

Comparative investigations of the thermotropic and optical refractive properties in micellar isotropic phase and nematic-calamitic mesophase of hexadecyltrimethyl ammonium bromide/water and hexadecyltrimethyl ammonium bromide/water/1-decanol lyotropic liquid crystalline systems

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Abstract We report the thermotropic properties and measurements of the temperature and concentration dependences of the refractive index in isotropic micellar L_1 phase and nematic-calamitic N_C mesophase of the hexadecyltrimethyl ammonium bromide/water (CTAB/ H_2O) and hexadecyltrimethyl ammonium bromide/water/1-decanol (CTAB/ H_2O /DeOH) lyotropic liquid crystalline systems. The effect of amphiphile and aliphatic alcohol concentrations on the refractive properties of L_1 phase and N_C mesophase has been studied. Jump-like changes of the refractive properties in regions of the L_1 phase– N_C mesophase lyotropic and thermotropic phase transitions have been found. The sufficient effect of CTAB addition on the refractive index value has been found for the CTAB/ H_2O and CTAB/ H_2O /DeOH lyotropic systems. In this work, the shape of micelles in L_1 phase and N_C mesophase has been also estimated. Estimation showed that structural units of L_1 phase are the isometric spherical micelles, and structural units of N_C mesophase are the anisometric rod-like micelles. Typical textures of N_C mesophase in the CTAB/ H_2O and CTAB/ H_2O /DeOH lyotropic systems and the heterophase regions of the N_C mesophase–isotropic liquid thermotropic phase transitions are presented.

Keyword Lyotropic systems · Textures · Phase states · Thermotropic properties · Optical refraction

Introduction

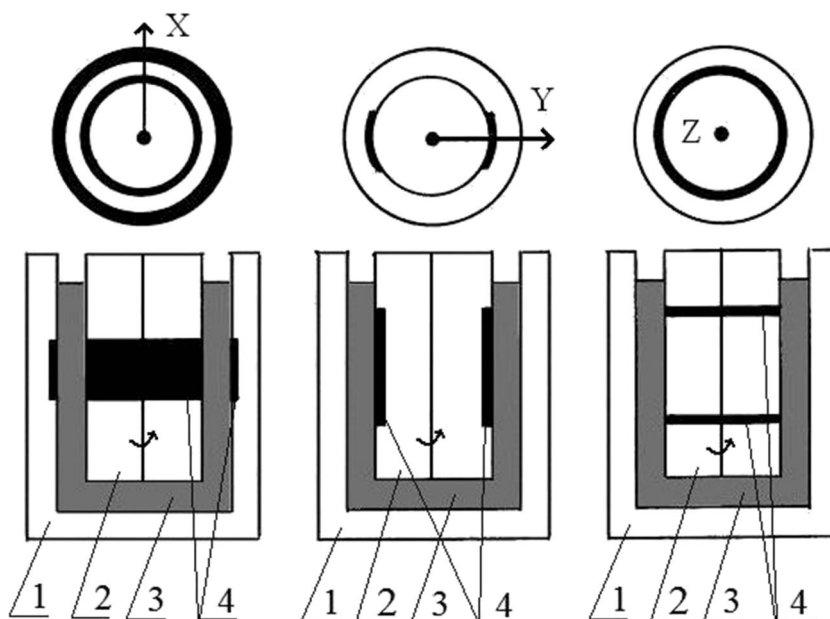
Lyotropic systems exhibit various types of optically isotropic phases and optically anisotropic mesophases. These phases and mesophases take place in the strongly definite temperature and concentration intervals [1–6]. Between these phases and mesophases, the biphasic and multiphasic regions and also the thermotropic and lyotropic phase transitions arise. Phase states, phase and mesophase boundaries, and the mesomorphic properties of lyotropic systems are determined by the phase diagrams [7–12].

Phases and mesophases of lyotropic systems display various physical properties (optical, dielectric, diamagnetic, viscous-elastic, acoustic, etc. properties). Because of the importance of the thermo-optical, magneto-optical, electro-optical, and acoustical-optical applications of lyotropic and thermotropic liquid crystalline systems, the optical properties of lyotropic phases and mesophases have special interest from both fundamental and application points of view. General optical parameter for isotropic phases and anisotropic mesophases in liquid crystals is the refractive index. This parameter determines the optical refractive properties of media and can change with concentration, temperature, number, and types of components in lyotropic liquid crystalline systems [13–18]. As it is noted in [19–21], study of the refractive indexes is a key for fundamental studies and practical applications of liquid crystals. Therefore, detailed investigations of the optical refractive properties and effect of components on

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Fig. 1 Scheme of the coaxial couette for the X - (a), Y - (b), and Z -directions (c). 1 Stator 2 – rotor, 3 lyotropic system, 4 electrodes



these properties in lyotropic liquid crystalline systems have special interest.

In this work, we are interested in the thermo-morphologic and optical refractive properties of optical isotropic micellar L_1 phase and optical anisotropic nematic-calamitic N_C mesophase in the binary and ternary lyotropic systems. The lyotropic systems under investigation are based on amphiphile as hexadecyltrimethyl (cetyltrimethyl) ammonium bromide (CTAB). The effect of amphiphile, water, and aliphatic alcohol on the thermo-morphologic and optical refractive properties of the abovementioned lyotropic systems have been investigated. We are also interested in the shape of micelles in the abovementioned lyotropic systems in L_1 phase and N_D mesophase for corresponding concentration intervals of lyotropic systems under investigations. Results of such investigations are presented in this work.

Experimental

In this work, isotropic micellar L_1 phase and nematic-calamitic N_C mesophase of the binary CTAB/ H_2O and ternary CTAB/ H_2O /DeOH lyotropic systems have been used. CTAB (cat. No.814119) and DeOH (cat. No.803463) were purchased from Merck. These materials have the high degree of purity and therefore were used without further purification. Water, which was used as the general solvent, was triple distilled and deionized.

The samples as the microslides were used in this work. The thickness of liquid crystalline layer in the microslides was $120 \pm 1.0 \mu\text{m}$. The samples were hermetically closed at once after filling by liquid crystalline system.

Investigations of the mesomorphic and thermo-morphologic properties of the CTAB/ H_2O and CTAB/ H_2O /DeOH lyotropic liquid crystalline systems have been carried

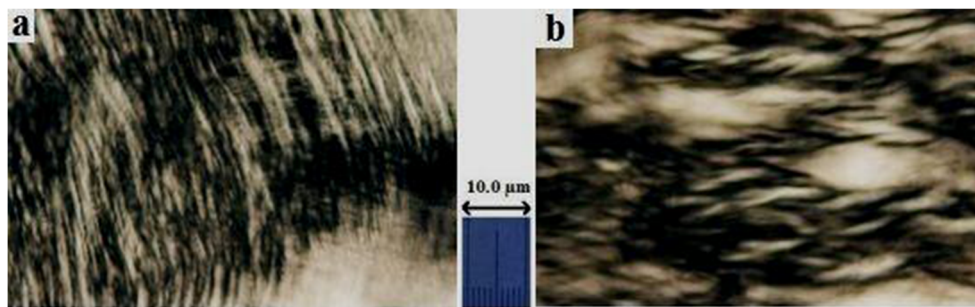
Table 1 Compositions of L_1 phase in the CTAB/ H_2O and the CTAB/ H_2O /+DeOH lyotropic systems

Samples	Compositions, wt%		
	CTAB	H_2O	DeOH
S1a	18	82	–
S2a	19	81	–
S3a	20	80	–
S4a	21	79	–
S5a	22	78	–
S6a	20	80	0.3
S7a	20	80	0.4
S8a	20	80	0.5
S9a	20	80	0.6
S10a	20	80	0.7

Table 2 Compositions of N_C mesophase in the CTAB/ H_2O and the CTAB/ H_2O /+DeOH lyotropic systems

Samples	Compositions, wt%		
	CTAB	H_2O	DeOH
S1b	23	77	–
S2b	24	76	–
S3b	25	75	–
S4b	26	74	–
S5b	27	73	–
S6b	24	76	0.3
S7b	24	76	0.4
S8b	24	76	0.5
S9b	24	76	0.6
S10b	24	76	0.7

Fig. 2 Typical textures of N_C mesophase in the CTAB/H₂O (a) and in the CTAB/H₂O/DeOH (b) lyotropic systems. Temperature 301.0 K; crossed polarizers; magnification $\times 100$



out using the polarizing optical microscopy (POM) method. Our setup consists of a trinocular polarizing microscope with orthoscopic/conoscopic observations, microphotographic system, Berek compensator and quartz plate from Olympus Optical Co., λ -plates ($\lambda=137\ \mu\text{m}$ and $\lambda=530\ \mu\text{m}$), optical filters, special heater thermostat with digital temperature control system, differential Cu–Co thermocouples, power supply, and multimeters.

The refractive properties of the CTAB/H₂O and CTAB/H₂O/DeOH lyotropic liquid crystalline systems have been investigated by using the polythermic refractometry setup (PR). The PR setup consists of Abbe's Precision Refractometer with Digital Thermometer from Atago Co. Ltd and recirculation immersion thermostat Ultraterm 200 Selecta. Accuracy of the refractive indices measurements was as 0.1 %. Temperature of liquid crystal under investigation was also controlled by Co–Co thermocouple with accuracy as $\pm 0.1\text{K}$. Thermocouple was placed in close vicinity of the lyotropic system.

For estimation of the shape of micelles in lyotropic phases and mesophases of lyotropic liquid crystalline systems under investigations, the method of the electrical conductivity anisotropy in the orientational shear flow has been used. Principles of this classic method were described in detail in [22–24]. In accordance with this method, the sum of changes of the electrical conductivity values for both the rod-like and disc-like (plate-like) micelles in the three mutually perpendicular directions must be equal to zero ($\sum \frac{\sigma_i - \sigma_0}{\sigma_0} = 0$) [22, 23, 25, 26] (here $i=x, y, z$). However, in the case of the rod-like micelles, a decrease of the electrical conductivity anisotropy in the X - and Z -directions and an increase of the electrical conductivity

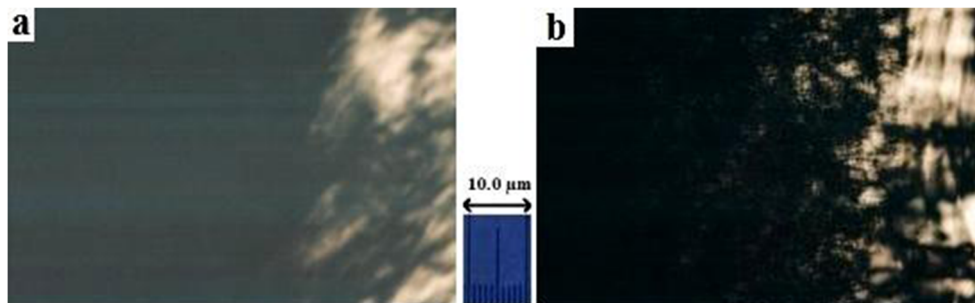
anisotropy in the Y -direction must take place. In the case of the disc-like (plate-like) micelles, a decrease of the electrical conductivity anisotropy in the X -direction and an increase of the electrical conductivity anisotropy in the Y - and Z -directions must take place [22, 23, 25, 26]. Therefore, by determination of the values of the electrical conductivity anisotropy in the three mutually perpendicular directions, the shape of micelles can be identified from the sign of these values [22–28]. In Fig. 1, the scheme of coaxial couette, which consists of the rotor, stator, and electrodes, is presented. The electrodes allow to determine the electrical conductivity anisotropy in the direction of the velocity gradient (X -direction), in the direction of the shift flow (Y -direction), and in the direction, which simultaneously is perpendicularly to the shift flow and to the velocity gradient (Z -direction).

We would like to note that the abovementioned method is similar to method of determination of the micelle shape by the optical birefringence of anisometric micelles in the shear flow [29].

Results and discussion

In this work, we are interested in the refractive properties of micellar L_1 phase and nematic-calamitic N_C mesophase. For L_1 phase in binary lyotropic system, samples S1a–S5a with variable ratio of CTAB/H₂O and in ternary lyotropic system, samples S6a–S10a with constant ratio of CTAB/H₂O, and variable concentration of DeOH have been investigated (Table 1). For N_C mesophase in binary lyotropic system, samples S1b–S5b with variable ratio of CTAB/H₂O and also in ternary

Fig. 3 Region of the N_C -isotropic liquid thermotropic phase transition in the CTAB/H₂O (a) and the CTAB/H₂O/DeOH (b) lyotropic systems. Crossed polarizers; magnification $\times 100$



lyotropic system, samples S6b–S10b with constant ratio of CTAB/H₂O, and variable concentration of DeOH have been investigated (Table 2). *L*₁ phase in the CTAB/H₂O and the CTAB/H₂O/DeOH lyotropic systems exhibits optical isotropic texture with black background. *N*_C mesophase in the CTAB/H₂O and the CTAB/H₂O/DeOH lyotropic systems displays by textures, which are presented in Fig. 4a, b. As seen in Fig. 2a, b, these textures are the same type and are typical schlieren textures for *N*_C mesophase. Textures consist of the thread-like formations, singular points, and small uniform regions and are well known for *N*_C mesophase of lyotropic liquid crystalline systems [9, 30–35].

The homeotropic uniform alignment of *N*_C mesophase has been obtained by application of magnetic field of 7.0 kG, which was applied parallel to the reference surfaces of the microslide. In the uniform-aligned regions, the rod-like micelles are oriented perpendicularly to the reference surfaces of the microslide. In this case, the director of *N*_C mesophase is oriented perpendicularly to the reference surfaces and molecules of amphiphile in the rod-like micelles are oriented parallel to these surfaces. Such arrangement of the optical axis and amphiphile molecules is cause of the negative optical birefringence ($\Delta n = n_{\parallel} - n_{\perp} < 0$) for *N*_C mesophase.

By the heating, The *N*_C-isotropic liquid (*N*_C-*I*) thermotropic phase transition has been observed in the CTAB/H₂O and CTAB/H₂O/DeOH lyotropic systems (Fig. 3). The thermo-morphologic investigations showed that schlieren texture of *N*_C mesophase exists without any change up to the thermotropic transition to an isotropic liquid state. This fact indicates on thermal stability of this mesophase in the abovementioned lyotropic systems.

For control and confirmation of presence of the spherical and rod-like micelles in the corresponding regions (regions of *L*₁ phase and *N*_C mesophase), the method of the electrical conductivity anisotropy in the orientational shear flow has been used. Investigations showed that for the samples in concentration region *L*₁ phase did not have any electrical conductivity anisotropy in the *X*-, *Y*-, and *Z*-directions. As an example, in Fig. 4a, the dependence of the electrical conductivity anisotropy vs. rotational frequency for *L*₁ phase is presented. As known, such behavior of the electrical conductivity anisotropy indicates on the spherical shape of micelles in lyotropic systems [19, 20, 23, 36, 37] (Fig. 5a). Investigations showed that for the samples in concentration region of *N*_C mesophase, the electrical conductivity anisotropy in the *X*- and *Z*-directions was negative and in the *Y*-direction was positive. As an example, in Fig. 4b, the dependences of the electrical conductivity anisotropy vs. rotational frequency for *N*_C mesophase are presented. As is known, such behavior of the electrical conductivity anisotropy indicates on the rod-like micelles in lyotropic systems [19, 20, 23, 36, 37] (Fig. 5b).

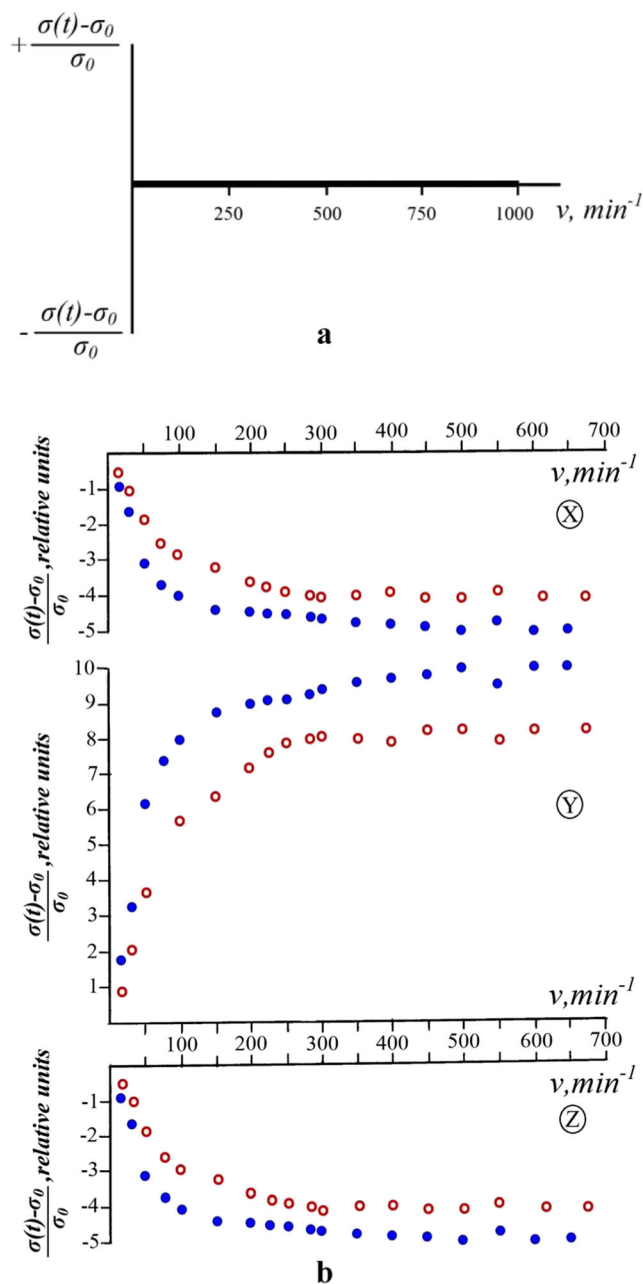


Fig. 4 The electrical conductivity anisotropy vs. rotational frequency for *L*₁ phase in 20 wt%CTAB/80 wt%H₂O and (20 wt%CTAB/80 wt%H₂O)+0.5 wt%DeOH lyotropic systems (**a**) and the electrical conductivity anisotropy vs. rotational frequency for *N*_C mesophase in 24 wt%CTAB/76 wt%H₂O (in red) and (24 wt%CTAB/76 wt%H₂O)+0.5 wt%DeOH (in blue) (**b**) lyotropic systems

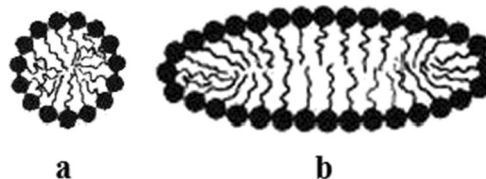


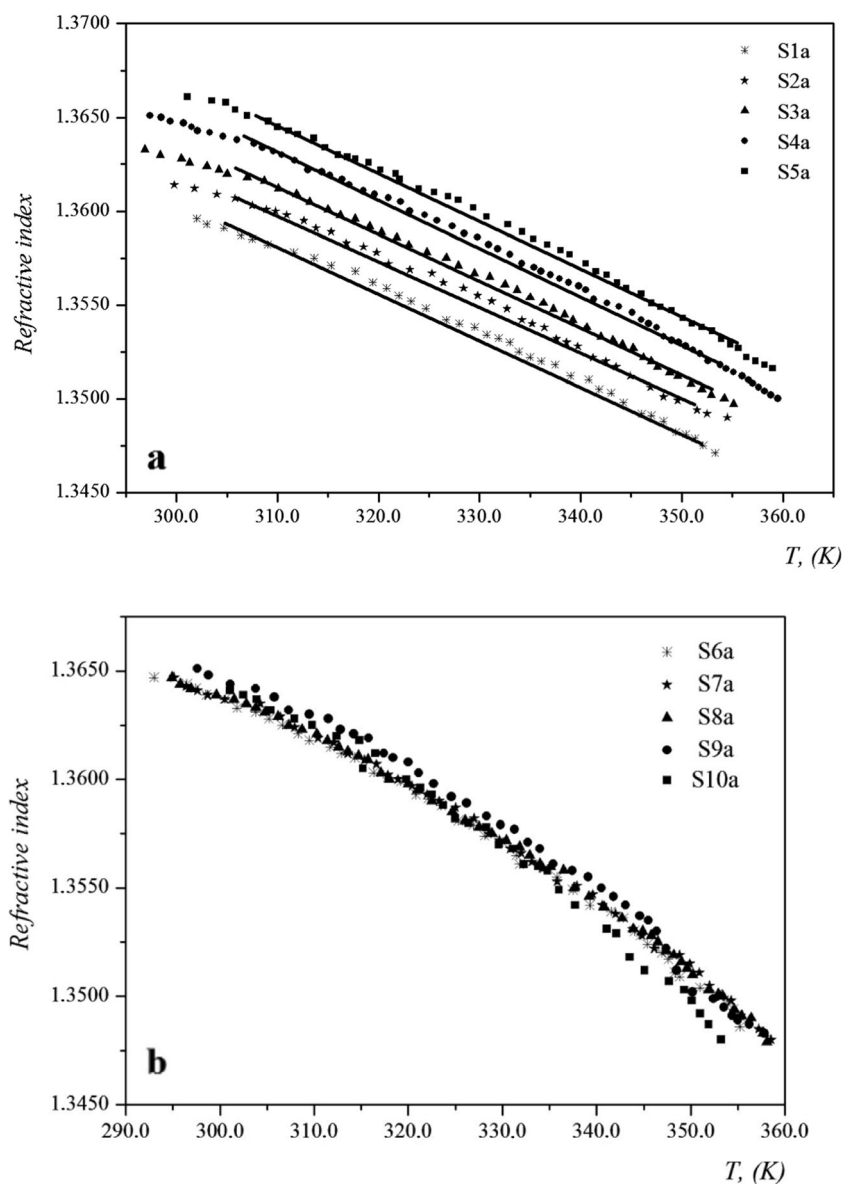
Fig. 5 Schematic representation of the spherical (**a**) and rod-like micelles (**b**) in lyotropic systems

Investigations of the temperature and concentration dependences of the refractive index for L_1 phase (samples S1a–S5a) showed that this index linearly decreases with an increase of temperature (Fig. 6). As is seen in Fig. 6a, an increase of CTAB concentration in binary, the CTAB/H₂O lyotropic system leads to an increase of the refractive index values. Behavior of the $n=n(T)$ dependences for S1a–S5a samples can be characterized by the $y=-2.6 \cdot 10^{-4} \cdot x + 1.3710$ equation. The addition of DeOH in lyotropic system with constant CTAB/H₂O ratio does not lead to a significant change of the refractive index values in L_1 phase for S6a–S10a samples (Fig. 6b). The effect of CTAB addition in lyotropic system is more significant for variation of the refractive index than effect of aliphatic alcohol. Thus, by variation of amphiphile concentration in the CTAB/H₂O lyotropic system is possible to regulate the refractive properties of L_1 phase in comparison

with variation of aliphatic alcohol concentration in such lyotropic systems.

Investigations of the temperature and concentration dependences of the refractive index for N_C mesophase (S1b–S5b samples) showed that the refractive index increases with an increase of CTAB concentration in the CTAB/H₂O lyotropic system (Fig. 7). As is seen in Fig. 7a, the $n=n(T)$ dependences for S1b–S5b samples exhibit linearly a decrease with an increase of temperature. Besides, these dependences exhibit the jump-like change at definite temperature. Investigations of the thermotropic properties of S1b–S5b samples showed that at these temperatures, the N_C mesophase–isotropic liquid (N_C-I) thermotropic phase transition and appearance of the heterophase regions of this transition took place. Thus, the jump-like change of the refractive index in S1b–S5b

Fig. 6 Temperature dependences of the refractive index for L_1 phase in the CTAB/H₂O lyotropic system for S1a–S5a samples (a) and in the (CTAB/H₂O)+DeOH lyotropic system for S6a–S10a samples (b)



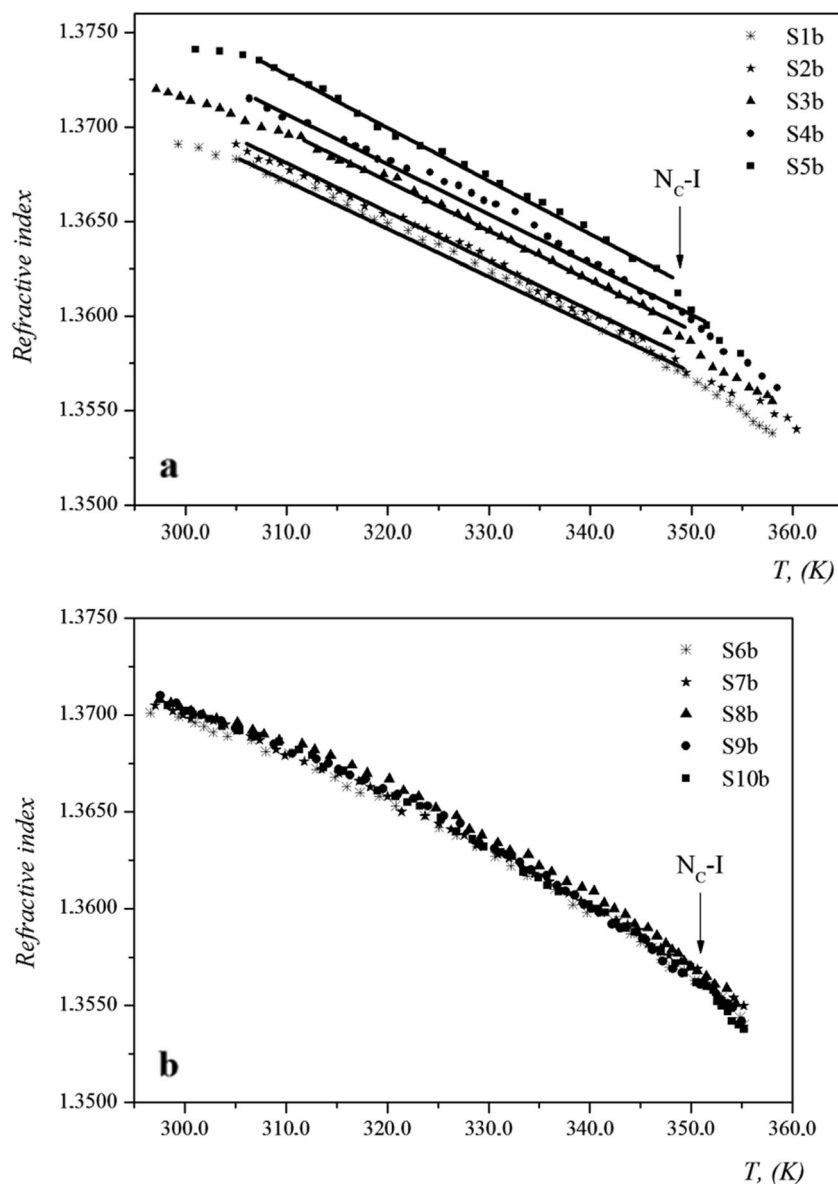
samples corresponds to the phase transition from N_C mesophase to an isotropic liquid.

Investigations showed that the addition of DeOH in lyotropic system with constant CTAB/H₂O ratio did not lead to a significant change of the refractive index values in N_C mesophase for S6b–S10b samples (Fig. 7b). As it is noted above, the addition of DeOH in lyotropic system with constant CTAB/H₂O ratio did not lead also to a significant change of the refractive index values in L_1 phase. The effect of CTAB addition in lyotropic system is more significant for variation of the refractive index in L_1 phase and N_C mesophase than effect of aliphatic alcohol. As known, an increase of amphiphile concentration in lyotropic system leads to an increase of number of micelles in volume of liquid crystalline system and to a decrease of distance between micelles [4, 38, 39]. Such effect leads to changes of interaction between micelles. Additionally,

as is known, an increase of amphiphile concentration in lyotropic system leads to an increase of the order degree of polar parts and non-polar chains of amphiphile molecules in micelles [1, 38, 40]. Obviously, such effects lead to change of the refractive index in L_1 phase and N_C mesophase. Thus, by variation of amphiphile concentration in the CTAB/H₂O, one can regulate the refractive properties of L_1 phase and N_C mesophase in comparison with variation of aliphatic alcohol concentration in such lyotropic system.

Concentration dependences of the refractive index $n=n(c)$ for the CTAB/H₂O lyotropic system at constant temperature conditions are presented in Fig. 8. As seen in this figure, the refractive index of L_1 phase at constant temperature condition exhibits the linear behavior. The linear behavior of the refractive index for these temperatures exhibits also N_C mesophase. However, the tilt angles for L_1 phase (θ_1) and N_C mesophase

Fig. 7 Temperature dependences of the refractive index for N_C mesophase in the CTAB/H₂O lyotropic system for S1b–S5b samples (a) and the (CTAB/H₂O)+DeOH lyotropic system for S6b–S10b samples (b)



(θ_2) are quite different. Results, which are presented in Fig. 8, show that interval of change of the refractive index in L_1 phase is larger than such interval in N_C mesophase. Besides, investigations showed that change of the tilt angle value for dependences, which are presented in Fig. 8, is corresponds to the L_1 phase– N_C mesophase (L_1 – N_C) lyotropic phase transition in the CTAB/H₂O lyotropic system, i.e., in the L_1 – N_C lyotropic phase transition region, the $n=n(c)$ dependences exhibit an alteration of the tilt angle. Thus, by the studies of the temperature and concentration dependences of the refractive index in lyotropic liquid crystalline systems, information about temperature and concentration phase transitions between lyotropic phases and mesophases can be obtained.

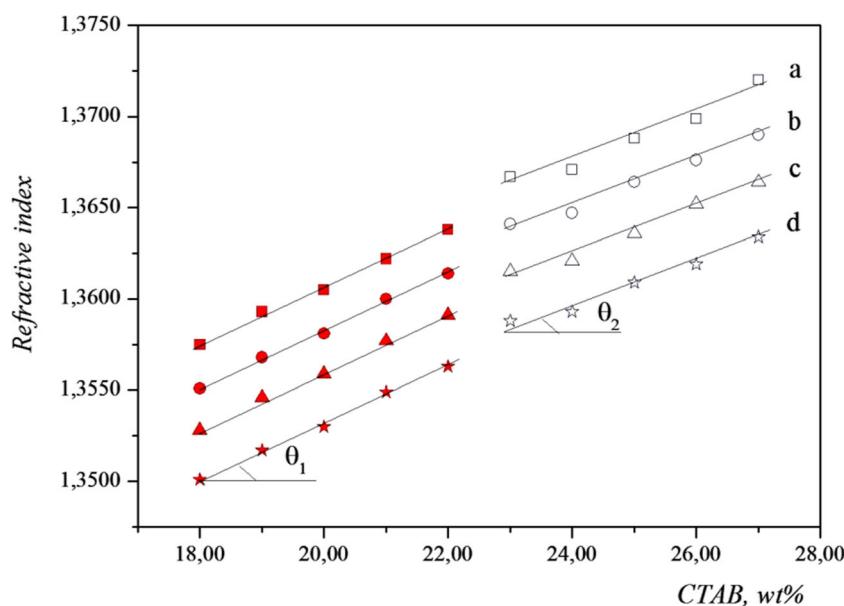
Summary

In this work, we are interested in the thermotropic and optical refractive properties of optical isotropic micellar L_1 phase and optical anisotropic nematic-calamitic N_C mesophase in the CTAB/H₂O and CTAB/H₂O/DeOH lyotropic liquid crystalline systems. The effect of amphiphile, water, and aliphatic alcohol on the thermomorphologic and optical refractive properties of the abovementioned lyotropic systems have been investigated. For control and confirmation of availability of the spherical and disc-like micelles in the corresponding concentration regions, the shapes of structural units were estimated. The method of the electrical conductivity anisotropy in the orientational shear flow has been used.

The results, obtained in this work may be shortly summarized as follows:

- An increase of CTAB concentration in the binary CTAB/H₂O lyotropic system leads to an increase of the refractive index values in L_1 phase and N_C mesophase. Such change of the refractive index value with change of CTAB concentration is obviously connected with the fact that an increase of CTAB concentration leads to an increase of the order of polar parts and non-polar chains of amphiphile molecules in micelles and also is connected with the fact that such increase in concentration leads to change of number of micelles in volume of lyotropic system.
- The addition of DeOH in the CTAB/H₂O lyotropic system with constant CTAB/H₂O ratio does not lead to a significant change of the refractive index values in L_1 phase and N_C mesophase. Obviously, the addition of non-polar solvent (decanol) in lyotropic system does not lead to change of order of amphiphile molecules in micelles and also does not lead to change of interaction between micelles.
- By change of CTAB concentration in both the CTAB/H₂O and CTAB/H₂O/DeOH lyotropic systems, the control of the refractive index values in L_1 phase and N_C mesophase can be carried out.
- Jump-like changes of the refractive index have been observed in the regions of the N_C – I thermotropic and L_1 – N_C lyotropic phase transitions. Such changes of the refractive index are connected with transformation of partially ordered structure with definite spatial symmetries of micelles to disordered state at the N_C – I phase transition and with transformation of the optical isotropic state to

Fig. 8 Concentration dependences of the refractive index for L_1 phase (in red) (S1a–S5a samples) and for N_C mesophase (in blue) (S1b–S5b samples) in the CTAB/H₂O lyotropic system for $a=313.0$ K, $b=323.0$ K, $c=333.0$ K, $d=343.0$ K temperatures



the optical anisotropic state in the L_1 – N_C phase transitions.

- Estimation of structural units of L_1 phase N_C mesophase showed that such units in L_1 phase are the isometric spherical micelles and in N_C mesophase are anisometric rod-like micelles. Additionally, L_1 phase is characterized by the absence of any electrical conductivity anisotropy, and N_C mesophase is characterized by definite electrical conductivity anisotropy in the mutually perpendicular directions (X -, Y -, and Z -directions).

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