase-isotropic liquid* phase transitions [1, 9, 11, 43, 44]. Theoretically, the width of this biphasic region can be determined by Eq. (6) which is also the coexistence of the two phases.

Summary

In this work, the behavior of the refractive indices n , n_o , and n_e , and birefringence Δn versus temperature for the *heating-cooling* circular cycle have been studied. Temperature dependences of the order parameter Q in large temperature interval and in the direct and reverse phase transitions have been estimated. The thermo-morphologic properties of the biphasic regions of the direct *nematic-isotropic liquid* and reverse *isotropic liquid-nematic* phase transitions have been investigated.

The results obtained in this work can be summarized as follows:

- $n = n(T)$ dependences for 5OCB, 6OCB and 7OCB exhibit linear behavior and monotonous decrease of the n with as increase of temperature. An increase of the alkyl chain in the 4-n-alkyl-4'-oxycyanobiphenyls leads to a decrease of values of the refractive index.
- The thermal hysteresis for the temperature behavior of the n_o , n_e , Δn and Q for the *heating-cooling* processes and the shift of temperatures of the reverse transitions to lower temperatures have been observed. The thermal hysteresis and the shift of phase transition temperatures are connected with differences in character of transformation between the ordered structure and disordered state for the direct and reverse phase transitions.
- The N-I and I-N phase transitions exhibit significant thermal hysteresis due to the supercooling of the isotropic liquid and superheating of the nematic mesophase. From this and other works, it appears that the supercooling and superheating are the intrinsic property of the phase transition between N mesophase and isotropic liquid and are not removed by impurities or the presence of other nucleation sites.

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