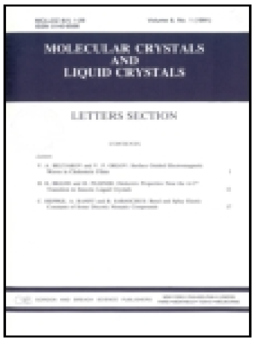

Molecular Crystals and Liquid Crystals

ISSN: 1542-1406 (Print) 1563-5287 (Online) Journal homepage: http://www.tandfonline.com/loi/gmcl20

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To cite this article: O. R. Santos, W. S. Braga, D. D. Luders, A. R. Sampaio, N. M. Kimura, M. Simões & A. J. Palangana (2016) Conoscopic image of a biaxial nematic phase in a sodium decyl sulfate – decanol – D_2O mixture, Molecular Crystals and Liquid Crystals, 632:1, 57-63

To link to this article: http://dx.doi.org/10.1080/15421406.2016.1185572

Published online: 17 Aug 2016.

MOLECULAR CRYSTALS AND LIQUID CRYSTALS
2016, VOL. 632, 57–63
http://dx.doi.org/10.1080/15421406.2016.1185572

Conoscopic image of a biaxial nematic phase in a sodium decyl sulfate – decanol – D_2O mixture

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ABSTRACT
The nematic phases of a lyotropic system *NadS*/ decanol/ heavy water are investigated using optical conoscopy and image processing. The phase diagram obtained from these lyotropic materials predicts the occurrence of a direct phase transition, which does not present the biaxial nematic phase, between the discotic (N_D) and calamitic (N_C) nematic phases. A biaxial nematic (N_B) phase is optically characterized and confirmed through conoscopic image, inside the biaxial range, between the two uniaxial nematic phases. Also, their respective transition points are determined by means of image processing. The N_B phase observed here is discussed as part of the nature of the micellar configuration of lyotropic materials which exhibit uniaxial nematic phases.

KEYWORDS
uniaxial nematic phases; biaxial nematic phase; conoscopic image

Introduction

The first conoscopic images featuring optically biaxial liquid crystals portrayed, a long time ago, a smectic *C* phase, reported by Taylor and co-workers [1], and a biaxial nematic phase (N_B) between the discotic (N_D) and calamitic (N_C) nematic phases, reported by Yu and Saupe [2]. This is an important line of research focusing on biaxial liquid crystal materials that continues to attract the interest of several researchers [3, 4]. In this way, conoscopic image results obtained in lyotropic nematic materials [2, 5] have shown and confirmed the existence of a biaxial nematic domain between uniaxial nematic phases [6 – 10]. Furthermore, the occurrence of a direct transition between the N_D and N_C phases, without a biaxial nematic phase between them, was also reported [11 – 13] in a phase diagram obtained from the decyl sulfate (*NadS*), decanol (*DeOH*) and D_2O mixtures. The optical characterization of the N_D - N_C phase transition made by these authors has been basically through optical microscopy observations. We highlight that the schlieren texture, characteristic of these uniaxial nematic phases, is not stable in N_D phase and, when observed with a polarizing microscope upon heating, it changes to a pseudo-isotropic texture [14]. On the other hand, the schlieren texture is more stable in N_B and N_C phases. This means that it is difficult to recognize the N_B to N_C transition point, by optical observations between schlieren textures [15, 16]. Thus, it is not

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an easy task into identify the occurrence of a direct transition between N_D and N_C phases via optical microscopy. This phase transition is provided in a phase diagram line for a lyotropic mixture of *NadS, DeOH* and D_2O compounds. In this context, we present an experimental investigation of the N_D - N_C phase transition by optical conoscopy.

The optical conoscopy [17, 18] has been used as a suitable tool to discriminate between uniaxial and biaxial liquid crystal phases [1, 2]. The conoscopic image produced by this optical technique in such homeotropic configuration as the uniaxial N_D phase presents two isogyres known as the Maltese Cross. When the N_D sample is rotated between crossed polarizers, the conoscopic image pattern does not change, which is an optical characteristic of this uniaxial material. The conoscopic image observed for a uniaxial sample in a planar configuration as the N_C phase between crossed polarizers occupies practically the entire field of view in the focal plane of the objective and consists of a very broad, fuzzy isogyre cross. In this way, when the N_C sample is rotated by a small angle, the isogyre splits and quickly leaves the optical microscopic field of view. Otherwise, for a biaxial N_B phase, the isogyres open when the sample is rotated, which is the fact used to identify the biaxial phase. In this work, these conoscopic images are presented and discussed near the N_D – N_C phase transition. The occurrence of a N_B phase, not provided in this lyotropic material, is clearly identified via conoscopic image between two uniaxial nematic (N_D and N_C) phases. In addition, their respective transition points are determined by means of image processing [19, 20].

Fundamentals

The nematic phases investigated in this work are provided in a phase diagram line [11] (Fig. 1) for a lyotropic mixture (concentration in weight percent) of decyl sulfate (*NadS : 35.0*), decanol (*DeOH : 7.0*) and deuterium oxide ($D_2O : 58.0$). These compounds are commercially available and have been obtained from Aldrich. The nematic phases are characterized by means of optical microscopy, image processing and optical conoscopy techniques. The nematic samples are prepared in sealed glass cells. The *1-2* plane of the sample is defined with

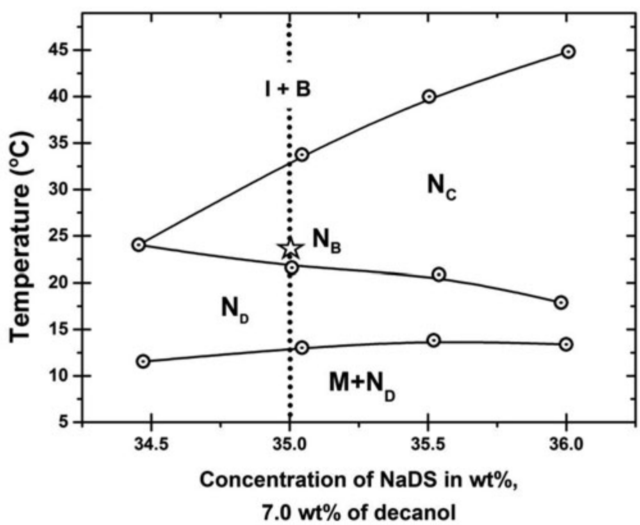

Figure 1. Phase diagram extracted from Ref.[11], N_D, N_B, N_C, *I*, *B* and *M* are the discotic nematic, biaxial nematic, calamitic nematic, isotropic, batonnets and middle soap, respectively. The new biaxial phase here reported, is shown by a star in this figure, between 21.1°C and 21.8°C.

1(2) axis parallel to the length (width) of the cells and *3* is the axis normal to the largest surface of the sample holder. We highlight that the N_D (N_C) phase presents positive (negative) optical birefringence and negative (positive) anisotropy of diamagnetic susceptibility [21]. The N_D phase is aligned in a homeotropic configuration through a magnetic field *(∼1T*) parallel to the 1-axis combined with rotations of the sample nematic around of the *3*-axis. Planar alignment of the N_C phase is obtained via magnetic field *(∼1T*) parallel to the *1*- axis.

The polarized light optical microscopy connected to the *CCD* camera (resolution of *1280×1024* pixels) is used to observe the nematic textures. Their respective nematic transition points are identified through image processing. A brief summary of the image processing technique is presented here. A more complete study of this technique is found in the references 19, 20]. The image frame of nematic textures is defined by the function *b(x,y)*,which represents the *32* bits true pixel color tone that ranges from *0* to *255* in red, green or blue colors (*RGB* image). The mean intensity of the color tones is given by $M_0 = 1/l_x l_y \int_0^{l_x} \int_0^{l_y} b(x, y)dxdy$ where *lx* and *ly* are the rectangular dimensions of the image frame. In this way, the *2*-rank statistical moments of the image frame are written by$M_2 = 1/l_x l_y \int_0^{l_x} \int_0^{l_y} [b(x, y) - M_0]^2 dxdy$. The root square $(M_2)^{1/2}$ is known as the mean square deviation (σ) [19, 20]. The parameter σ is determined (Delphi program) as a function of the temperature, for each *RGB* component of the nematic textures, in the range of nematic phases. We chose, the red color (the most sensitive one), which best identifies the phase transition point via parameter σ, in agreement with other experimental techniques [22, 23].

The optical conoscopy is obtained inserting an Amici-Bertrand lens positioned in the optical system of the polarized light microscope. The interference figure (conoscopic image) of light waves produced by the oriented N_D sample, in this optical configuration, is observed in the focal plane near the top of the objective. As mentioned before, this conoscopic image is known as the Maltese cross. Its center is called the melatope, corresponding to the optical axis [17, 18]. The conoscopic image characteristic of uniaxial N_C phase consists of a very broad, fuzzy isogyres cross [17, 18]. The isogyres open when the biaxial liquid crystal sample in thermal equilibrium is rotated from the °C position. This opening reaches a maximum value when the stage is rotated from the *45°C* position [1 – 4, 6 – 10]. We would like to emphasize that the opening of isogyres in uniaxial nematic phases subjected to thermal gradient has been observed [24, 25] while the temperature is changed (in either cooling or heating cycles) with a given rate. This conoscopic image (apparently biaxial) occurs when there is a flow-induced reorientation of the molecules (or micelles) caused by the fluctuation of the thermal expansion coefficient of nematic materials. The conoscopic image with characteristic biaxial, returns to uniaxial nematic (Maltese cross) configurations when the thermal gradient and material flow disappear [24, 25].

Results and discussion

The biaxial nematic range between the N_D and N_C phases is presented and discussed now via optical microscopy, optical conoscopy and image processing results. Figure *2* shows the nematic textures obtained upon heating from the N_D phase, near the $N_D \rightarrow N_C$ phase transition and N_C phase. The schlieren texture in N_D phase is transformed into a pseudo-isotropic texture (see Fig. *2(a)*). Note that this texture turns to a bright irregular domain with strong evidence of a N_D - N_B *(21.1°C*) phase transition [15, 20], as exhibited in Fig. *2(b)*, and, after the transition is completed, the N_B phase is characterized by the presence of smooth schlieren texture [15]. The schlieren texture characteristic of the N_C phase is shown in Fig. *2 (d)*. It is

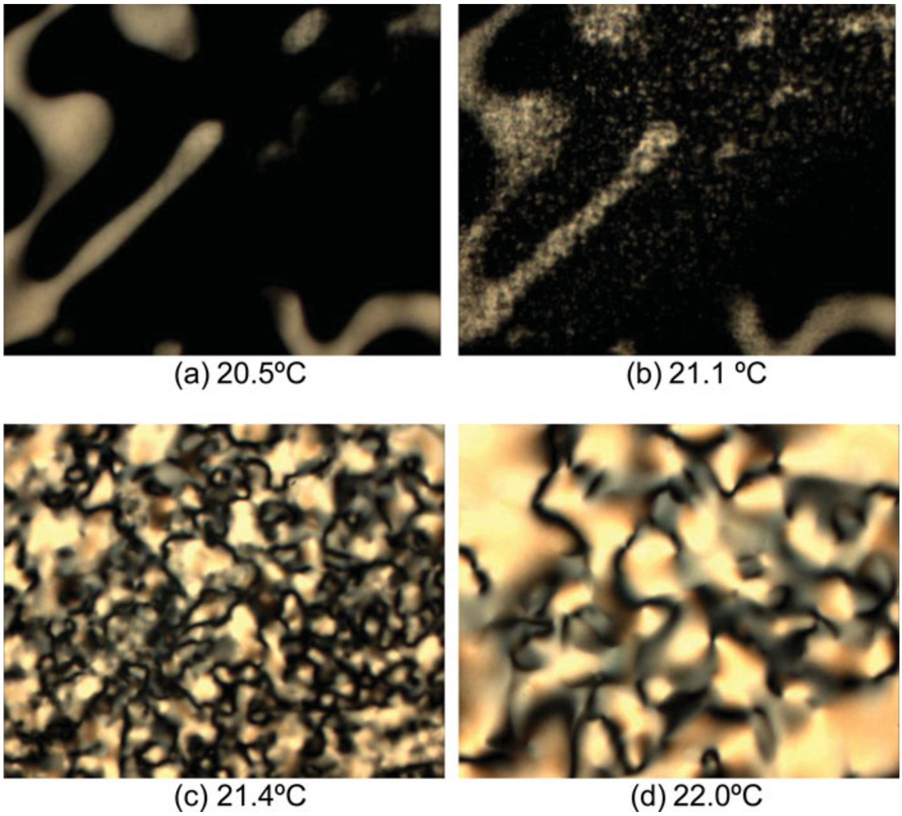

Figure 2. Nematic texures. (a) N_D (discotic), (b) N_D - N_B (biaxial) transition, (c) N_B and (d) N_C (calamitic) phases.

important to emphasize that the surface alignment, induced by the boundary conditions, of N_C *(*N_D*)* phase is parallel (perpendicular) to the biggest surface of the glass cells while that the surface alignment in the N_B phase is parallel to the *1* - axis and perpendicular to the *3* – axis [15, 16, 20, and 26]. Therefore, the recognition of the transition point N_B to N_C (*21.8°C*) by optical observations between schlieren textures is difficult to read [15, 16, and 20]. Moreover, our conoscopic image data, discussed below, confirm this phase sequence.

Figure *3* shows the conoscopic images obtained upon heating the discotic nematic near the $N_D - N_B - N_C$ phase transitions. The conoscopic images, for the N_D and N_B phases, are determined when the nematic sample ***(in thermal equilibrium)*** is rotated (anticlockwise) from the *0°* position to the *45°* position and then fixed at *45°*. The Maltese cross characteristic of the N_D phase is shown in Fig. *3(a)*. The isogyres open near the $N_D - N_B$ phase transition and becomes maximum in the biaxial nematic range and with the melatopes positioned along the *NE* and *SW* quadrants as depicted in Fig. *3*(*b*). The N_B phase is confirmed here by conoscopic image [2, 6]. The N_C uniaxial phase is optically negative where the conoscopic image consists [7, 8 and 26] of a very broad, fuzzy isogyre cross as exhibited in Fig. *3 (c)*. In this context, our conoscopic image results clearly indicate the occurrence of a biaxial nematic configuration from a N_D (homeotropic optical axis) to a N_C (planar optical axis) phase. The $N_D - N_B - N_C$ – transition points are determined by image processing [19, 20]. Figure *4* shows the mean square deviation (σ), obtained from nematic textures, versus temperature near these phase transitions for the heating cycle. Note that σ near the $N_D - N_B$ phase transition decreases as the temperature increases. This behavior of σ is consistent with the appearance of pseudo-isotropic schlieren texture of N_D phase. In the biaxial nematic range, σ presents a rapid growth,

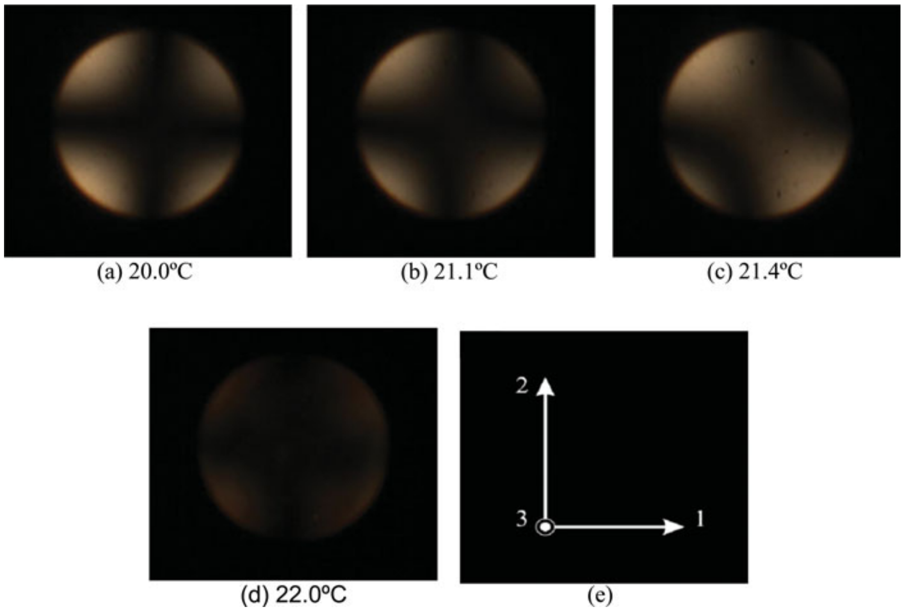

Figure 3. Conoscopic images of nematic phases. (a) N_D, (b) N_D - N_B, (c) N_B, (d) N_C, and (e) laboratory frame axis adopted [7].

followed by a slight decrease in direction of the N_C phase. The growth of σ at N_D - N_B transition reflects the appearance of bright irregular spots [15, 19, and 20] and subsequently disappears and gradually transforms to a smooth schlieren texture [15, 16] in direction of the N_C phase. The biaxial nematic phase occurs between two minimum values of σ, corresponding to N_D - N_B and N_B - N_C phase transitions in accordance with optical microscopy and optical conoscopy experiments.

In summary, we have carried out an optical conoscopy study near the N_D – N_C phase transition in a lyotropic mixture of decyl sulfate, decanol and D_2O. The occurrence of a N_B phase,

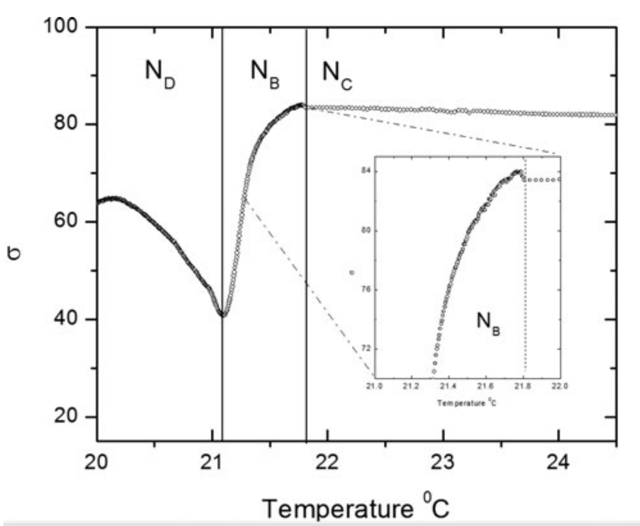

Figure 4. Mean square deviation (σ) versus temperature. The insert shows an enlarged scale, to better observe the N_B - N_C transition point.

optically characterized through conoscopic image, appears as an intermediate biaxial configuration between the N_D and N_C phases. We would like to emphasize that the N_B phase observed here is part of the micellar configuration of lyotropic materials. The optical conoscopy utilized here is indeed a very useful tool to discriminate between uniaxial and biaxial nematic phases. The image processing is a sensitive technique and is used to detect the uniaxial and biaxial nematic transition points. To the best of our knowledge, this is the first time that this N_B phase is optically characterized by means of conoscopic images [27]. As a final note, we mention that the experimental result presented and discussed in this work is a complementary study of the Saupe diagram [11] since it does not provide the N_B phase.

Acknowledgment

We are thankful to the Brazilian Agencies CAPES, CNPq (INCT of Complex Fluids), and Fundação Araucária (PR) for the financial support. We are also thankful to T.A. Pedreira (Brazil) for the careful proofreading of the manuscript.

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