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Biaxial phase and coexistence of the two uniaxial nematic phases in the system sodium dodecyl sulphate–decanol–D_2O

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The system sodium dodecyl sulphate (SDS)/decanol (DeOH)/water presents, with temperature increase, an unusual phase transition between a stable biaxial phase and a coexistence region of the two uniaxial nematic phases (discotic N_D + cylindrical N_C). This has been detected previously by several methods for a sample with water/SDS molar ratio M_w = 36. Here, this system is investigated changing the ratio M_w to 32, where previously the coexistence region was reported after a discotic N_D phase, without the biaxial phase. We report now the existence of a biaxial N_B in the temperature range of ~2°C, defining for both values of M_w and the phase sequence as N_B – (N_D + N_C) – N_C. The change in temperature is followed by conoscopic and orthoscopic optical techniques and also analysed through the curve obtained by the technique of digital image processing of the textures, which reveals a continuous transition N_D – N_B – (N_D + N_C). While the biaxial phase is stable for at least 10 hours, in a reproducible way, the coexistence region evolves with time, and drops of the discotic phase grow immersed in a N_C matrix. Results are explained in terms of recent theories dealing with stabilisation of mixtures of cylinders and discs.

Keywords: uniaxial lyotropic nematic; biaxial lyotropic nematic; conoscopy; digital image processing; phase coexistence

1. Introduction

Sodium dodecyl sulphate (SDS) in water solution was well studied in lyotropic phases with long range order, since the 1960s,[1] but the discovery of the nematic domain in the SDS system was made by a group in São Paulo, Brazil, in the end of the 1980s,[2,3] after the discovery of the nematic biaxial phase with potassium laurate (KL).[4] The dependence of the SDS phase diagram on the molar ratios [decanol]/[SDS] = M_d and [water]/[SDS] = M_w was stressed.[3] For M_w = 45, there is a sharp first-order transition between the discotic (N_D) and the cylindrical (N_C) nematic phases at M_d ~0.38.[3,5]

Existence of biaxial islands immersed in coexistence regions of the two uniaxial nematic phases in the lyotropic system SDS/D_2O/decanol (DeOH) has been later discovered in studies with ^2H NMR (nuclear magnetic resonance spectroscopy of deuterium) made by Quist in Lund, Sweden,[6] about 20 years ago. But the behaviour of this system was quite different from that of the one discovered by Saupe in the KL/D_2O/DeOH system, which showed the biaxial phase between the two uniaxial phases, with second-order transitions, a predicted behaviour, although inexistent among thermotropic liquid crystals.[7,8] No explanation existed for the reported phase diagram of the SDS system, which indicated first-order transitions, common in the complex phase diagrams of lyotropic systems. The biaxial islands immersed in a coexistence region N_C + N_D, signature of a first-order transition, have been found in specific molar regions of the SDS phase diagram.[6] A review on the micelles forming biaxial lyotropic nematic phases, focused on the indication of changes in micellar form, was published by one of us in the volume dedicated to Saupe.[9]

Figure 1 gives an adaptation of the SDS phase diagrams from literature, including the biaxial islands reported by Quist.[6] Figure 1(a) is a conventional ternary phase diagram of H_2O/SDS/DeOH, at 25°C, showing the isotropic I (made by spherical micelles), hexagonal H_α (made of cylindrical micelles with two-dimensional order) and lamellar L_α (bilayers with one-dimensional order) domains defined by Ekwall [1] and the nematic domain obtained by Amaral [3] and Quist. [6]. Lyotropic uniaxial nematic phases are made of finite micelles, N_C phase of prolate form and N_D phase of oblate form; the biaxial N_B phase is subject to debates. Results obtained with partially deuterated samples were transformed by Quist in wt% as if obtained with water, to make the comparison possible.[6] Figure 1(b) is a phase diagram in the function of molar ratio D_2O/SDS (called M_w in this article), varying the temperature, for a fixed DeOH/SDS molar ratio 0.324.

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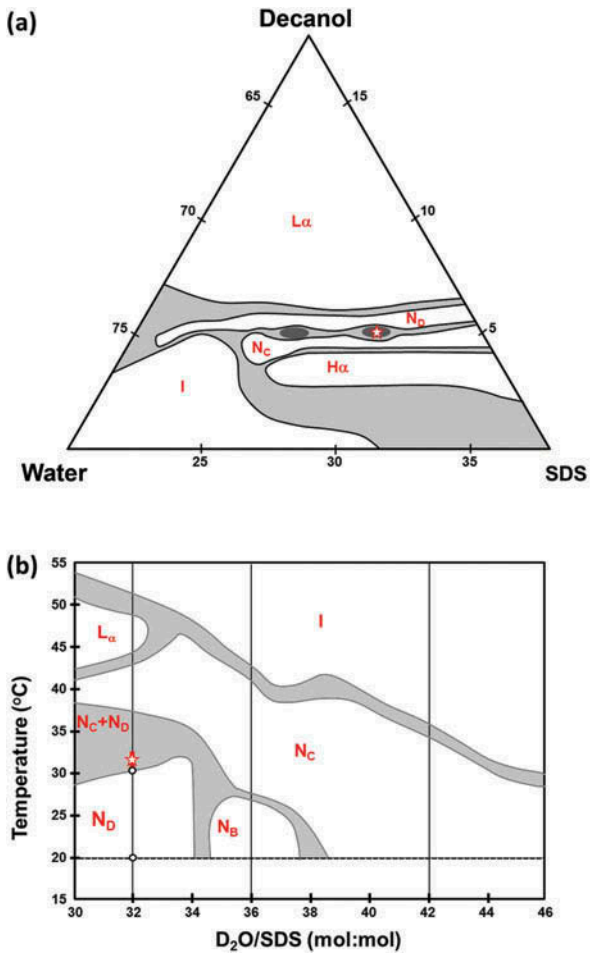

Figure 1. (colour online) Adaptation of the SDS phase diagram from literature.
(a) This is a conventional ternary diagram of H_2O/SDS/decanol, at 25°C, in wt%, as explained in the text, showing the two biaxial islands N_B reported by Quist.[6] A star in Figure 1(a) marks the confirmation of the biaxial phase by our group [11, 12]; (b) This is an adaptation of Quist's [6] phase diagram in the function of molar ration D_2O/SDS and temperature, as explained in the text. The lines marked at M_w = 32, 36 and 42 were explored by the work of our group.[10–12] Quist's results were confirmed along these lines, except for the discovery of a new biaxial phase at M_w = 32 here reported, shown by a star in this figure, between 30°C and 32.5°.

The SDS system was recently investigated by measurements of the refractive indexes in the coexistence region and in the biaxial island,[10,11], and the results show the differences in phase transitions between the SDS and KL systems. Later, polarised optical microscopy was used to obtain texture and conoscopy results to characterise the uniaxial phases and also their coexistence region as well as the biaxial phase in the SDS system,[12] with the same methods used to study the KL system.[13,14] This study confirmed the existence of the sequence N_B – (N_D + N_C) – N_C for M_w = 36 (with M_d = 0.324) [12] and the existence of the coexistence region also for M_w = 32, following the published phase diagram by Quist.[6] A star in Figure 1(a) marks the confirmation of the biaxial phase by our group.[11,12]

Here, this SDS system is investigated with M_w = 32, where previously the coexistence region was reported after a discotic N_D phase,[6,10] without the biaxial phase. The focus here is the search for a biaxial phase between the reported N_D phase and the coexistence region (N_D + N_C). A new biaxial phase, not previously reported, and here presented, was now localised and is shown by a star in Figure 1(b). The assignment of L_α phase in Figure 1(b) was made by analysing the previous phase diagrams, since the techniques here employed were not focused on the detection of the lamellar phase.

2. Materials and methods

Commercially available compounds SDS, DeOH and D_2O were obtained from Merk and Aldrich laboratories (St. Louis, MO, USA), with purity > 99%. The samples were prepared in sealed flat rectangular glass cells,[12] from Hellma GmbH & Co (Müllheim, Germany; length 44 mm, width 12.5 mm and 1.0 mm of light path), at a room temperature of 23°C, above the Krafft crystallisation temperature. Thus the prepared samples will remain in good conditions for at least a month, but samples investigated were at maximum 1 week old. The orientation of the samples is discussed in the presentation of the results.

These samples are characterised using a commercial polarised optical microscope (POM) Leica DMLP,[12,13] connected to a CCD camera to register the images. Two magnets of neodymium were adapted to the sample thermostat, in order to have a magnetic field of 1.3 kgauss in the direction of the sample length.

With Amici-Bertrand lens inserted for conoscopy, the interference figure (Maltese cross) is observed in the focal plane near the top of the objective. The isogyres open when the biaxial nematic sample in thermal equilibrium is rotated from the 0° position, and this opening reaches a maximum value when the stage is rotated to the 45° position. For this reason, the sample is placed, and observed, with its length in the position 45°. A gypsum plate (red 1 lambda plate) is inserted into the optical path to obtain the sign of the axiality, through the colours (yellow/blue) of the quadrants, for both biaxial and uniaxial nematic phases.[12,15,16] It is well known that the uniaxial lyotropic nematic phase N_C presents negative optical birefringence and positive anisotropy of diamagnetic susceptibility, while N_D is optically positive and magnetically negative.[15]

The orthoscopic textures are seen with amplification 50x and 100x and are also analysed through image processing. Only a brief summary of the image processing technique is presented here. A more complete study of this technique is found in the references.[17–19] So, let us consider the function $b(x, y)$, which represents the 32 bits true pixel colour tone that ranges from 0 to 255 in red, green or blue colours (RGB image). The mean intensity of the colour tones is given by

$$M_0 = \frac{1}{l_x l_y}\int_0^{l_x}\int_0^{l_y} b(x,y)\mathrm{dxdy}$$

where l_x and l_y are the rectangular dimensions of the image frame. In this way, the 2-rank statistical moment of the image frame is written by

$$M_2 = \frac{1}{l_x l_y}\int_0^{l_x}\int_0^{l_y} [b(x,y) - M_0]^2\mathrm{dxdy}$$

The root square $(M_2)^{1/2}$ is known as the mean square deviation (σ). The parameter σ is determined (Delphi program) as a function of the temperature, for each RGB component of the nematic textures. In this texture study, we chose red colour (the most sensitive one in transitions involving the dark pseudo-isotropic texture of N_D phases) which best identifies the phase transition point via parameter σ, in agreement with other experimental techniques. [14,18,19]

The sample temperature was controlled by a water-flow regulator (Brookfield TC-502), yielding a temperature stability of ± 0.01°C. Because the coexistence region evolves with time,[12] the thermal treatment is discussed in the presentation of the results.

3. Results

As mentioned in our previous study,[12] all published phase diagrams by Quist start at 20°C,[6] because below that temperature, crystallisation occurs. When the sample is cooled into the crystalline state, the nematic phase is not easily recovered on heating. For that reason, results here presented were also investigated above 20°C.

The published phase diagram by Quist [6] in Figure 3(a), for M_d = [DeOH]/[SDS] ~ 0.324, gives for M_w = 32 the sequence N_D – coexistence (N_D + N_C) – N_C, while for M_w = 36 a biaxial island exists before the coexistence region. The biaxial island and the two coexistence regions were confirmed in our previous study with POM,[12] but the temperature interval between N_D and the coexistence region, for M_w = 32, was not discussed in[12] and is being further investigated in detail here, focusing the search for a biaxial phase.

Quist's work with NMR covered phase diagrams with different compositions, in the isothermals at 20°C and 30°C, and biaxial islands were found at 20°C. Temperature-dependent phase diagrams were reported for two M_d values, but his temperature resolution was 1°C, with an equilibration period of ~ 10 minutes after each temperature change. With M_w = 32, there are only two points for N_D at 20°C and 30°C; then a point around 32°C is already in the coexistence region (N_D + N_C).[6]

In the study now reported, the temperature resolution is 0.01°C, with variation of 0.3°C, and an equilibration time of 10 minutes. The orientation of the biaxial samples combined surface orientation of the N_D phase for 7 days at 22°C, followed by orientation by a magnetic field of 1 tesla in the direction of the longer axis of the rectangular sample. The oriented sample was then placed in the microscope, with its length in the position 45° of the stage, with the small magnetic field to keep its orientation. The cell was heated until the interference image typical of conoscopy is formed. When biaxiality was detected by conoscopy, the sample was left stabilising for 10 hours and remained stable. Reproduction of the biaxial phase was checked in more than 15 runs, with several samples. The conoscopy results for the phase sequence are: N_D from 20°C to 30°C – N_B from 30°C to 32.5°C – (N_D + N_C) from 32.5°C.

Figure 2 shows the obtained conoscopy results; the sequence is N_D – N_B with increasing temperature. The biaxial phase is optically positive (N_B^+); the signal corresponds to the relative values of the three different refractive indices. Upon entrance on the coexistence region (N_D + N_C), the conoscopic image disappears. Conoscopy image of N_C phase image is rather dark (not shown) and consists of a very broad, fuzzy isogyres cross, as shown in the previous work.[12]

The textures were observed as direct images and at the same time in the curve of the σ parameter, which gives the mean square deviation of the mean intensity of the red-colour tones, through digital image processing technique. In this case, the measurements required a continuous temperature variation, at a rate of 0.04°/minute, so that it takes more than 10 hours to span the temperature interval 20°C–50°C.

Figure 3 shows the characteristic texture images upon heating, while Figure 4 shows the corresponding σ curve. The texture images in Figure 3 do differentiate and well characterise all the different phases, in the sequence N_D – N_B – (N_D + N_C) – N_C – Lamellar L_α, as defined by Quist.[6] The intensity curve of

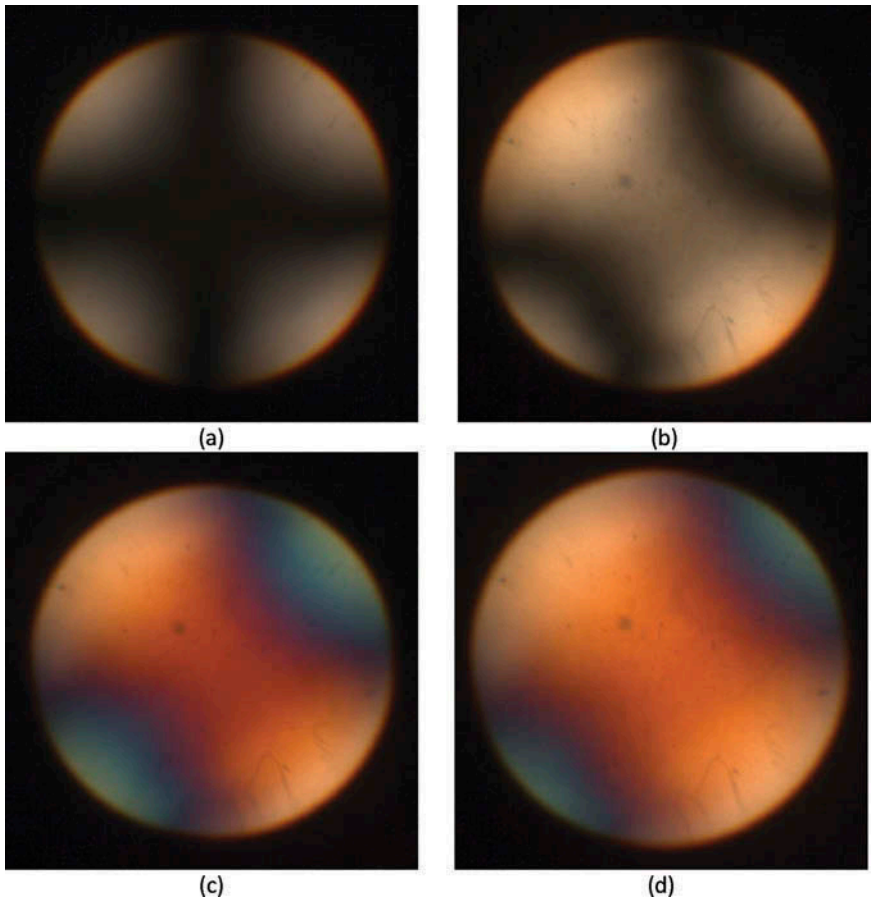

Figure 2. Conoscopic images of nematic phases. (a) N_D (discotic nematic) at 25°C, (b) N_B (biaxial nematic) at 30.5°C (without the gypsum plate), (c) N_B^+ (biaxial positive nematic, with the gypsum plate) at 30.5°C and (d) at 31.5°C.

Figure 4 shows a marked increase in σ at ~30°C, defining clearly a continuous transition from the N_D phase to the biaxial phase here reported. This good definition in the intensity curve arises from the defined change in average colour tone between the texture of the pseudo-isotropic N_D phase and the texture of the biaxial phase. The other transitions are not so well defined in the intensity curve of Figure 4 because in their textures, seen in Figure 3, the change in average intensity of colour tones is relatively small, with similar light/dark proportions, which define the behaviour of the curve of the parameter σ. An insert in Figure 4 shows an expansion of the region, indicating changes at 32.5°C, 34.7°C and 36.9°C, related to the phase sequence $N_B - (N_D + N_C) - N_C$.

In the SDS case, the three techniques employed here (conoscopy, textures images and digital analysis) are necessary to completely characterise the phase transitions.

When the system is kept in the temperature of the coexistence region ($N_D + N_C$) for a long period of time, formation of defined drops of uniaxial phases, in which size grows with time, is observed, as shown and discussed in the previous work.[12] There the evolution of the coexistence region was seen for a sample with M_w = 36, at the initial time and after 10 hours.

Figure 5 shows the evolution with time of a sample with M_w = 32, at a fixed temperature of 33.5°C, in the coexistence region. Figure 5(a) shows the texture observed after 5 hours, with the gypsum plate, and regions with opposed optical sign are clear, showing coexistence of drops of the two uniaxial phases. Figure 5(b) shows the same texture without the gypsum plate. Figure 5(c) shows the texture after 10 hours, with a matrix of oriented N_C phase (clear texture with the stage at 45° position, direction of the magnetic field) and drops of N_D phase, of the order of 100 micra, and this assignment is confirmed in Figure 5(d), turning the stage to 0°.

To be noticed that the time period in which the sample stayed in the coexistence region for the measurements in Figures 3 and 4 is about 2.5 hours, so

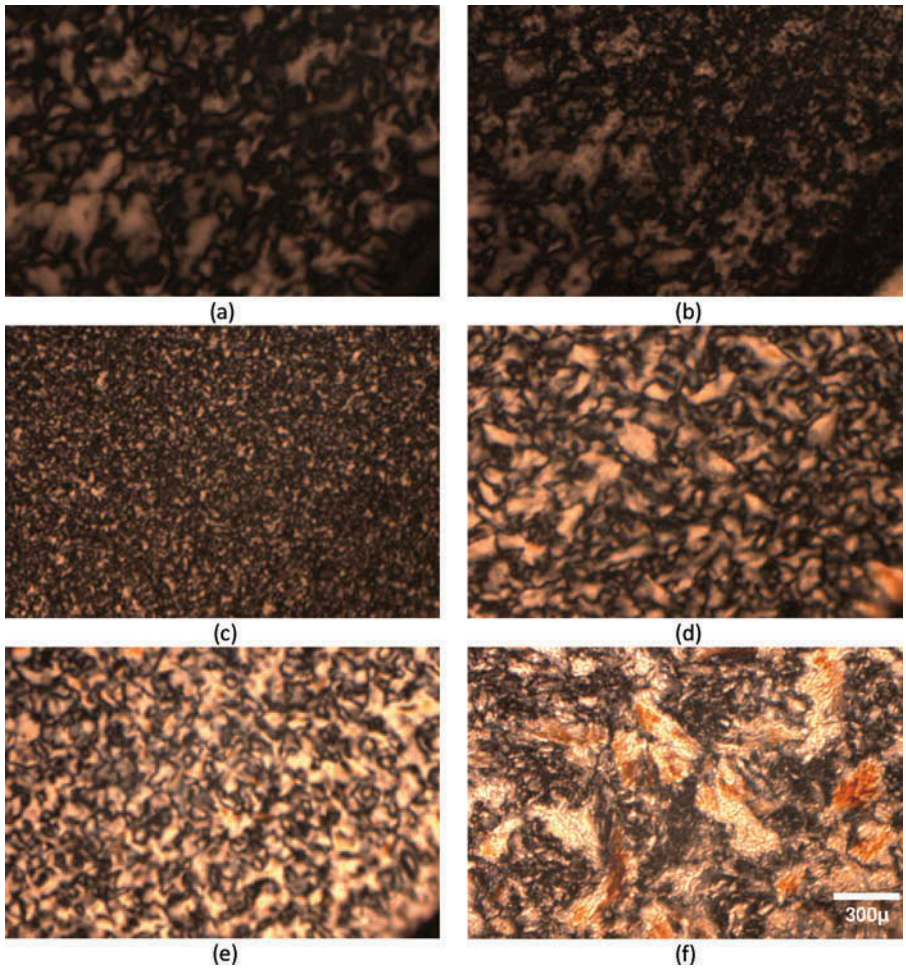

Figure 3. (colour online) Nematic textures. (a) N_D phase – 25°C, (b) transition N_D – N_B phase – 29.5°C, (c) N_B phase – 31 °C, (d) coexistence (N_D + N_C) phases – 34°C, (e) N_C phase – 35.5°C, (f) Lamellar phase Lα – 45°C.

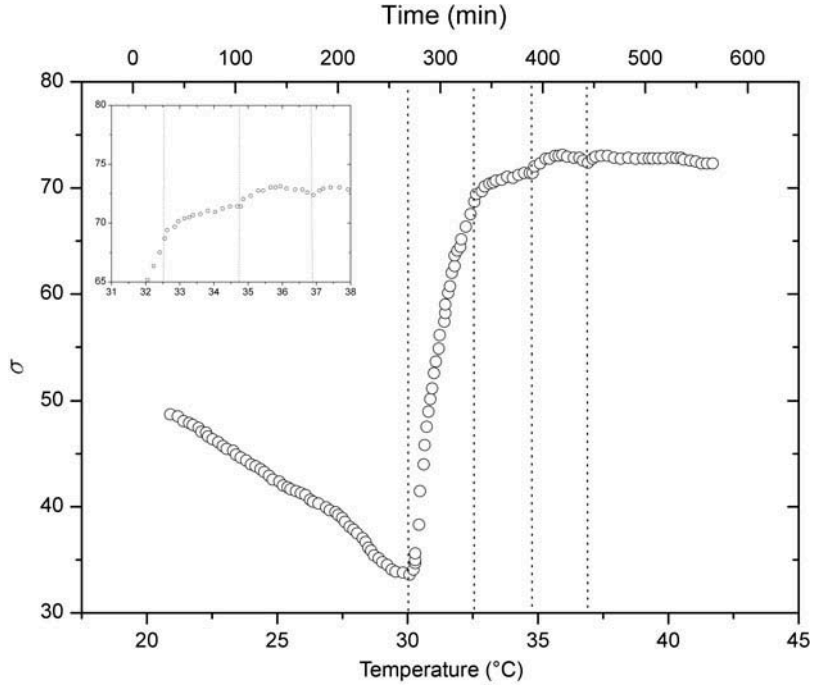

Figure 4. Mean square deviation (σ) versus temperature, showing the continuous increase of the curve from the N_D to the N_B phase at 30°C. The insert shows an enlarged scale, to better observe the other transitions.

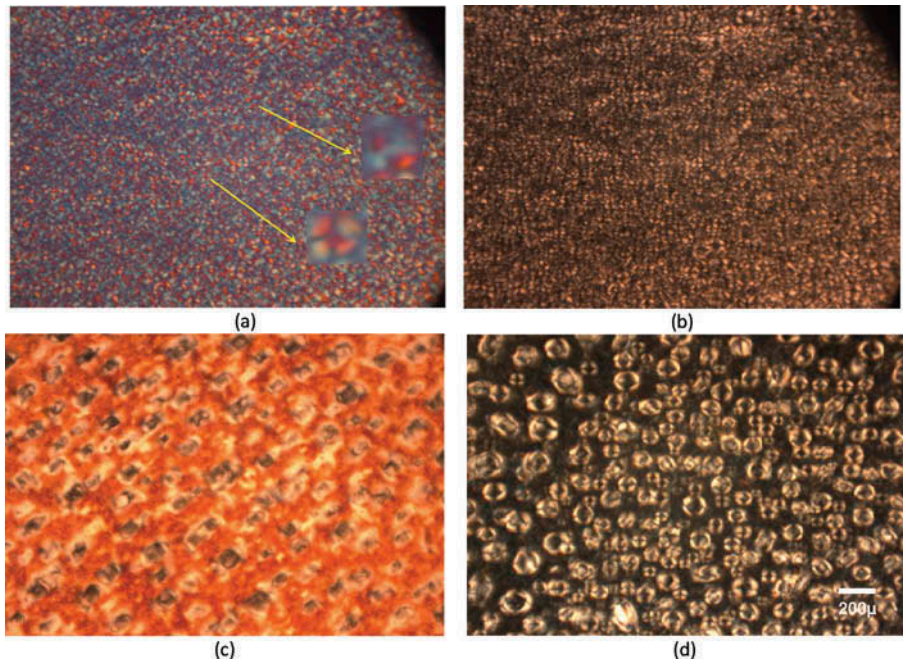

Figure 5. (colour online) Coexistence region at a fixed T = 33.5°C, along the time: (a) and (b) – after 5 hours, in (a) with gypsum plate, showing regions with different uniaxial signal and (b) without the gypsum plate; (c) and (d) after 10 hours, in (c) with stage at 45° and (d) with stage at 0°.

that the evolution with time competes with the temperature evolution. Results indicate that the time evolution is not the same when the temperature varies slowly and when it is kept constant for many hours. It is however outside the scope of the present work to study the detailed time behaviour of the system.

The new results here reported give evidence that the characteristic transition sequence for the SDS system is – N_B – coexistence (N_D + N_C) – N_C, not only for M_w = 36, as previously reported,[6,12] but also for M_w = 32.

4. Discussion

The review paper on micelles in biaxial phases stressed the open problems for understanding lyotropic nematic phases.[9] The SDS system here reported as the very unusual sequence of phases N_B – coexistence (N_D + N_C) – N_C. Landau theories predict two possibilities,[20] a direct transition N_D – N_C, or a sequence of second-order transitions N_D – N_B – N_C, as observed in the KL system. The direct transition is expected to lead to spinodal decomposition into the two uniaxial phases, without an intermediate biaxial phase. All attempts in several published papers to produce a biaxial thermotropic phase with mixtures of the two uniaxial phases, with two different thermotropic compounds, have failed, but the direction of mixtures is actively investigated.[7,8,21]

The SDS system, showing both a biaxial phase and a coexistence region, is particularly an interesting system to investigate the formation of biaxial phases. This unique behaviour is revealed by different techniques, such as NMR,[6] measurements of the refractive indices and with POM, in the previous paper and in the present article.[10–12] The possible explanation for such behaviour has started to be built in these previous works, and a step further is presented here.

The biaxial phase might be understood from a theoretical model worked out for mixtures of cylinders and discs, in which explicit relations between the quadrupoles and the anisometries of the two shapes have been obtained.[22] Demixing of the two forms is prevented if the two uniaxial forms are transforming in one another, as proposed in the model worked out for a single micelle on basis of the elastic bending energy connected with molecular shapes of surfactant and cosurfactant.[23] The condition is that micelles are changing form at a rate higher than micelle diffusion. The change in shape and symmetry as a function of concentration and DeOH addition is clearly understood, in the face of the phase diagrams of lyotropic systems. It was later shown that a two-temperature formalism to mimic the presence of two distinct relaxation times gives theoretical basis for a stable biaxial phase in the mixture.[24,25]

The unusual phase transitions with temperature are possible to be understood taking into account the basic

question of changes in molecular chain length and volume with temperature.[9] Hydrocarbon chains are characterised by a chain length contraction with increasing temperature, expressed by a negative temperature coefficient, which holds only in the chain direction, while the paraffin volume as a whole has a small positive temperature coefficient.[26] While the direction of the chain is contracting with increasing temperature, its other dimensions are expanding; thus, cylinders and discs become more anisotropic with increasing temperature. In a cylinder, two dimensions are contracting with temperature, while in discs only one dimension contracts, so that cylinders grow more quickly with increasing temperature. The population of forms and the corresponding average quadrupoles therefore suffer distortions as a function of temperature, inducing the change from N_D to N_C with increase in temperature.

The results with the SDS system indicate that the biaxial islands correspond to compositions with specific fractions of cylinders and discs, in a mixture which equilibrates the transformation of the two forms in one another, as occurs in chemical reactions in equilibrium, resulting a stable biaxial phase. This work shows that the temperature interval of the biaxial phase depends on the M_w value, which might be related to the proportions of cylinders and discs in the mixture.

The change in asymmetry and quadrupoles of the two forms with increasing temperatures breaks the equilibrium conditions, and the system enters in the region where the two forms tend to separate, with a time-dependent spinodal decomposition.

5. Conclusion

It was clearly shown by optical techniques here presented that in the system SDS/DeOH/heavy water, for M_w = 32, a biaxial nematic phase exists in the temperature interval 30°C–32.5°C and is stable for at least 10 hours. The transition $N_D - N_B$ is continuous as revealed by the intensity curve of the σ parameter. At higher temperatures, the system enters in a coexistence region of the two uniaxial phases and suffers spinodal decomposition if kept at 33.5°C for about 10 hours. Longer time periods have not been explored yet. For M_w = 36, the same transition $N_B - (N_D + N_C)$ occurs, but at 26°C. These results show the possibility of design of mixtures able to display biaxial phases, under controlled conditions.

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