Colloid Polym Sci (2017) 295:837–847
DOI 10.1007/s00396-017-4068-y

ORIGINAL CONTRIBUTION

Lyotropic mesophase in amphiphile + aliphatic alcohol mixtures with additions of water: mesomorphic, thermomorphologic, and optical refracting properties

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Received: 13 December 2016 / Revised: 7 March 2017 / Accepted: 7 March 2017 / Published online: 24 March 2017

Abstract Amphiphile + aliphatic alcohol lyotropic systems with addition of water can form micelles with normal, mixed, and inverse type. Such systems display various types of mesophases and exhibit interesting structural, physical, and physicochemical properties. Therefore, lyotropic systems are important objects from both fundamental and application points of view. In this work, shape of anisometric micelles has been determined, and also, the magneto-morphologic properties of textures and optical refractive properties of mesophase have been investigated in *hexadecyltrimethylammonium bromide* (HDTMABr) + 1-*decanol* (DeOH) lyotropic system with various additions of water (H_2O). Dependences of the magneto-morphologic properties vs. time have been obtained. Temperature and concentration dependences of the optical refractive index have been investigated. The effect of the DeOH/H_2O concentration ratio on the refractive properties has been studied.

Keywords Lyotropic system · Amphiphile · Micelle · Magnetically induced textures · Refractive index

PACS numbers 61.30.St61.30.Jf64.70.M64.70.pp

Introduction

Mixtures of amphiphiles in different solvents form micelles with the spherical, disc-like, and rod-like shapes. Such micelles in definite concentration and temperature regions form various lyotropic phases and lyotropic liquid crystalline mesophases. These phases and mesophases exhibit different spatial packing and point-like symmetries and display various physical and physicochemical properties. Lyotropic liquid crystalline mesophases are binary and multicomponent mixtures of amphiphile (anionic, cationic, or zwitterionic amphiphile), polar or/and non-polar solvents, optical active material, non-organic salt, etc. additions [1–7]. Water, aliphatic alcohol, and mixture of water with aliphatic alcohol (i.e., mixture of polar and non-polar organic solvent) are the most important solvents for amphiphile materials.

Liquid crystalline mesophases with structural units as the anisometric micelles arise when molecules of amphiphiles self-assemble in polar or/and non-polar solvents. In the case of amphiphile + water systems, lyotropic mesophases with normal micelles are arisen. Lyotropic mesophases with normal micelles also arise in amphiphile + water + aliphatic alcohol, when concentration of water is bigger than concentration of aliphatic alcohol. In the case of amphiphile + aliphatic alcohol mixture, lyotropic mesophases with inverse micelles are arisen. Lyotropic mesophases with inverse micelles also arise in amphiphile + water + aliphatic alcohol, when concentration of aliphatic alcohol is bigger than concentration of water. If the concentration of water is approximately equal concentration of aliphatic alcohol, anisometric micelles of mixed shape can be arisen in lyotropic systems [1–3, 8–11]. Thus, a change of *polar solvent/non-polar solvent* concentration ratio leads to a change of type of micelles and to a transformation of the spatial structure and point-like symmetry of liquid crystalline mesophase.

Lyotropic mixtures as amphiphile + aliphatic alcohol are very important materials in colloid systems, techniques, and technology of detergents and surfactants and also for studies of the thermotropic and lyotropic phase transitions. Besides, these mixtures are also sufficiently important as model system for investigations of biological function and processes. Unfortunately,

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many problems of physics and physicochemistry of such lyotropic liquid crystalline systems with specific micelles have not been sufficiently investigated.

Alkyltrimethylammonium bromide amphiphiles {hexadecyltrimethylammonium bromide (HDTMABr), tetradecyltrimethylammonium bromide (TDTMABr), and dodecyltrimethylammonium bromide (DDTMABr)} are sufficiently important materials for scientific investigations and application in techniques and technology. The point is that such amphiphiles form colloid systems with isometric and anisometric micelles of normal and inverse shapes in water and water + aliphatic alcohol mixtures. Such systems exhibit physically isotropic phases and physically anisotropic mesophases. The phase diagrams and peculiarities of phases and mesophases in HDTMABr + water (H_2O) and HDTMABr + H_2O + aliphatic alcohol are presented in [12–20]. The phase diagrams and peculiarities of phases and mesophases have been given for low concentration of aliphatic alcohol and high concentration of H_2O, namely, for the aliphatic alcohol/H_2O concentration ratios as $0 \leq c \leq 0.12$ [12–20]. Unfortunately, scientific information about phase states, physical and physicochemical properties of isotropic phases, and anisotropic mesophases in lyotropic mixtures for the aliphatic alcohol/H_2O concentration ratio as $c > 1.0$ is absent.

In this work, we are interested in shape of micelles in HDTMABr + 1-decanol (DeOH) + H_2O lyotropic system and type of liquid crystalline mesophase for the DeOH/H_2O concentration ratio as $c > 1.0$. The typical textures, dependences of the magneto-morphologic properties vs. time, and also temperature and concentration dependences of the optical refractive index have been investigated. The effect of the DeOH/H_2O concentration ratio on absolute value of the electrical conductivity anisotropy and on the refractive index in HDTMABr + DeOH + H_2O lyotropic system has been estimated.

Experimental

Materials and samples

Ionic amphiphile HDTMABr with molecular formula as $CH_3(CH_2)_{15}N(Br)(CH_3)_3$ (cat. No. SigmaUltra H9151) was purchased from Sigma. DeOH (cat. No. 803463), which was used as the general solvent, was purchased from Merck. HDTMABr was characterized by the CMC value as $0.90 \cdot 10^{-4}$ mol L^{-1}. HDTMABr and DeOH have the high degree of purity (non less than 99%) and therefore were used without further purification. Water, which was used as polar solvent, was triple distilled and deionized.

The preparation process of the lyotropic mixtures under investigations followed known procedure. HDTMABr and water were weighted into glass ampoules by a precision balance with an accuracy of $\pm 10^{-4}$ g. The mixtures were kept in thermostat at 308.0 ± 0.1 K for homogenization. After homogenization of binary mixtures, DeOH was added in these mixtures. Both binary and ternary lyotropic mixtures were periodically mixed by a shaker in hermetically closed ampoule. Homogeneity of the obtained lyotropic mixtures was controlled by the crossed polarizers and by study of the typical textures, using a polarizing optical microscopy.

For investigation of the magneto-morphologic properties of lyotropic liquid crystalline mesophase under investigation, the microslide samples as the sandwich cell were used. The thickness of liquid crystalline layer, which was placed between reference surfaces of the sandwich cell, was 100.0 ± 0.1 μm. The samples were hermetically closed at once after filling by lyotropic mixture.

Methods

In this work, for the study of the magneto-morphologic properties of lyotropic mixtures under investigations, the magneto-optical setup has been used. A permanent magnet from Lebold was used for the experiments to obtain magnetically induced textures and for investigations of the magneto-morphologic properties. The magnetic field as $H = 0.93$ T was available. Magnetic field was applied perpendicularly to the reference surfaces of the sandwich cells and accordingly perpendicularly to the liquid crystalline layer. During the magnetic field influence, the samples were kept at a stable temperature as 302.5 ± 0.1 K.

Investigations of the thermomorphologic properties of HDTMABr + DeOH lyotropic system with additions of 1-decanol have been carried out by the polarizing optical microscopy (POM) method. As is known, the POM method is a sufficiently convenient and informative method for investigation of the mesomorphic and morphologic properties of liquid crystalline phases and mesophases [1, 21–23]. Our setup consisted of a trinocular polarizing microscope with orthoscopic/conoscopic observations, microphotographic system, and Berek compensator from Olympus Optical Co., Cannon 6D digital system, optical filters, λ-plates ($\lambda = 137$ μm and $\lambda = 530$ μm), quartz plate, heater thermostat with a digital temperature control system, differential Cu–Co thermocouples, power supply, and multimeters.

In this work, the temperature dependences of the refractive index for lyotropic mixtures have been measured. For these measurements, the polythermic refractometry setup, based on an Atago Abbe refractometer, has been used. Accuracy for the refractive index measurements was 0.1%. The temperature changes of Abbe refractometer have been carried out by recirculation immersion thermostat Ultraterm 200. Temperature of mixtures under investigation was controlled by the Atago digital temperature controller with accuracy as ±0.1 K.

For the determination of the shape of micelles in lyotropic mixtures under investigation, the method of the electrical conductivity anisotropy in the orientational shear flow has been used. The principles of this classic method were described in detail in [24–27]. This method was modified by us and allowed to measure the electrical conductivity values simultaneously in both parallel and perpendicular directions to the shear flow. The setup was also capable for investigation of the dynamics of orientational processes in lyotropic liquid crystalline mesophases [28, 29]. The method, which has been used in this work, is connected with the anisometricity of micelles in lyotropic liquid crystalline systems. This method is based on the fact that anisometric micelles with the plate-like and rod-like shapes exhibit the translational mobility in the shear flow [24–27]. The sum of changes of the electrical conductivity for both the plate-like and rod-like micelles in the three mutually perpendicular directions (i.e., X-, Y-, and Z-directions) should be equal to zero [24–27, 30, 31]. The connection between the electrical conductivity in these directions is as follows:

$$2\left|\frac{\sigma_Y(t)-\sigma_0}{\sigma_0}\right| = 2\left|\frac{\sigma_Z(t)-\sigma_0}{\sigma_0}\right| = \left|\frac{\sigma_X(t)-\sigma_0}{\sigma_0}\right| \tag{1}$$

and

$$2\left|\frac{\sigma_X(t)-\sigma_0}{\sigma_0}\right| = 2\left|\frac{\sigma_Z(t)-\sigma_0}{\sigma_0}\right| = \left|\frac{\sigma_Y(t)-\sigma_0}{\sigma_0}\right| \tag{2}$$

for the plate-like and rod-like micelles, accordingly. As seen from Eqs. (1) and (2), estimation of the shape of the anisometric micelles is sufficient to determine the electrical conductivity in two directions, i.e., in direction of the shear flow (X-direction) and in direction perpendicular to the shear flow (Y-direction).

Results and discussion

In this work, five mixtures of HDTMABr + DeOH lyotropic system with additions of H_2O have been used. Compositions of lyotropic mixtures are presented in Table 1. These compositions were chosen for obtaining of lyotropic mesophase with micelles, which consist of low concentration of water and large concentration of aliphatic alcohol. We would like to note that HDTMABr + H_2O + DeOH lyotropic liquid crystalline system with low concentration of DeOH and high concentration of H_2O exhibits isotropic micellar L_1 phase, hexagonal E phase, lyotropic nematic phase, and lamellar D mesophases [15, 18, 32].

In Fig. 1, textures of S1–S5 samples are presented. As seen in this figure, textures of these samples consist of the so-named "oily streak" formations. As is known, texture with the oily streak formations is the most common texture for layered liquid crystalline mesophases and was observed only for such mesophases [22, 33–39]. The oily streaks are the

Table 1 Compositions of lyotropic liquid crystalline mixtures

Samples	Compositions (wt%)			1-Decanol/water ratio
	HDTMABr	1-Decanol	Water	
S1	40	32.00	28.00	1.14
S2	40	35.00	25.00	1.40
S3	40	38.00	22.00	1.73
S4	40	40.00	20.00	2.00
S5	40	42.00	18.00	2.33

bright birefringent bands. These bands consist of small confocal formations and form the network on the pseudoisotropic background. Textures with the oily streaks have been also observed by various researches in thermotropic cholesteric mesophase and lyotropic lamellar mesophase [22, 40–47]. As an example, in Fig. 2, textures with the oily streak formations, which have been obtained by us for lamellar D mesophase and thermotropic cholesteric mesophase, are presented. As seen from comparison Figs.1 and 2a, the morphological peculiarities of these textures have both some differences and some common peculiarities. In Fig. 1, the destroyed and bundle oily streak formations, which form dense net, are observed. In Fig. 2b, classic texture of thermotropic cholesteric mesophase with the oily streak formations is observed. Investigations showed that the morphological and optical peculiarities of textures, which are presented in Figs. 1 and 2a, are also quite different from texture, which is presented in Fig. 2b. Namely, the background of texture in Fig. 2b has planar alignment and is optically active. Optical activity of texture with the oily streaks in cholesteric mesophase is typical peculiarity for mesophases with the chiral structure of mesophase [22, 39–41]. Besides, the optical investigations showed that the optical sign of the planar aligned background in this texture (Fig. 2b) (i.e., sign of the birefringence) is negative. But, as it is noted earlier, the background in textures with the oily streaks in Figs. 1 and 2a is pseudoisotropic. Thus, it can be concluded that textures, which are presented in Fig. 1, are typical for mesophase with layered structure and are typical for lyotropic lamellar mesophase.

As seen from the comparison of the oily streak textures in Fig. 1, textures of S1–S5 samples are of the same type but have some differences in the morphologic properties. Namely, the density of the oily streak formations and small confocal formations in volume of the sandwich cell is different. Comparison of the density of the oily streak formations with component compositions of the previously mentioned samples showed that an increase of the DeOH/H_2O concentration ratio in HDTMABr + DeOH + H_2O lyotropic liquid crystalline system leads to an increase of this density in S1–S5 samples. Thus, an increase of DeOH concentration in the

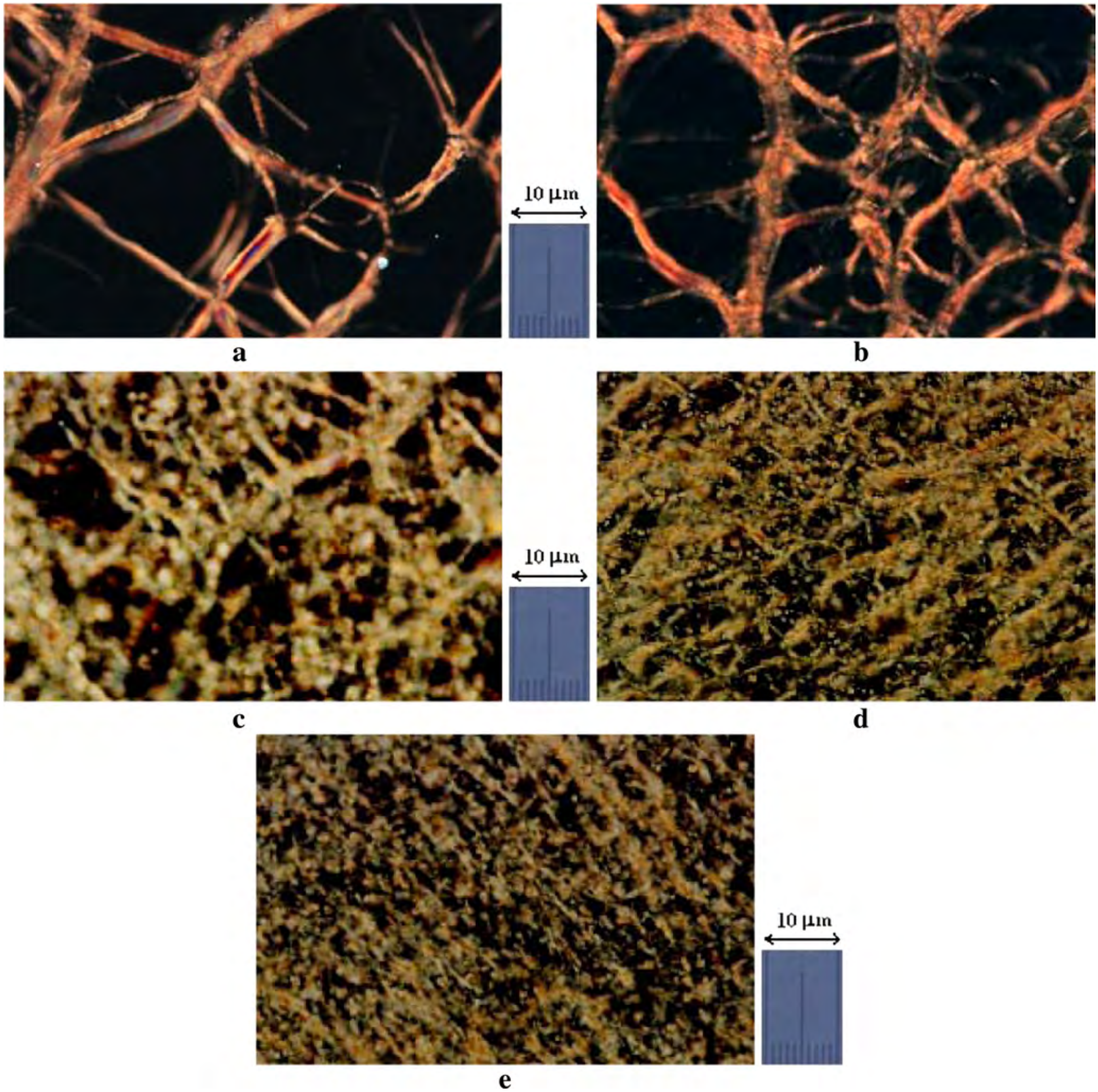

Fig. 1 Microphotographs of liquid crystalline textures in HDTMABr + DeOH mixture with addition of H_2O. **a** Sample S1. **b** Sample S2. **c** Sample S3. **d** Sample S4. **e** Sample S5. Temperature 302.5 K. Crossed polarizer and analyzer. Magnification ×100

previously mentioned lyotropic mixtures is an efficient way for an increase of the optical density of texture of mesophase under investigation.

As is mentioned earlier, for the determination of the shape of micelles in lyotropic liquid crystalline system under investigation, the character of the electrical conductivity anisotropy in the orientational shear flow has been investigated. As an example, in Fig. 3, dependences of the electrical conductivity anisotropy vs. the rotational frequency for samples S1, S3, and S5 in the X- and Z-directions are presented. As seen in this figure, the values of the electrical conductivity anisotropy in the X-direction are about two times bigger than that in the Y-direction. Such correlation between dependences for the X-direction and Y-direction corresponds to the Eq. (1) and indicates the fact that micelles of lyotropic mixtures under investigations have plate-like shapes [24, 25, 29, 48, 49]. Besides, as seen in Fig. 3, the absolute value of the electrical conductivity anisotropy of the samples under investigations increases with an increase of the rotational frequency in both X-direction (i.e., along the direction of the velocity gradient)

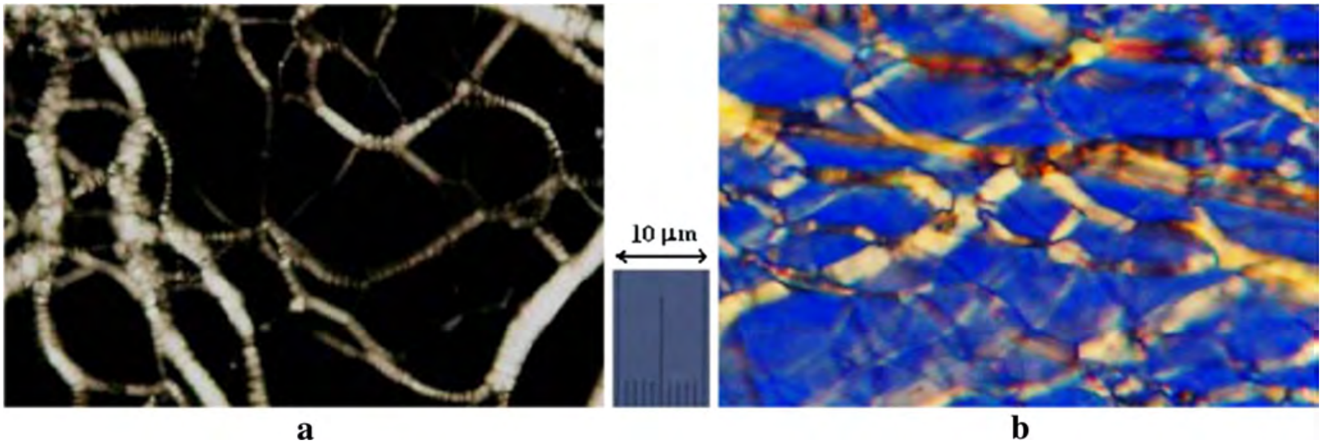

Fig. 2 Typical textures with "oily streaks." **a** Lyotropic lamellar mesophase of HDTMABr + H_2O + DeOH mixture with low concentration of DeOH. **b** Thermotropic cholesteric mesophase. Crossed polarizer and analyzer. Magnification ×100

and Y-direction (i.e., perpendicular to the velocity gradient). This increase is connected with an increase of the orientation degree of micelles under influence of the shear flow. Then, at definite rotational frequency values, the linear behavior of the electrical conductivity anisotropy takes place for all of the investigated samples. Such situation corresponds to full orientation of micelles in the shear flow.

Besides, as seen in Fig. 3, the behavior of the electrical conductivity anisotropy vs. the rotational frequency depends on the concentration ratio of component of lyotropic mixtures under investigation. In Fig. 4, dependences of the absolute value of the electrical conductivity anisotropy vs. the DeOH/H_2O concentration ratio are presented. As seen in this figure, an increase of the DeOH/H_2O concentration ratio in HDTMABr + DeOH + H_2O lyotropic liquid crystalline system leads to a decrease of the absolute value of the electrical conductivity anisotropy in samples S1–S5. An increase of DeOH concentration in samples under investigation (i.e., an increase of the DeOH/H_2O concentration ratio) leads obviously to a change of number of micelles in volume of liquid crystalline system, to a change of distance between the plate-like micelles and to a change of interaction between micelles and the counter ions [6, 45, 48]. In consequence of these changes, the anisometricity of micelles is changed. Besides, as is known, an increase of concentration of components in lyotropic liquid crystalline system leads to a change of the order degree of polar parts and non-polar chains of amphiphile molecules in micelles [45, 47, 50]. Such effects lead to a change of the electrical conductivity and, accordingly, to a

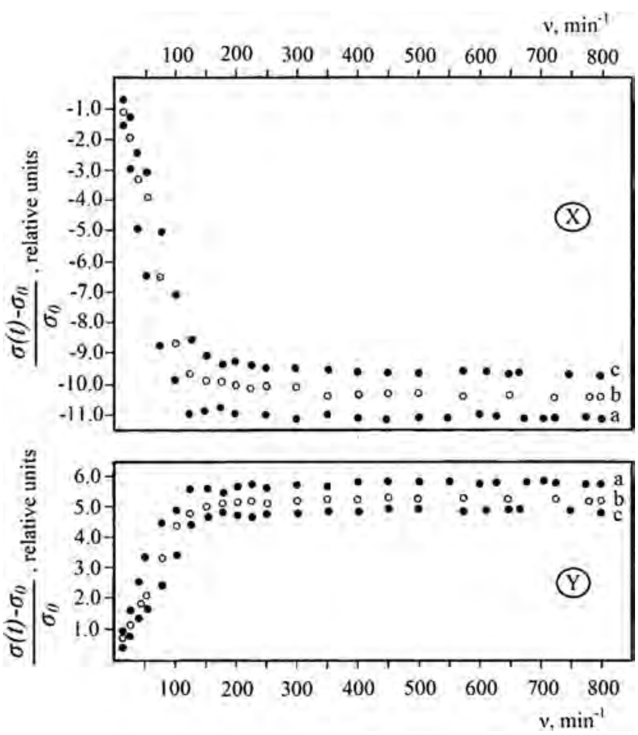

Fig. 3 The electrical conductivity anisotropy vs. rotational frequency for samples S1 (*a*), S3 (*b*), and S5 (*c*)

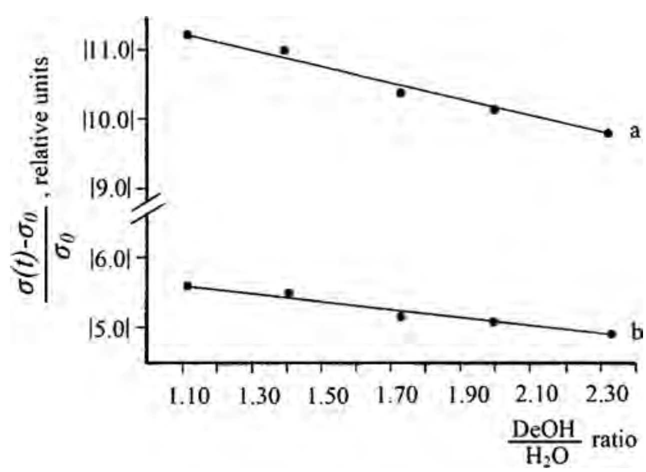

Fig. 4 Dependences of absolute value of the electrical conductivity anisotropy vs. DeOH/H_2O concentration ratio in HDTMABr + DeOH + H_2O lyotropic system. *a* X-direction. *b* Y-direction

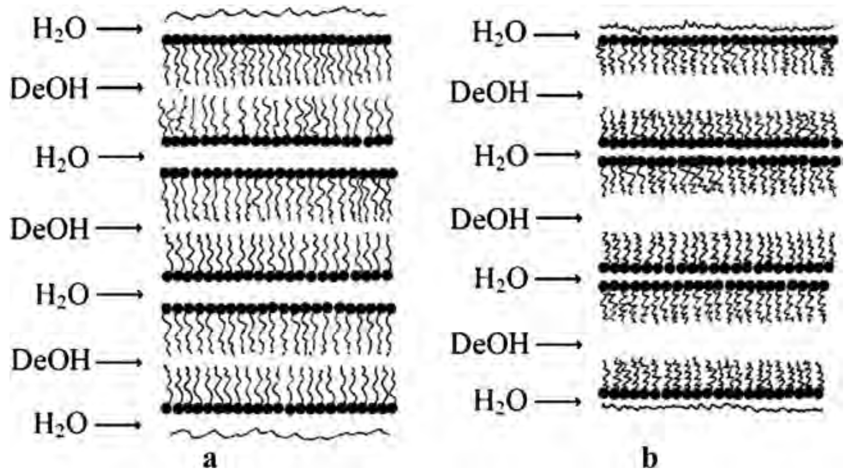

Fig. 5 Schematic sketch of micelles in lamellar mesophase of HDTMABr + DeOH + H_2O lyotropic system for case of the DeOH/H_2O <1.0 (**a**) and DeOH/H_2O >1.0 (**b**) concentration ratios

change of the electrical conductivity anisotropy in lyotropic mesophases.

Additionally, a change of the DeOH/H_2O concentration ratio leads to a change of the thicknesses of DeOH and H_2O layers in micelles of lamellar mesophase. In Fig. 5, schematic representation of micelles for the DeOH/H_2O <1.0 and DeOH/H_2O >1.0 concentration ratios is presented. As is indicated in [51–54], because of the flexibility of the non-polar part of amphiphile molecule, thickness of micelles (i.e., double length of amphiphile molecule) decreases with addition of aliphatic alcohol. Therefore, we can infer that such changes in the shape and sizes of micelles lead to a change of the physical and physicochemical properties of lyotropic systems.

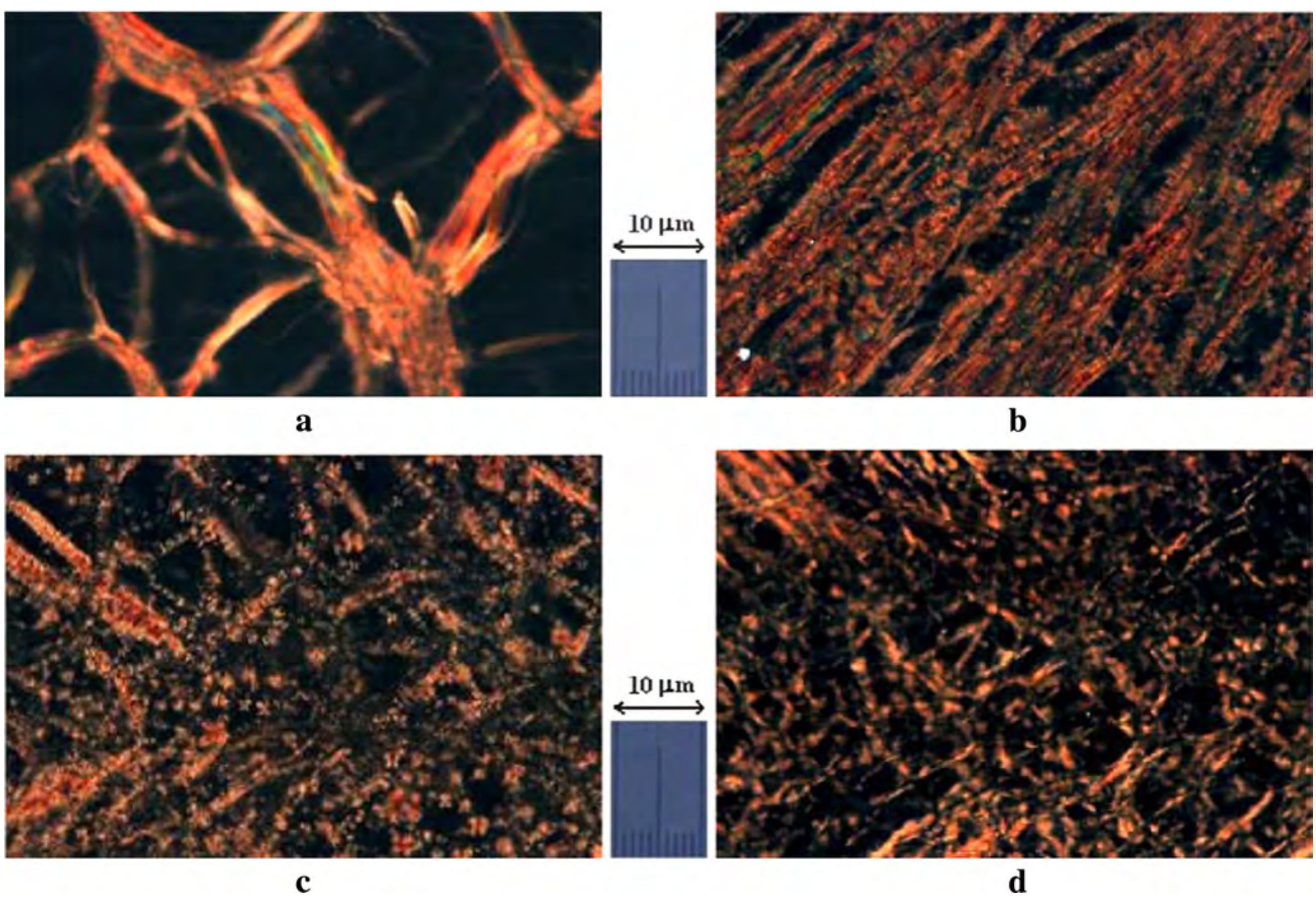

Fig. 6 Magnetically induced textures of sample S1. **a** 1.5 h in magnetic field. **b** 4.5 h in magnetic field. **c** 7.0 h in magnetic field. **d** 24.0 h in magnetic field. Temperature 302.3 K. Magnification ×100. Crossed polarizer and analyzer

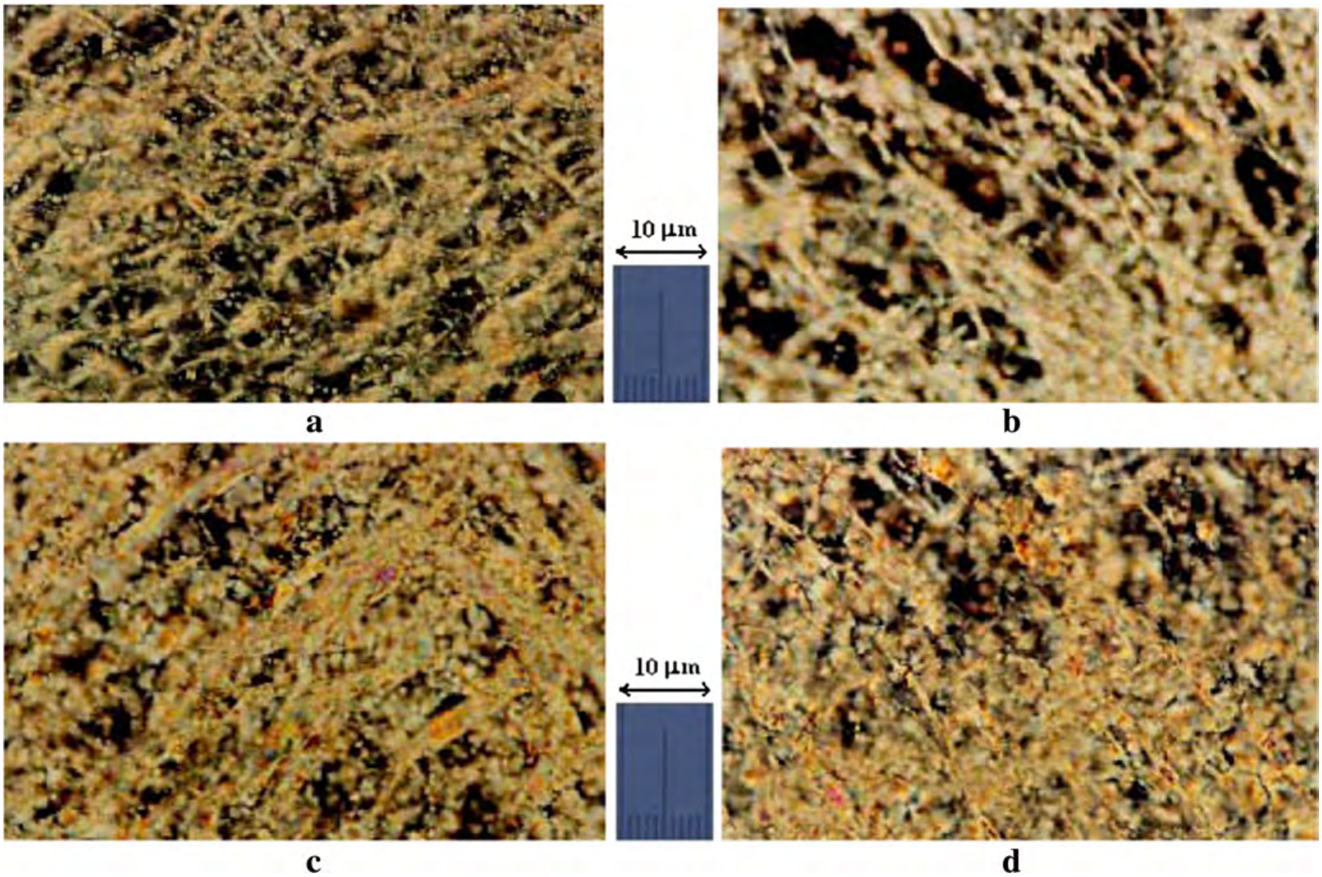

Fig. 7 Magnetically induced textures of sample S3. **a** 1.5 h in magnetic field. **b** 4.5 h in magnetic field. **c** 7.0 h in magnetic field. **d** 24.0 h in magnetic field. Temperature 303.0 K. Magnification ×100. Crossed polarizer and analyzer

In this work, the magneto-morphologic properties of samples S1–S5 have been investigated. Investigations showed that the external magnetic field has some influence on the morphologic properties of S1 and S2 samples and is some efficient for obtaining the non-equilibrium magnetically induced textures. But such field has no sufficient influence on the morphologic properties of S3, S4, and S5 samples. As an example, in Figs. 6, 7 and 8, texture transformations under influence of magnetic field for S1, S2, and S5 samples are presented. As seen in Fig. 6, transformations of the oily streak formations and destruction of the network of these formations take place. As the results of these transformations, a system of small confocal formations is formed. During these transformations, the pseudoisotropic background of textures was kept. Thus, the external magnetic field is effective for realization of *the oily streak formations → the system of scattered confocal formation* morphologic transformations in lyotropic mixtures under investigations. We would like to note that investigation of the magneto-morphologic properties of lamellar D mesophase in lyotropic liquid crystalline system amphiphile + H_2O + DeOH with low concentration of DeOH showed that the external magnetic field has low effect on the morphologic properties of lamellar mesophase D [44]. As seen in Figs. 7 and 8, sufficient transformations of typical textures and changes of types of textures have not been observed for S3, S4, and S5 samples. Thus, an increase of DeOH concentration in HDTMABr + DeOH + H_2O lyotropic liquid crystalline system caused a decrease of sensitivity of lyotropic mixture to the external magnetic field.

Investigations of the thermomorphologic properties of the reverse *isotropic liquid–lyotropic mesophase* phase transition in S1–S5 samples showed that in the biphasic region of this transition, the elongated germs of the mesophase under investigation have been observed (Fig. 9). These germs of the mesophase are so-named “batonnets” and arise in temperature region of isotropic liquid. Optical investigations by the quartz wedge showed that these batonnets are optically uniaxial and have positive optical sign. Availability of such formations indicates the layered structure of liquid crystalline mesophase. Such batonnets have been observed by various scientists at the thermotropic phase transition from isotropic liquid to layered liquid crystalline mesophase [22, 55, 56]. The availability of the batonnets in region of the isotropic liquid–lyotropic mesophase phase transition in samples under investigations, also as a character of the electrical conductivity anisotropy in the shear flow, indicates the availability of the layered structure of lyotropic

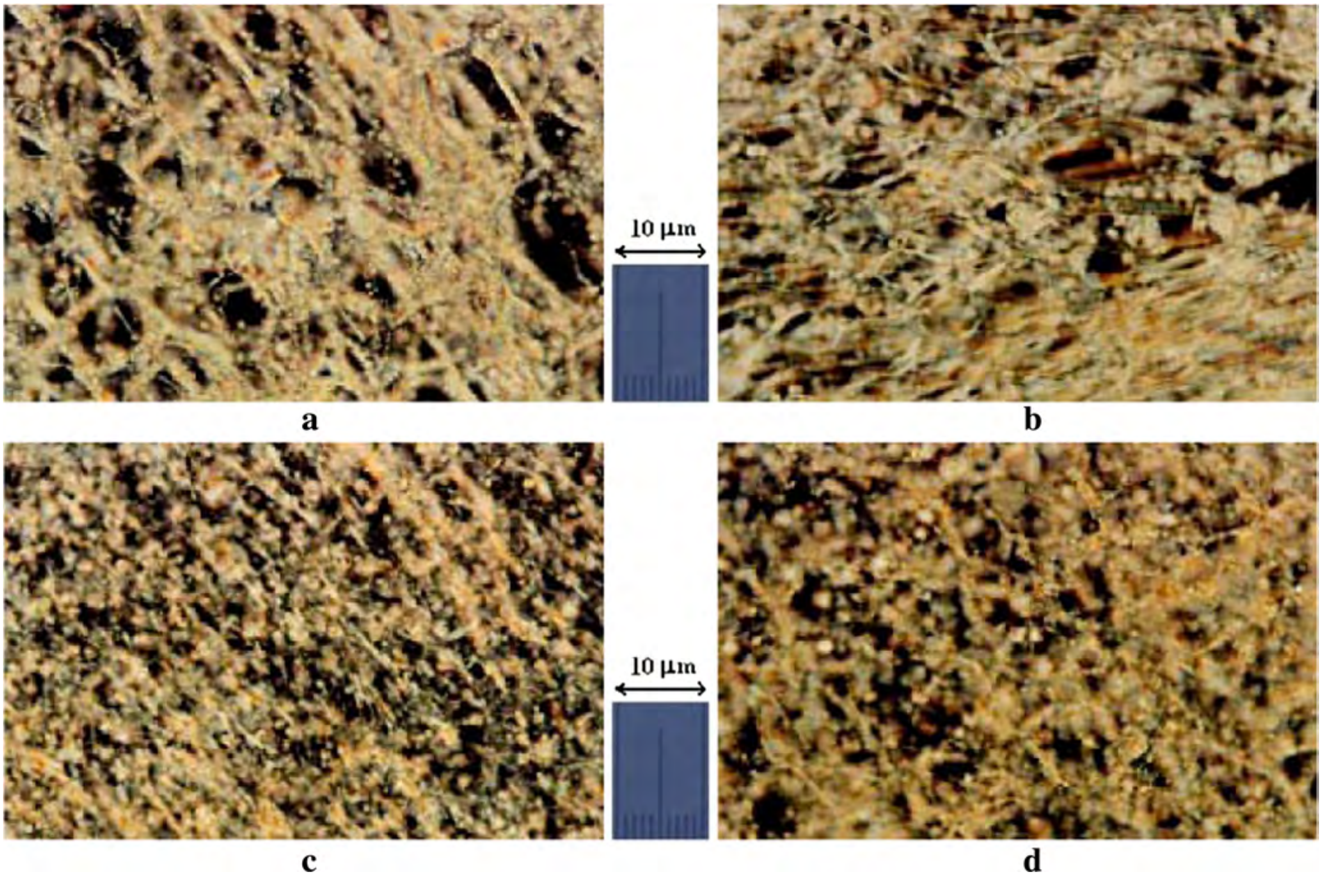

Fig. 8 Magnetically induced textures of sample S5. **a** 1.5 h in magnetic field. **b** 4.5 h in magnetic field. **c** 7.0 h in magnetic field. **d** 24.0 h in magnetic field. Temperature 302.5 K. Magnification ×100. Crossed polarizer and analyzer

mesophase in S1–S5 samples, i.e., availability of lamellar mesophase in mixtures under investigations.

In this work, we are also interested in the temperature and concentration behavior of the optical refractive index {$n = n(T)$ and $n = n(c)$, accordingly} in HDTMABr + DeOH + H_2O lyotropic liquid crystalline system. Investigations showed that this index linearly decreases with an increase of temperature for all the investigated samples (Fig. 10). Such character of the $n = n(T)$ dependences in S1–S5 samples indicates the stabile decrease of the refractive properties in lyotropic mixtures with large content of aliphatic alcohol. Besides, as seen in Fig. 10, a change of the DeOH/H_2O

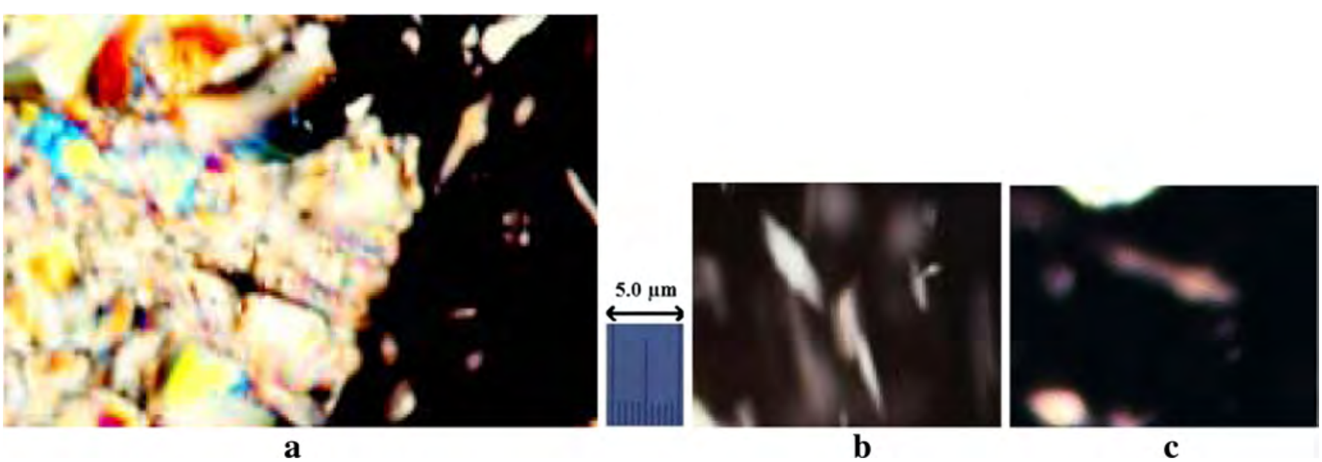

Fig. 9 **a** Region of the lamellar mesophase–isotropic liquid phase transition. **b**, **c** Batonnets in the lamellar mesophase–isotropic liquid phase transition. Magnification ×200. Crossed polarizer and analyzer

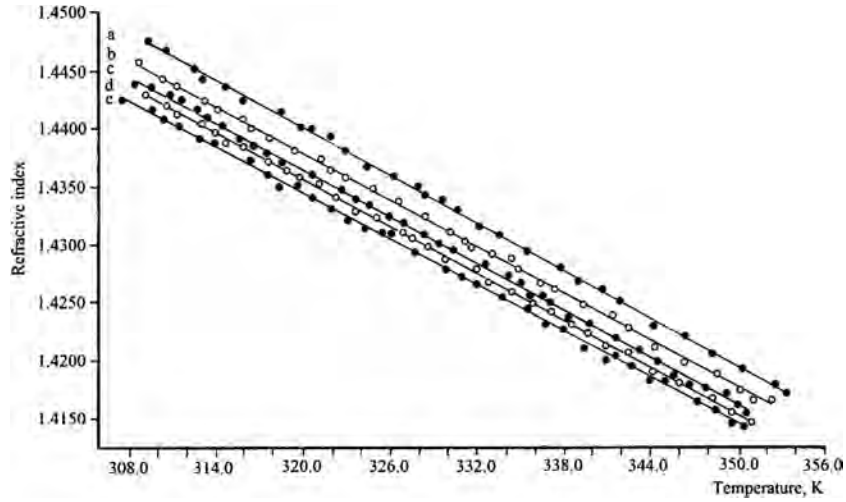

Fig. 10 Temperature dependences of the refractive index for samples S1 (*a*), S2 (*b*), S3 (*c*), S4 (*d*), and S5 (*e*)

concentration ratio with constant concentration of HDTMABr leads to a change of value of the optical refractive index. Namely, an increase of the DeOH/H_2O concentration ratio leads to a decrease of the refractive properties of lyotropic mixtures under investigations. The concentration dependences of the refractive index $n = n(c)$ for S1–S5 samples at constant temperature conditions are presented in Fig. 11. As seen in this figure, the refractive index for these samples at constant temperature condition exhibits the linear decrease with an increase of the DeOH/H_2O concentration ratio. By that, the interval of change of the refractive index for the presented temperatures is the same, i.e., as $\delta n \approx 0.0056$ (Fig. 11). Thus, the variation of the DeOH/H_2O concentration ratio is an effective way for change of the refracting properties in lyotropic system HDTMABr + DeOH with additions of H_2O; i.e., by variation of the DeOH/H_2O concentration ratio, it is possible to control the temperature and concentration dependences of the refracting properties in lyotropic mesophase of HDTMABr + DeOH + H_2O lyotropic liquid crystalline system.

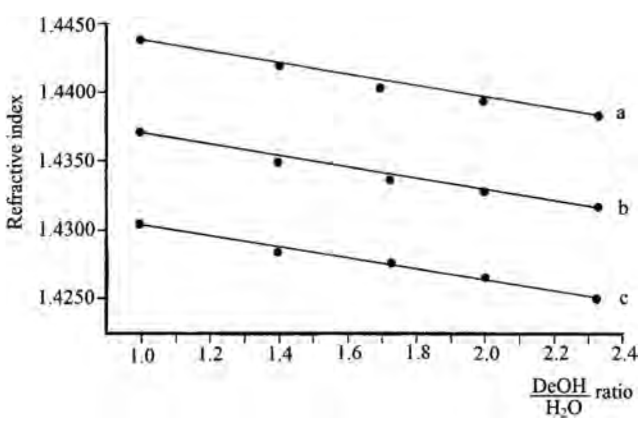

Fig. 11 Dependences of the refractive index vs. DeOH/H_2O ratio. *a* 313.0 K. *b* 323 K. *c* 333.0 K

Summary

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

HDTMABr + DeOH lyotropic mixtures with low concentration of H_2O exhibit textures with the oily streak formations. Such type of textures is typical for liquid crystalline mesophases with layered structures. Textures of HDTMABr + DeOH + H_2O lyotropic mixtures with the oily streaks have morphologic peculiarities, which are some different from such textures of lamellar mesophase in lyotropic mixtures of HDTMABr + H_2O with low concentration of DeOH. An increase of DeOH concentration in HDTMABr + DeOH + H_2O lyotropic mixtures leads to an increase of density of the oily streak formations and number of small confocal formations.

Studies of shapes of micelles in HDTMABr + DeOH lyotropic liquid crystalline system with additions of H_2O by method of the electrical conductivity anisotropy in the shear flow showed that these micelles in S1–S5 samples have the plate-like shapes. An increase of the DeOH/H_2O concentration ratio in lyotropic liquid crystalline system under investigations leads to a decrease of the absolute value of the electrical conductivity anisotropy in S1–S5 samples.

The external magnetic field has an effect on typical textures of lyotropic mesophase in the investigated lyotropic liquid crystalline system. Such field leads to the oily streak formations → the system of scattered confocal formation morphologic transformations in lyotropic mixtures under investigations.

Temperature dependences of the refractive index exhibit the linear decrease with an increase of temperature. An increase of the DeOH/H_2O concentration ratio in lyotropic liquid crystalline system leads to a decrease of the refractive properties of S1–S5 samples. By variation of the DeOH/H_2O concentration ratio, it is possible to control the temperature and concentration dependences of the refracting properties in lyotropic mesophase of HDTMABr + DeOH + H_2O lyotropic liquid crystalline system.

Acknowledgements This work has been partially supported by the Research Foundation of Mugla Sitki Koçman University, Grant No. BAP 15/124.

Funding This study was funded by the Research Foundation of Mugla Sitki Koçman University, Grant No. BAP 15/124.

Compliance with ethical standards

Conflict of interest The author declares that he has no conflict of interest.

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