



Molecular Crystals and Liquid Crystals

Publication details, including instructions for authors and subscription information:

<http://www.tandfonline.com/loi/gmcl20>

INVESTIGATION OF UNIAXIAL AND BIAxIAL LYOTROPIC NEMATIC PHASE TRANSITIONS BY MEANS OF DIGITAL IMAGE PROCESSING

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Version of record first published: 15 Jul 2010

To cite this article: A. R. Sampaio, A. J. Palangana & R. C. Viscovini (2004): INVESTIGATION OF UNIAXIAL AND BIAxIAL LYOTROPIC NEMATIC PHASE TRANSITIONS BY MEANS OF DIGITAL IMAGE PROCESSING, *Molecular Crystals and Liquid Crystals*, 408:1, 45-51

To link to this article: <http://dx.doi.org/10.1080/15421400490425838>

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INVESTIGATION OF UNIAXIAL AND BIAxIAL LYOTROPIC NEMATIC PHASE TRANSITIONS BY MEANS OF DIGITAL IMAGE PROCESSING

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Lyotropic nematic textures are investigated, by image processing, near the reentrant isotropic (I_{RE})–discotic nematic (N_D)–biaxial nematic (N_B)–clamitic nematic (N_C)–isotropic (I) phase transitions in a lyotropic mixture of potassium laurate, decanol, and D_2O . The (N_B), intermediate phase between the two uniaxial nematic ones, is characterized by optical birefringence measurements and discussed in terms of structural changes in the image frame by means of an adequate statistical approach.

PACS: 61.30.-v, 64.70.Md, 42.65.Jx

Keywords: biaxial nematic phase; digital image processing; lyotropic nematic textures

INTRODUCTION

Lyotropic liquid crystals (LC) are formed by mixtures of amphiphilic molecules and a solvent (usually water), under convenient temperature and concentration conditions [1]. The basic units of the LC are anisotropic micelles [2]. In the temperature–concentration phase diagram, two uniaxial and one biaxial nematic phases have been observed [3]. The uniaxial nematic phases have been shown to be prolate (calamitic– N_C) and oblate (discotic– N_D) micellar aggregates dispersed in water [2]. The biaxial nematic phase (N_B) appears to be an intermediate phase along the border between the two uniaxial nematic ones [4]. It has been shown that the

Many thanks are due to L. R. Evangelista (Brazil) for useful discussions, and the Brazilian Agencies CAPES, Fundação Araucária (PR) and CNPq (PADCT) for the financial support.

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micelles, in this nematic phase, have the shape of biaxial platelets [5,6]. Since this discovery, the N_B biaxial phase has been investigated with the use of different experimental techniques, such as optical birefringence, X-ray and neutron scattering and NMR measurements [2,3,6]. The phase diagram studied by Yu and Saupe [3] shows that for a particular choice of temperature and concentration conditions for the ternary system (potassium laurate, 1-decanol, and D_2O) there occurs a rich sequence of phase transitions, i.e., re-entrant isotropic \leftrightarrow discotic nematic \leftrightarrow biaxial nematic \leftrightarrow calamitic nematic \leftrightarrow isotropic. This phase sequence is investigated in this work.

The optical birefringence [7,8] near the discotic nematic (N_D) \leftrightarrow biaxial nematic (N_B) \leftrightarrow calamitic nematic (N_C) \leftrightarrow isotropic (I) phase transitions is determined through a Berek compensator to investigate the biaxial domain and its transitions to the uniaxial phases [7]. The image statistical treatment has been applied, particularly, to thermotropic liquid crystals [9,10]. By using a polarizing microscope and color CCD digital camera, we report a study on the topological texture configurations near the re-entrant isotropic (I_{RE}) \leftrightarrow $N_D \leftrightarrow N_B \leftrightarrow N_C \leftrightarrow I$ phase transitions as a function of temperature in a lyotropic mixture of potassium laurate, 1-decanol, and D_2O . In this context, these phase transitions are investigated via the digital image processing of the nematic textures. To our knowledge, this important method for analyzing phase transition has not yet been applied to lyotropic liquid crystals.

FUNDAMENTALS

The digital image of the nematic sample textures, detected by the CCD camera, is directly stored in a file driven by an IBM-PC-compatible computer with a resolution of 640×480 pixels, and a *Delphi* program was utilized to analyze these image frames. Further details about the method of image processing discussed in this work are given in Montrucchio et al. [9,10]. In this sense, it is convenient to consider the two-dimensional function $b(x, y)$, which represents the 24 bit true color pixel that ranges from 0 to 255 in red, green or blue colours. In our case we chose the green color (the most sensitive one) for the lyotropic nematic texture analysis. The average statistical directional moments $M_o^i(x, y)$ and $M_k^i(x, y)$ of the image frame are defined by [9]

$$M_o^i(x, y) = \frac{1}{l_{oi}} \int_0^{l_{oi}} b(x + r \sin \theta_i, y + r \cos \theta_i) dr, \quad (1)$$

$$M_k^i(x, y) = \frac{1}{l_{ki}} \int_0^{l_{ki}} [b(x + r \sin \theta_i, y + r \cos \theta_i) - M_o^i(x, y)]^k dr, \quad (2)$$

where k is the moment order, r is the radial distance from an arbitrary point $P(x, y)$ of the tone $b(x, y)$, along the eight radial directions at 45 degrees from each other, θ_i is the angle formed by the i direction with the y axis, and l_{0i} and l_{ki} are local coherence lengths of the image frame [9,10]. In this work, we compute the mean square deviation (σ) of the mean values of the coherence lengths (L_{0i} and L_{ki}) in 640×480 squared pixels with second-moment order ($k = 2$) for the 8 radial directions ($i = 1, \dots, 8$) that satisfy the following condition: $|M_k^i(x, y) - M_O^i(x, y)| \leq \delta M_O^i(x, y)$, where $\delta = 5\%$ is the confidence level [9]. Note each pixel has eight immediate neighbor pixels.

The lyotropic mixture investigated in this work was prepared with the following concentrations in weight percent: potassium laurate (KL: 25,30), decanol (DeOH: 6.24) and D₂O(68.46). KL was synthesized from lauric acid (Sigma-Aldrich Ltda, Sao Paulo, Brazil) via neutralization with potassium hydroxide (Merck, Brazil) and was further purified by recrystallization with hot ethanol (Merck, Brazil) several times in the laboratory, DeOH, and D₂O (> 99% purity from Sigma-Aldrich Ltda, Sao Paulo, Brazil). The lyotropic nematic phases were determined by optical microscopy and optical birefringence measurements [8], presenting the following sequence: $I_{RE} - N_D$ (13.8°C), $N_D - N_B$ (18.6°C), $N_B - N_C$ (21.2°C), and $N_C - I$ (40.3°C). Temperature dependences of optical birefringence, near the $N_D - N_B$ and $N_B - N_C$ phase transitions, were performed through a Berek compensator [7]. The nematic sample is encapsulated in sealed planar microslides with 0.2 mm and 0.1 mm of light path from Vitro Dynamics. The laboratory frame axes are defined with the boundary surfaces parallel to the 1-2 plane, and 3 is the axis normal to the biggest surface of the microslide [11]. The biaxial nematic phase is characterized by three orthogonal axes of symmetry, \vec{l} , \vec{m} , and \vec{n} , phase, fixed in the micelle. This biaxial nematic with the principal diamagnetic susceptibilities $\chi_1 > \chi_2 > \chi_3$, aligns in a magnetic field along with the χ_1 , which is parallel to the director \vec{n} [7,12]. This procedure was utilized to determine the optical birefringence ($\Delta n = n_2 - n_1$) of this lyotropic mixture [11]. The optical investigation was carried out using a *Leica* polarizing microscope connected to the Sony color CCD digital camera. The sample, not aligned, is placed in a hot stage (*MK200*) device. The sample temperature was controlled by a Heto circulating temperature bath stable within 0.01 K. The optical measurements were performed only when the temperature of the sample was stabilized to better than 0.1°C. A heating rate of $\sim 0.8 \text{ mKs}^{-1}$ was used during the acquisition of the experimental image frames.

RESULTS AND DISCUSSION

The optical birefringence (Δn) as functions of temperature, near the $N_D - N_B - N_C - I$ phase transitions, are presented in Figure 1. As one can

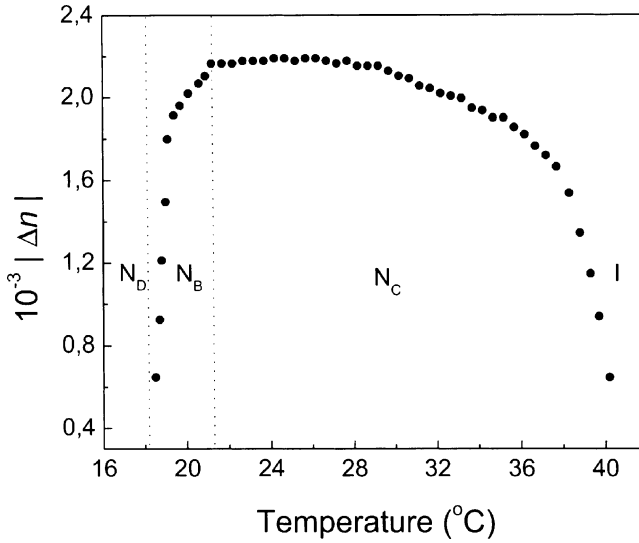


FIGURE 1 Birefringence (Δn) as a function of the temperature of KL/DeOH/D₂O system. N_D , N_B , N_C , and I are the dicotic nematic, biaxial nematic, calamitic nematic, and isotropic phases, respectively.

see from Figure 1, the temperature range of the biaxial nematic phase is around 2.6°C. This result is in agreement with the phase diagrams reported in the literature [3,4], and it is also consistent with optical birefringence measurements performed on the similar lyotropic mixtures [7,11]. Figure 2 shows the nematic textures obtained upon heating from the re-entrant isotropic phase in the N_D phase, near the $N_D-N_B(N_B-N_C)$ phase transition and N_C phase. The schlieren texture in N_D phase is transformed into a pseudisotropic texture (see Figure 2(a)). Note that this texture turns to a bright irregular domain at N_D-N_B phase transition, as exhibited in Figure 2(b), and after the transition is completed the N_B phase is characterized by the presence of smooth schlieren texture [13]. It is important to mention that the surface alignment, induced by the boundary conditions, of N_D phase is perpendicular to the biggest surface of the microslide. On the contrary, the surface alignment of N_C phase (Figure 2(d)) is parallel to the surface of the microslide, while the surface alignment in the N_B phase is parallel to the 1 axis and perpendicular to the 3 axis. On the other hand, the recognition of the transition point N_B to N_C , by optical observations between schlieren textures, is difficult to read [13]. Therefore, our birefringence data show that this transition point occurs at 21.2°C (Figure 2c). This fact is justified by the result of the digital image processing method of the nematic textures discussed in the context of this article.

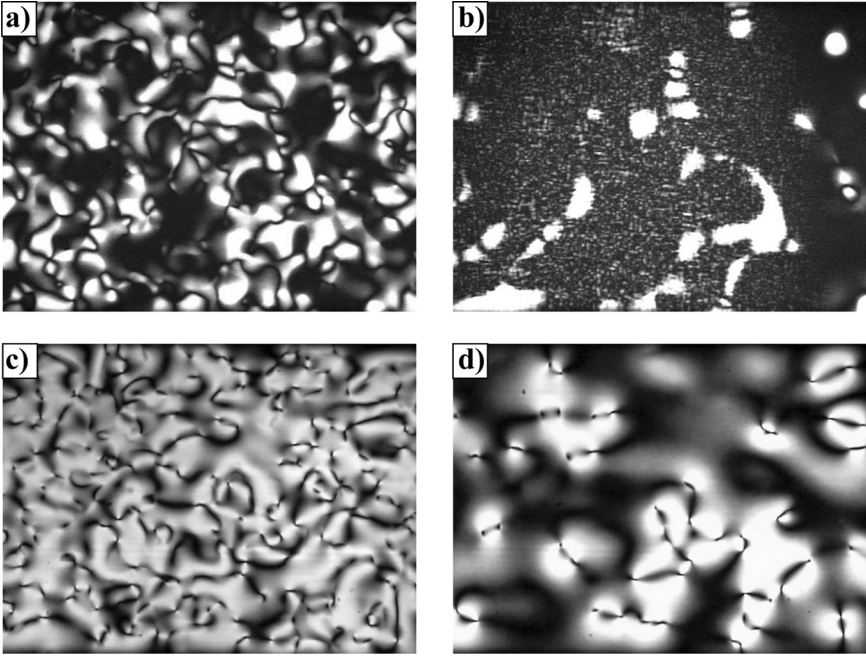


FIGURE 2 Lyotropic nematic textures: (a) discotic at 16.3°C, where the mean square deviation (σ) reaches the maximum value; (b) near the N_D-N_B phase transition; (c) near the N_B-N_C phase transition; and (d) calamitic at 31.6°C, where σ reaches the maximum value.

The mean square deviation (σ), obtained by using Equations (1) and (2), as a function of temperature near the $I_{RE} \leftrightarrow N_D \leftrightarrow N_B \leftrightarrow N_C \leftrightarrow I$ phase transitions, is presented in Figure 3. It can be observed that σ near the $I_{RE} - N_D$ phase transition increases and becomes maximum around 16.3°C and subsequently decreases as the temperature increases. This behavior of σ is consistent with the appearance of pseudoisotropic schlieren texture of N_D phase. In the temperature range of N_B phase, σ presents a rapid growth, followed by a decrease in direction of the N_C phase. This important result is in accord with the texture characteristics investigated by optical microscopy near the $N_D-N_B-N_C$ phase transitions exhibited in Figure 2. In this sense, the growth of σ at N_D-N_B transition reflects the appearance of bright irregular spots [13]. According to Saupe et al. [13], this irregularity disappears and transforms gradually to a smooth schlieren texture. This fact is associated with the decrease of σ until the N_B-N_C phase transition (see Figure 3). Note that the biaxial nematic phase occurs between two minimum values of σ concerning the N_D-N_B and N_B-N_C

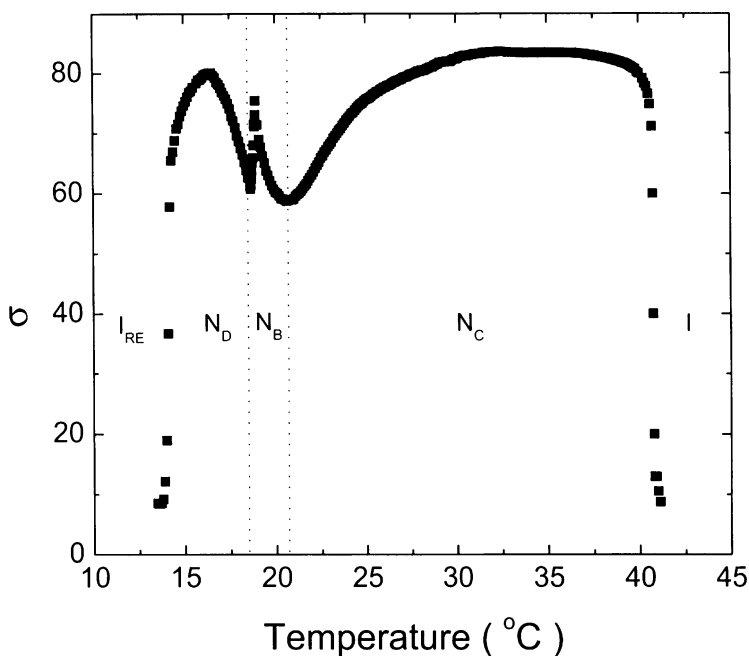


FIGURE 3 Temperature dependence of the mean square deviation (σ) in KL/DeOH/D₂O system. I_{RE} (isotropic reentrant), N_D , N_B , N_C , and I phases, respectively.

phase transitions, respectively. From this N_B-N_C phase transition, the classic *schlieren* texture of N_C phase may spontaneously get some kind of orientation of the micelles. We remember that, in this case, the geometry of the microslide [14] favors the orientation of the director n parallel to the 1 axis. In this sense, as one can see from Figure 3, near the $N_B-N_C(N_C-I)$ phase transition, σ increases (decreases) as the temperature increases and disappears in the isotropic phase. The $I_{RE}-N_D$ and N_C-I phase transitions are clearly identified by our experimental results. The optical measurements reported here were performed only for the heating cycle. However, for cooling with the same rate of $\sim 0.8 \text{ mKs}^{-1}$, we are convinced that the minimum values of σ , determined near the phase transitions investigated in the article, will be reproduced. On the other hand, a small shift in the position of the maxima of σ is expected.

To sum up, we have carried out a nematic texture study by image processing near the $I_{RE} \leftrightarrow N_D \leftrightarrow N_B \leftrightarrow N_C \leftrightarrow I$ phase transitions of a lyotropic mixture (KL/DeOH/D₂O). The investigation method utilized in this work is sensitive and constitutes a relevant technique to detect changes in the

topological configuration of lyotropic nematic textures. It can be decisive for the recognition of the transition point connected to the biaxial phase. In fact, the obtained results are consistent with the occurrence of a biaxial phase between the two uniaxial nematic ones proposed by Saupe et al. [3,13]. Therefore, the existence of the biaxial nematic phase is yet an open question [15]. As a final remark, we mention that similar investigations [16] in a reentrant isotropic–calamitic (discotic) nematic phase transition are in progress.

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