## INVITED ARTICLE



# 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  $N_{\rm C}$  mesophase of the hexadecyltrimethyl ammonium bromide/water (CTAB/H<sub>2</sub>O) and hexadecyltrimethyl ammonium bromide/water/1-decanol (CTAB/H<sub>2</sub>O/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/H2O and CTAB/H<sub>2</sub>O/DeOH lyotropic systems. In this work, the shape of micelles in  $L_1$  phase and  $N_{\rm C}$  mesophase has been also estimated. Estimation showed that structural units of  $L_1$  phase are the isometric spherical micelles, and structural units of  $N_{\rm C}$  mesophase are the anisometric rod-like micelles. Typical textures of  $N_{\rm C}$ mesophase in the CTAB/H2O and CTAB/H2O/DeOH lyotropic systems and the heterophase regions of the N<sub>C</sub> mesophase-isotropic liquid thermotropic phase transitions are presented.

Arif Nesrullajev arifnesr@mu.edu.tr **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-  $(\mathbf{a})$ , Y-  $(\mathbf{b})$ , 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  $N_{\rm C}$ 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<sub>1</sub> phase and N<sub>D</sub> mesophase for corresponding concentration intervals of lyotroopic systems under investigations. Results of such investigations are presented in this work.

<b>Table 1</b> Compositionsof $L_1$ phase in theCTAB/H <sub>2</sub> O and theCTAB/H <sub>2</sub> O/+DeOHlyotropic systems	Samples	Compositions, wt%		
		CTAB	H <sub>2</sub> O	DeOH
	Sla	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

#### **Experimental**

In this work, isotropic micellar  $L_1$  phase and nematiccalamitic  $N_{\rm C}$  mesophase of the binary CTAB/H<sub>2</sub>O and ternary CTAB/H<sub>2</sub>O/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  $\pm 1.0$  µm. The samples were hermetically closed at once after filling by liquid crystalline system.

Investigations of the mesomorphic and thermomorphologic properties of the CTAB/H<sub>2</sub>0 and CTAB/H<sub>2</sub>0/ DeOH lyotropic liquid crystalline systems have been carried

<b>Table 2</b> Compositions of $N_{\rm C}$ mesophase in the CTAB/H <sub>2</sub> O and the CTAB/H <sub>2</sub> O/+DeOH lyotropic systems	Samples	Compositions, wt%		
		CTAB	H <sub>2</sub> O	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  $N_{\rm C}$  mesophase in the CTAB/H<sub>2</sub>O (a) and in the CTAB/H<sub>2</sub>O/DeOH (b) lyotropic systems. Temperature 301.0 K; crossed polarizers; magnification ×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.,  $\lambda$ -plates ( $\lambda$ =137 µm and  $\lambda$ =530 µm), optical filters, special heater thermostat with digital temperature control system, differential Cu–Co thermocouples, power supply, and multimeters.

The refractive properties of the CTAB/H<sub>2</sub>0 and CTAB/H<sub>2</sub>0/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 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 disclike (plate-like) micelles in the three mutually perpendicular directions must be equal to zero  $\left(\sum \frac{\sigma_i - \sigma_0}{\sigma_0} = 0\right)$  [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/H<sub>2</sub>0 and in ternary lyotropic system, samples S6a–S10a with constant ratio of CTAB/H<sub>2</sub>0, 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/H<sub>2</sub>0 and also in ternary

Fig. 3 Region of the  $N_{\rm C}$ isotropic liquid thermotropic phase transition in the CTAB/H<sub>2</sub>O (**a**) and the CTAB/H<sub>2</sub>O/DeOH (**b**) lyotropic systems. Crossed polarizers; magnification ×100



lyotropic system, samples S6b–S10b with constant ratio of CTAB/H<sub>2</sub>0, and variable concentration of DeOH have been investigated (Table 2).  $L_1$  phase in the CTAB/H<sub>2</sub>0 and the CTAB/H<sub>2</sub>0/DeOH lyotropic systems exhibits optical isotropic texture with black background.  $N_{\rm C}$  mesophase in the CTAB/H<sub>2</sub>0 and the CTAB/H<sub>2</sub>0 and the CTAB/H<sub>2</sub>0/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<sub>C</sub> mesophase. Textures consist of the thread-like formations, singular points, and small uniform regions and are well known for  $N_{\rm C}$  mesophase of lyotropic liquid crystalline systems [9, 30–35].

The homeotropic uniform alignment of  $N_{\rm C}$  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  $N_{\rm C}$  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  $N_{\rm C}$  mesophase.

By the heating, The  $N_{\rm C}$ -isotropic liquid (N<sub>C</sub>-I) thermotropic phase transition has been observed in the CTAB/H<sub>2</sub>O and CTAB/H<sub>2</sub>O/DeOH lyotropic systems (Fig. 3). The thermo-morphologic investigations showed that schlieren texture of  $N_{\rm C}$  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  $N_{\rm C}$  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  $N_{\rm C}$  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  $N_{\rm C}$ 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%H<sub>2</sub>0 and (20 wt%CTAB/80 wt%H<sub>2</sub>0)+ 0.5 wt%DeOH lyotropic systems (**a**) and the electrical conductivity anisotropy vs. rotational frequency for  $N_{\rm C}$  mesophase in 24 wt%CTAB/76 wt%H<sub>2</sub>0 (*in red*) and (24 wt%CTAB/76 wt%H<sub>2</sub>0)+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/H<sub>2</sub>O 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

Fig. 6 Temperature dependences of the refractive index for  $L_1$ phase in the CTAB/H<sub>2</sub>0 lyotropic system for S1a–S5a samples (a) and in the (CTAB/H<sub>2</sub>0)+DeOH lyotropic system for S6a–S10a samples (b) with variation of aliphatic alcohol concentration in such lyotropic systems.

Investigations of the temperature and concentration dependences of the refractive index for  $N_{\rm C}$  mesophase (S1b–S5b samples) showed that the refractive index increases with an increase of CTAB concentration in the CTAB/H<sub>2</sub>O 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  $N_{\rm C}$  mesophase–isotropic liquid ( $N_{\rm C}$ –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



samples corresponds to the phase transition from  $N_{\rm C}$  mesophase to an isotropic liquid.

Investigations showed that the addition of DeOH in lyotropic system with constant CTAB/H<sub>2</sub>O ratio did not lead to a significant change of the refractive index values in  $N_{\rm C}$ mesophase for S6b–S10b samples (Fig. 7b). As it is noted above, the addition of DeOH in lyotropic system with constant CTAB/H<sub>2</sub>O 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  $N_{\rm C}$  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,

Fig. 7 Temperature dependences of the refractive index for  $N_{\rm C}$ mesophase in the CTAB/H<sub>2</sub>0 lyotropic system for S1b–S5b samples (**a**) and the (CTAB/H<sub>2</sub>0)+DeOH lyotropic system for S6b–S10b samples (**b**) 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/H<sub>2</sub>O, 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/H<sub>2</sub>O 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  $N_{\rm C}$  mesophase. However, the tilt angles for  $L_1$  phase ( $\theta_1$ ) and  $N_{\rm C}$  mesophase



 $(\theta_2)$  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  $N_{\rm C}$  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– $N_{\rm C}$  mesophase  $(L_1-N_{\rm C})$  lyotropic phase transition in the CTAB/H<sub>2</sub>O lyotropic system, i.e., in the  $L_1-N_{\rm C}$  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  $N_{\rm C}$  mesophase in the CTAB/H<sub>2</sub>0 and CTAB/H<sub>2</sub>0/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/H<sub>2</sub>O 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/H<sub>2</sub>O lyotropic system with constant CTAB/H<sub>2</sub>O 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/H<sub>2</sub>O and CTAB/H<sub>2</sub>O/DeOH lyotropic systems, the control of the refractive index values in L<sub>1</sub> phase and N<sub>C</sub> mesophase can be carried out.
- Jump-like changes of the refractive index have been observed in the regions of the  $N_{\rm C}$ -I thermotropic and  $L_{\rm 1}$ - $N_{\rm C}$ 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  $N_{\rm C}$ -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/H<sub>2</sub>0 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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