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## Liquid Crystals

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## Phase behaviour of *n*-alkyl- and bi-alkylbenzenesulphonates Nematic lyotropics from double chain surfactants

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The lyotropic mesophases in binary systems of surfactants in water: *n*-alkylbenzenesulphonates ( $C_8$ - $C_{12}$ ), two chain  $C_{1,2}$ -surfactants, and dodecylbenzenesulphonic acid, were investigated. The micellar properties were examined by conductometry and viscosimetry. The phase diagrams were determined using crossed polarizers,  $^2H$  NMR spectroscopy and polarization microscopy. Besides lamellar and inverse cubic phases, new nematic lyotropic phases have been found, presenting precursors for the lamellar phases, and exhibiting very fast alignment in a magnetic field.

### 1. Introduction

While the phase equilibria and the phase structures are often examined in multicomponent systems with alkylbenzenesulphonates, mostly in the mixtures with alcohol and water, this has not been the case for binary mixtures of alkylbenzenesulphonates and water. The phase behaviour and phase equilibria have been examined in ternary systems for several anionic [1] and cationic [2] surfactants. Since the membranes of living cells have a lamellar liquid crystal phase, whose preferential model systems include bilayers and unilamellar liposomes [3], the phase behaviour in bi- and multicomponent systems consisting of water, amphiphile, salts and/or drugs, are considered to be very interesting to investigate. Double chain surfactants in water are an important model for the investigation of phospholipid membrane behaviour.

In the present paper the formation of micelles and liquid-crystalline phases in binary systems of single and double chain ionic surfactants has been investigated. The following were used as the single chain surfactants: sodium *n*-alkylbenzenesulphonates: octyl (SOBS), nonyl (SNBS), decyl (SDeBS), undecyl (SUBS) and dodecyl (SDBS). The formation and the structures of mesophases in aqueous solution are presented in this paper for sodium 1'-(4)dodecylbenzenesulphonate (4SDBS) and sodium 1'-(6)dodecylbenzenesulphonate (6SDBS), which are rather small surfactant molecules.

### 2. Experimental

#### 2.1. Materials

The analytically pure samples of sodium *n*-alkylbenzenesulphonates were obtained by courtesy of the Henkel Co., Germany (SOBS, SDeBS, 4SDBS and 6SDBS), and the Hüls Co., Germany (SNBS, SUBS and SDBS). The dodecylbenzenesulphonic acid

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(HDBS) was used from Ventron-Alfa, Germany, without further purification. The  $^{13}\text{C}$  NMR characterization of HDBS has shown that it is an isomeric mixture of  $\text{C}_8$ – $\text{C}_{14}$  single and double chain compounds. Doubly distilled water was used for all of the experiments.

## 2.2. Methods

The liquid-crystalline phases were characterized by  $^2\text{H}$  NMR (JEOL FX-90 Q with an external lithium lock). The samples for phase diagrams and  $^2\text{H}$  NMR measurements were prepared with the solvent five per cent  $\text{D}_2\text{O}$  in  $\text{H}_2\text{O}$ . The characteristic  $^2\text{H}$  NMR spectra recorded for nematic, lamellar and inverse cubic phases are presented later. Phase diagrams were determined by using optical microscopy with crossed polarizers. The samples for phase diagrams were prepared by weighing the pure substances (w/w in 5 per cent  $\text{D}_2\text{O}/\text{H}_2\text{O}$ ), and by keeping the samples in a thermostat ( $0.1^\circ\text{C}$ ) for days in order to equilibrate them. The microscopic determination of the phase textures were performed on a Zeiss polarization microscope with a Mettler FP 82 hot stage and FP 80 central processor. The samples were prepared between object and cover glass or in microslides (Camlab) with a pathlength of 0.2 mm. The characterization of the micellar properties of the surfactants was made by conductivity measurements using the Wayne–Kerr conductivity bridge. The viscosity of 6SDBS solutions in the isotropic phase was determined with a Chempro PAAR OCR-D oscillating capillary rheometer and densitometer.

## 3. Results and discussion

### 3.1. Micellar properties

The characterization of the micellar properties of surfactants in low concentrations was made by conductometry and viscosimetry. The CMC and transition concentrations ( $c_t$ ) [4, 5] of the surfactants were determined by conductivity measurements. The values obtained are listed in the table. It is obvious that the agreement of tabulated values with corresponding results obtained by other techniques [6–10] is quite good.

The free energy of micellization ( $\Delta G_{\text{CMC}}$ ), as well as the free energy of micellar transition ( $\Delta G_{c_t}$ ) increases with the length of the paraffin chain as expected [11], showing middle values for 4SDBS and 6SDBS between SNDS and SDeBS corresponding to the chain lengths. It also seem reasonable that  $\Delta G_{c_t}$  has lower values than  $\Delta G_{\text{CMC}}$ .

Both viscosity and density are low for 6SDBS. Due to the increase in viscosity within the region 7–10 per cent of surfactant, the beginning of liquid crystal formation

Micellar parameters of alkylbenzenesulphonates.

Surfactant	CMC/mol dm $^{-3}$	$\Delta G_{\text{CMC}}/\text{kJ mol}^{-1}$	$c_t/\text{mol dm}^{-3}$	$\Delta G_{c_t}/\text{kJ mol}^{-1}$
SOBS	$1.3 \times 10^{-2}$ ( $1.4 \times 10^{-2}$ ) [7, 10]	10.94	$7 \times 10^{-2}$	6.70
SNBS	$6 \times 10^{-3}$	12.90	$1.37 \times 10^{-2}$	10.81
SDeBS	$1.16 \times 10^{-3}$	17.03		
SUBS	$4 \times 10^{-4}$	19.71		
SDBS	$4.4 \times 10^{-4}$	19.47	$9.4 \times 10^{-4}$	17.56
4SDBS	$1.95 \times 10^{-3}$	15.73	$4 \times 10^{-3}$	13.91
6SDBS	$2.6 \times 10^{-3}$ ( $2.5 \times 10^{-3}$ ) [8]	14.81	$7.5 \times 10^{-3}$	12.47

can be assumed. It will be shown later that this precursor phase is a nematic. The formation of spherical and disc-like micelles even in the CMC and  $c_t$  region of surfactant concentration can be assumed, respectively. Such an assumption was easily confirmed by the microscopic and time-resolved  $^2\text{H}$  NMR measurements presented in this paper. The aggregation numbers of the micelles calculated from the experimental data in the paper of Magid *et al.* [9] show a linear dependence on the surfactant concentration, asymptotically approaching the value of 25 [8] for the CMC of 6SDBS. At the critical point of 7 per cent ( $0.21 \text{ mol dm}^{-3}$ ), the aggregation number of 6SDBS can be taken to be 78 (from linear extrapolation of the data [9]). By calculating the aggregation number of surfactant ( $n$ ) from

$$c = \frac{(c_0 - \text{CMC})N_A}{1000n},$$

the value for the CMC at  $30^\circ\text{C}$  was used from this work,  $c_0$  is the total concentration of surfactant and  $c$  is the directly dependent value on micellar size.

### 3.2. Phase diagrams

All the systems used to determine the phase diagrams were prepared with 5 per cent  $\text{D}_2\text{O}$  in  $\text{H}_2\text{O}$ , so that the samples could also be used for the  $^2\text{H}$  NMR measurements. The samples were equilibrated for each temperature before the measurements were made, and the results are presented in figure 1 for  $n$ -compounds. All of the phase diagrams show a shift of isotropic and solid crystal regions (I + C) from  $n\text{-C}_8$  to  $n\text{-C}_{12}$  towards smaller concentrations and higher temperatures. The isotropic and liquid crystal (I + L) and the liquid crystal (L) regions are generally followed by a three phase transition region (T): isotropic and liquid crystal and solid crystal between the liquid and solid crystal phase regions. All of the liquid-crystalline phases are lamellar. This was observed by crossed polarizers, by polarization microscopy and confirmed by  $^2\text{H}$  NMR spectroscopy. The phases in the isotropic and solid crystalline region (I + C) near the transition three phase region (I + C + L) show birefringent crystalline phases and a broadened peak in the  $^2\text{H}$  NMR spectrum with a very small quadrupole splitting ( $\delta = 3.8 \text{ Hz}$ ).

The characteristic phase diagrams for binary mixtures of double-chain surfactants 4SDBS and 6SDBS are presented in figures 2 and 3, respectively. There are no C regions between  $20\text{--}80^\circ\text{C}$ . The isotropic region for 4SDBS is rather wide; increasing the surfactant concentration of the regions of (I + L) and lamellar (L) phases have been observed. The phase diagram of 6SDBS exhibits a smaller isotropic region than 4SDBS, a large lamellar region in higher concentrations of 6SDBS, and nematic as well as a mixture of nematic and lamellar phase regions in between. At lower temperatures the regions appear as two phase isotropic and lamellar (I + L). The transition region in the 20 per cent sample shows at  $30^\circ\text{C}$  several different splittings. This sample shows at  $45^\circ\text{C}$  two different  $^2\text{H}$  NMR doublets  $\Delta^2\text{H}_i$  of 485 Hz and  $\Delta^2\text{H}_j$  of 987 Hz belonging to lamellar and nematic phases, respectively. The isotropic solution on the top is well separated in this sample from the liquid-crystalline phase in the bottom of the tube, although the system has been equilibrated for several days, and exhibits a sharp isotropic peak and a small reminder of lamellar phase. At higher temperatures there is only a nematic phase. This nematic phase is similar to that in the narrow region in the HDBS–water systems. Neither isotropic nor lamellar phase spectra are observed by  $^2\text{H}$  NMR in this region (for the detailed description of  $^2\text{H}$  NMR spectra see § 3.4).

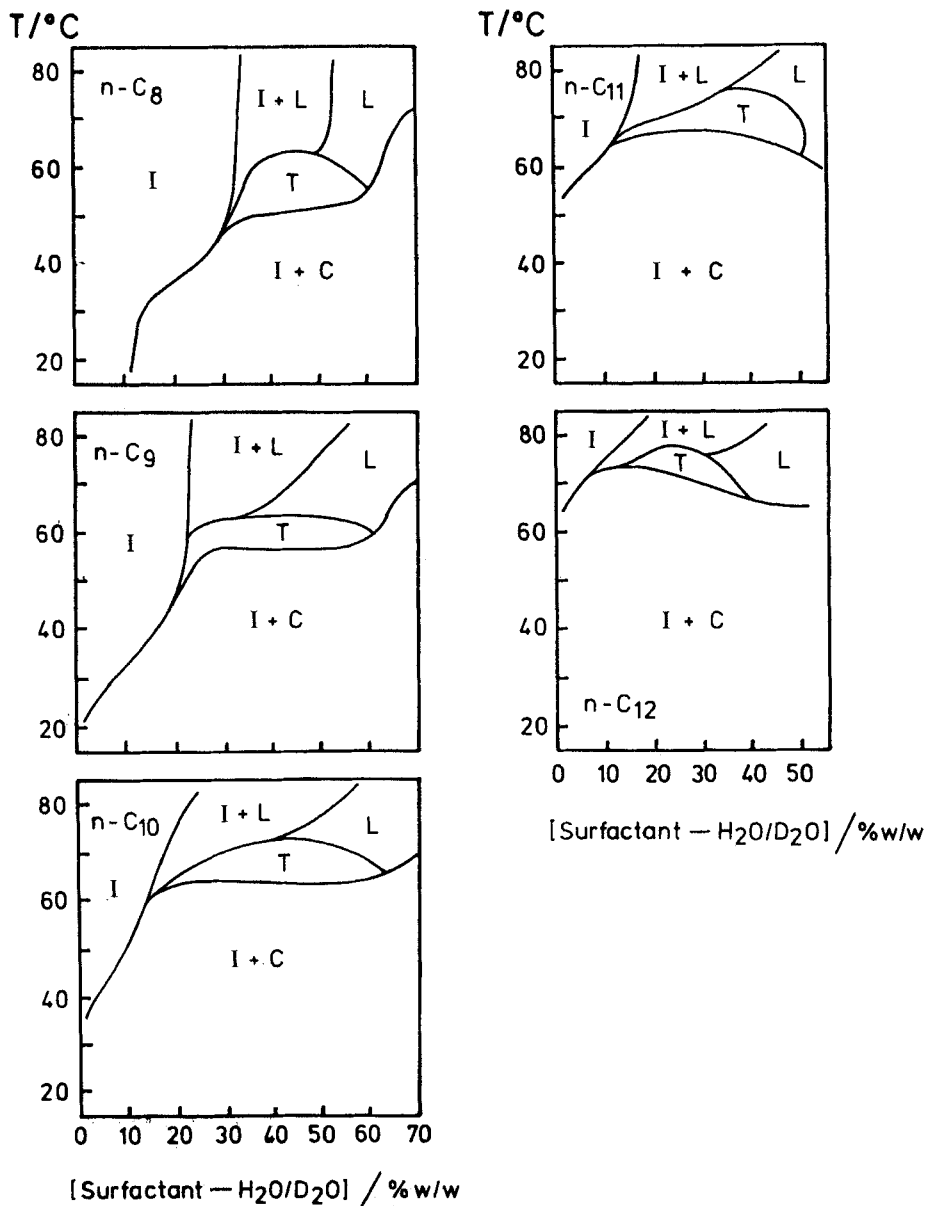


Figure 1. Phase diagrams of *n*-alkylbenzenesulphonates in water.

The phase diagram for the HDBS–water system is very simple, exhibiting at lower concentrations of HDBS at all temperatures between 20 and 80°C isotropic behaviour (see figure 4). The 35 per cent sample exhibits a nematic/lamellar transition phase at low temperature (20–30°C) and the nematic phase in the higher temperature region. The region of the lamellar phase spreads from 40–80 per cent HDBS–for water mixtures, and the inverse cubic phase from 85–98 per cent of HDBS–water mixtures. The lamellar phase was found also by Krishnamurti and Somashekar [12]. The last phase, an inverse cubic, was recognized by its optical isotropy, and because of the very low water

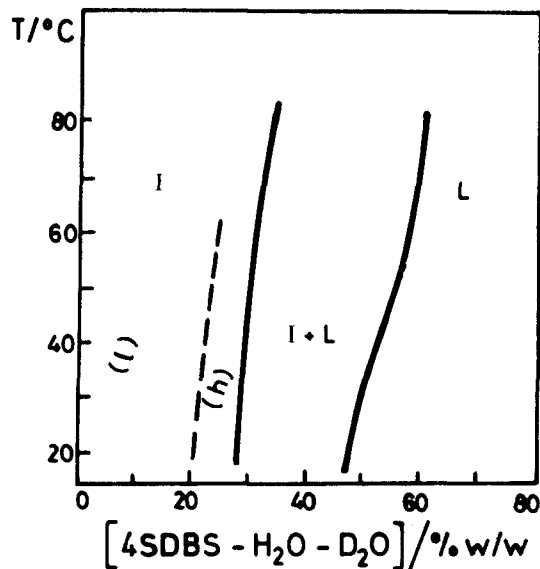


Figure 2. Phase diagram of 4SDBS-water: I = isotropic solution (*l* = low viscosity, *h* = high viscosity), N = nematic, L = lamellar phase.

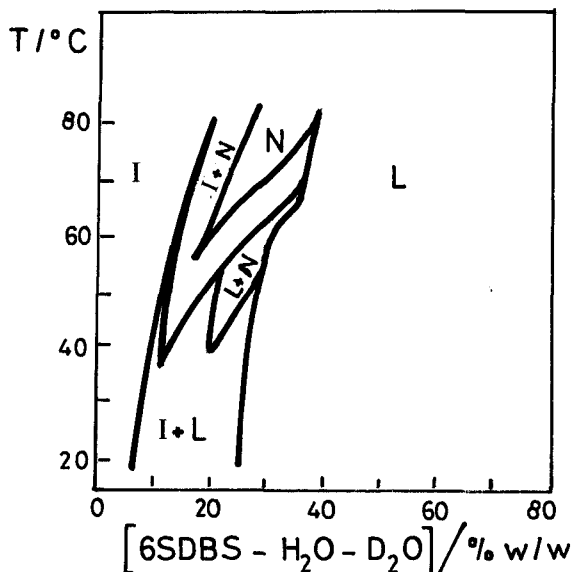


Figure 3. Phase diagram of 6SDBS-water.

content as well as its very high viscosity. The contact preparation in figure 5(a) exhibits the textures of the complete phase diagram at 20°C achieved by water penetration into a pure HDDBS sample. The phases from right to left are as follows: isotropic, spherulites and batonnets on the borderline, nematic (pseudoisotropic), lamellar, inverse cubic (pseudoisotropic). The further characterization of the nematic phase by magnetic field alignment will be discussed later.

The lamellar phases (characteristic  $^2\text{H}$  NMR spectrum in figure 6) can also be recognized by the strong dependence of the  $\Delta\nu(^2\text{H})$  NMR splittings on concentration

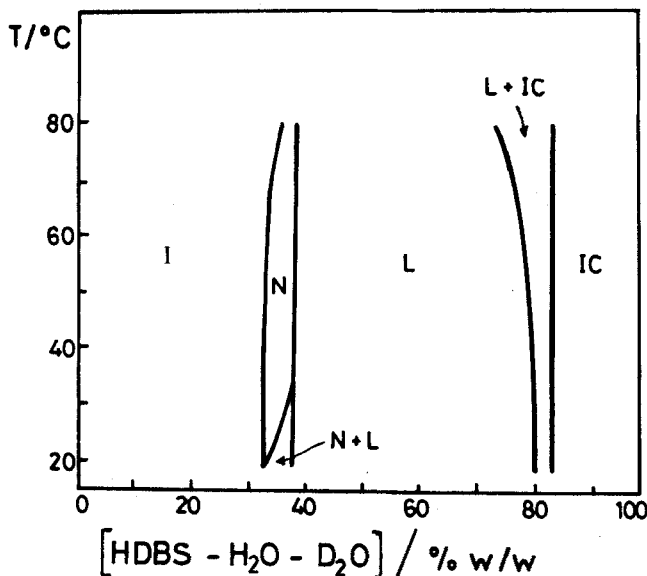


Figure 4. Phase diagram of HDBS-water: I=isotropic solution, N=nematic, L=lamellar, IC=inverse cubic phase.

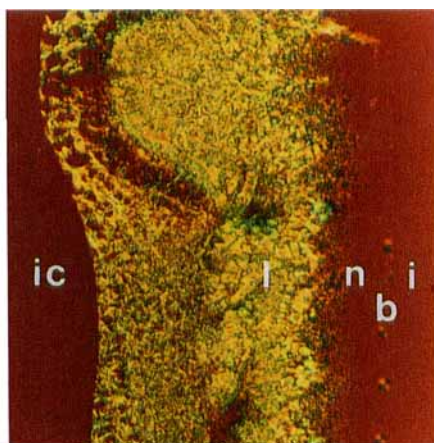
and temperature. For the L–N transition, a sudden broadening of  $\Delta\nu(^2\text{H})\text{NMR}$  occurred (see figure 7 for 6HDBS sample) giving the possibility to determine the transition temperature. Boden *et al.* have also shown that the first order lamellar to nematic transition can be observed by  $^2\text{H}$  NMR spectroscopy [13]. Hakala and Wong showed [14] that the magnitude of  $\Delta\nu(^2\text{H})$  and  $\Delta\nu(^{17}\text{O})$  quadrupolar splitting with changing temperature may reflect the continuous change of the orientation of water molecules with respect to the mesophase director.

From the appearance of two doublets a larger quadrupolar splitting can be predicted for the lamellar phase than for the hexagonal phase [15] as well as for the nematic phase than for the hexagonal phase [16]. The inverse cubic phase exhibits a broader peak than an isotropic phase and not a powder pattern as for lamellar phases (see figure 6) [15, 17].

### 3.3. Microscopic observations

The crystalline and liquid crystal textures of *n*-surfactants are presented in figures 8 (a) and (b). Only lamellar (focal conic) phases and birefringent crystalline phases were observed in the two phase equilibrium state with water. Strong optical birefringency of crystalline surfactants, as well as high X-ray diffraction maxima [18] show that the surfactants in a crystalline state exhibit a multilayer stack. A three phase equilibrium state is seen for a 25 per cent binary mixture of *n*-C<sub>10</sub> and water at 33.7°C (see figure 8 (b)). The sample is also characteristic of the transition state for the other *n*-surfactants. It was crystalline at room temperature and was transformed to the lamellar phase by heating, exhibiting a temperature hysteresis on cooling. The phase equilibrium state shows a characteristic fan shaped texture with two crystals growing very fast within and an isotropic region dividing the liquid crystal and the solid crystal region.

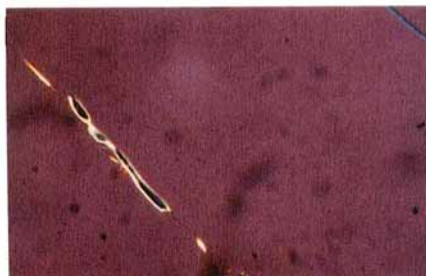
The characteristic textures of HDBS–water mixtures are shown in figure 5 (some of them were presented by Rosevear [19, 20]). The nematic, lamellar and inverse cubic



(a)



(b)

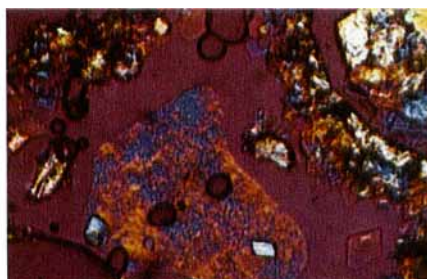


(c)

Figure 5. (a) The contact preparation of HDBS-water system at room temperature. The exhibited phases: isotropic (I), batônnet (B), nematic (N), lamellar (L) and inverse cubic (IC);  $\lambda$ -plate, total magnification  $\times 40$ . (b) 35 per cent HDBS-water mixture at  $33.3^\circ\text{C}$ . The isotropic/pseudoisotropic texture characterizes the isotropic+nematic phase. (c) Batônnet appearing at lamellar/inverse cubic phase transition limit. The shape of such a batônnet is changing fast dividing very viscous phases (HDBS, 80 per cent,  $49.1^\circ\text{C}$ ). Crossed polarizers,  $\lambda$ -plate. Total magnification  $\times 160$ .



(a)



(b)

Figure 8. The texture of (a) anisotropic solid crystals of  $n\text{-C}_9$  in water (25 per cent,  $5.5^\circ\text{C}$ ). The hexagonal crystal plates are characteristic of all  $n$ -alkylbenzenesulphonates, appearing with smaller dimensions on increasing the  $n$ -alkyl chain length. The crystal plates change in colour on varying the angle between the light and director due to the Bragg reflections. Crossed polarizers,  $\lambda$ -plate, (b) three phase equilibrium state of  $n\text{-C}_{10}$  in water (25 per cent  $33.7^\circ\text{C}$ , decreasing temperature): liquid-crystalline phase with two octahedral crystals growing within, isotropic and anisotropic solid crystals and isotropic phase. Crossed polarizers,  $\lambda$ -plate. For all photomicrographs total magnification  $\times 160$ .



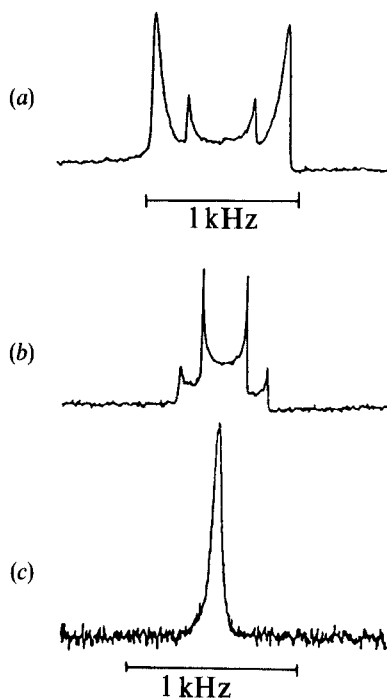


Figure 6. (a)  $^2\text{H}$  NMR spectrum of 6SDBS–water mixture: 20 per cent mixture at  $45^\circ\text{C}$  of well expressed two phases in the bottom part of test tube ( $\Delta^2H_i=987$  Hz belonging to the nematic and  $\Delta^2H_i=485$  Hz belonging to the lamellar phase); (b)  $^2\text{H}$  NMR spectrum of HDBS–water mixture: two lamellar doublets of 40 per cent mixture at  $60^\circ\text{C}$ ; (c) inverse cubic phase wide peak of 90 per cent mixture of the same system as (b) at  $60^\circ\text{C}$ ; such a spectrum is observed for all temperatures between 20 and  $90^\circ\text{C}$ .

phases were observed in HDBS samples. In the transition phase regions (nematic/lamellar and lamellar/inverse cubic) the appearance of batônnetts is always possible [21]. Batônnetts were formed in the nematic/lamellar region at  $20^\circ\text{C}$ . A nematic texture was observed on increasing the temperature from 20 to  $33^\circ\text{C}$ . Figure 5 (b) shows such an isotropic and pseudoisotropic system. The texture with a batônnet in figure 5 (c) shows the phase sequence lamellar/inverse cubic at  $49.1^\circ\text{C}$ .

The textures of nematic and lamellar phases of 15, 20 and 25 per cent 6SDBS–water mixtures are presented in figures 9 (b) and (c) and 10 (a), (b) and (c). The lamellar phase is always exhibited at  $25^\circ\text{C}$ . The 20 per cent shows the formation of lamellar and nematic phases at  $45^\circ\text{C}$ . The texture with small bubbles shows the lamellar–nematic transition i.e., lamellar phases in the concentration region between 15 and 25 per cent are transformed to nematic phases on heating (see figure 9 (b)). Further heating of the samples causes the appearance of batônnetts and nematic droplets at the isotropic/pseudotropic limit (see figure 9 (c)). The pseudoisotropic system shows the optical birefringency on tilting the samples between crossed polarizers in the microscopic field (see figures 10). Under these conditions darker and lighter illumination can be observed, even with the appearance of batônnetts appearing and disappearing within a short time interval. The circular bubbles in figures 10 (a) and (b) show the isotropic and pseudoisotropic, i.e., the isotropic and nematic textures characteristic for the wall-aligned lyotropic nematic phase with the director perpendicular to the light vector. A nematic schlieren texture has never been found in these lyotropic systems.

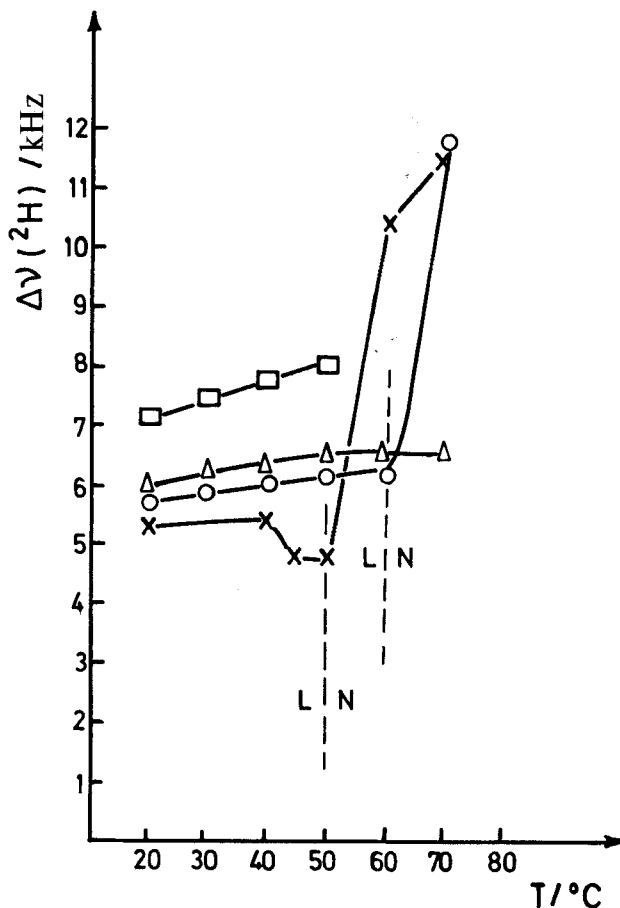


Figure 7. The variation with temperature of the deuterium quadrupole splitting of  $D_2O$  in four samples of 6SDBS.  $\times$ , 20 per cent;  $\circ$ , 25 per cent;  $\Delta$ , 30 per cent;  $\square$ , 40 per cent.

#### 3.4. Characterization of nematic phase by time-resolved $^2H$ NMR

The 6SDBS–water and HDBS–water binary systems form nematic phases in a narrow concentration region between isotropic and lamellar regions. Figure 11 shows the  $^2H$  NMR spectra of the 25 per cent 6SDBS nematic phase at  $70^\circ C$ . The two doublets (see figure 11(a)) show the parallel (higher splitting  $1.0 \delta_{max}$ ) and the perpendicular  $-1/2 \delta_{max}$  orientation of the director to the magnetic field. The spectra were recorded at 0, 5 and 10 min after the samples had been placed in the magnetic field. The first spectrum (see figure 11(a)) is a powder pattern characteristic for a non-aligned nematic phase. An increase in intensity of the shoulders of this powder pattern with time is typical for parallel alignment to the magnetic field (see figure 11(b)); complete parallel alignment is shown in figure 11(c). This parallel alignment can also be seen with a sample tube spinning, and the quadrupole splitting here is a function of the angle made by the director with the magnetic field. If the splitting is larger than the spinning speed, many rotation peaks can be observed as in figure 11(a). The distance of these rotation peaks is a function of the spinning speed. This nematic phase is in the vicinity of the lamellar phase (see phase diagram). From pseudotropic alignment under the

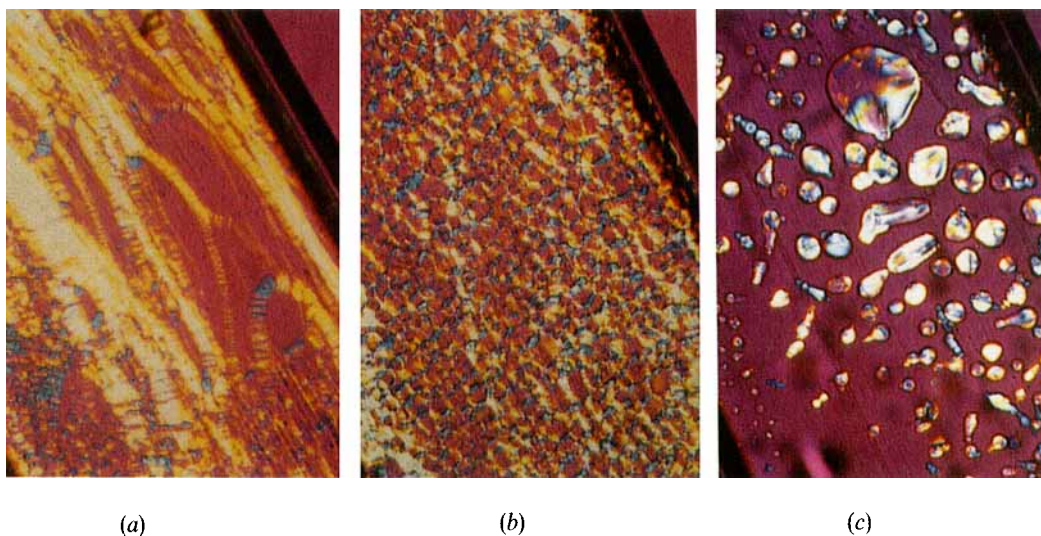


Figure 9. (a) Isotropic and lamellar texture of the 15 per cent 6SDBS mixture at 25°C. (b) The same system at 54.8°C, biphasic: transition lamellar/nematic. Texture exhibits a transition of spherulitic chains to nematic bubbles. (c) Isotropic and nematic texture of the same system at 80.4°C with batônnets and large nematic droplets. Crossed polarizers,  $\lambda$ -plate, total magnification  $\times 160$ .

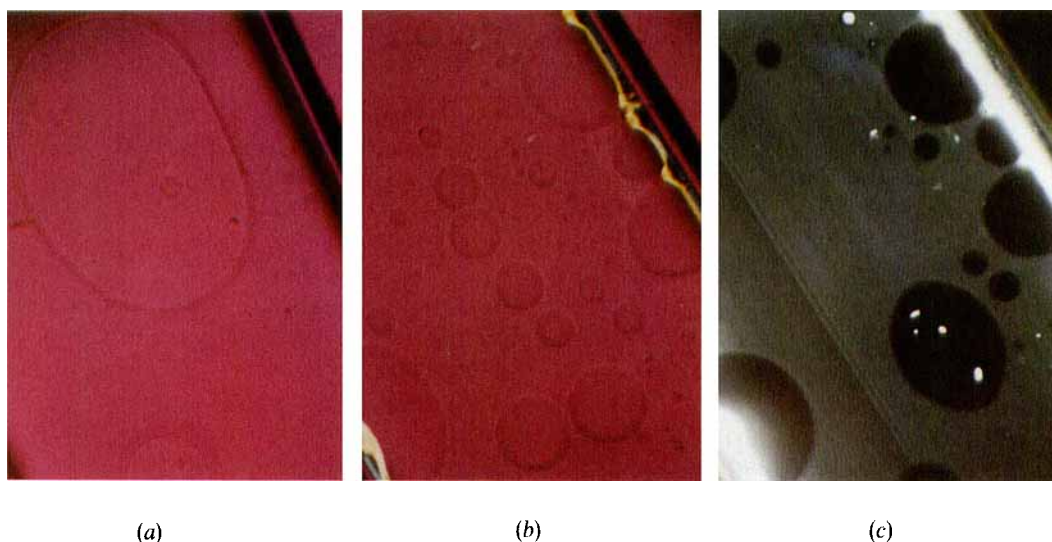


Figure 10. Nematic textures: (a) 20 per cent 6SDBS at 74.5°C, isotropic and nematic with a large circular bubble; crossed polarizers,  $\lambda$ -plate. (b) 25 per cent 6SDBS at 89°C, isotropic and nematic with large bubbles. Crossed polarizers,  $\lambda$ -plate. (c) The same sample without  $\lambda$ -plate showing different illumination by tilting the sample between the polarizers. Total magnification  $\times 160$ .

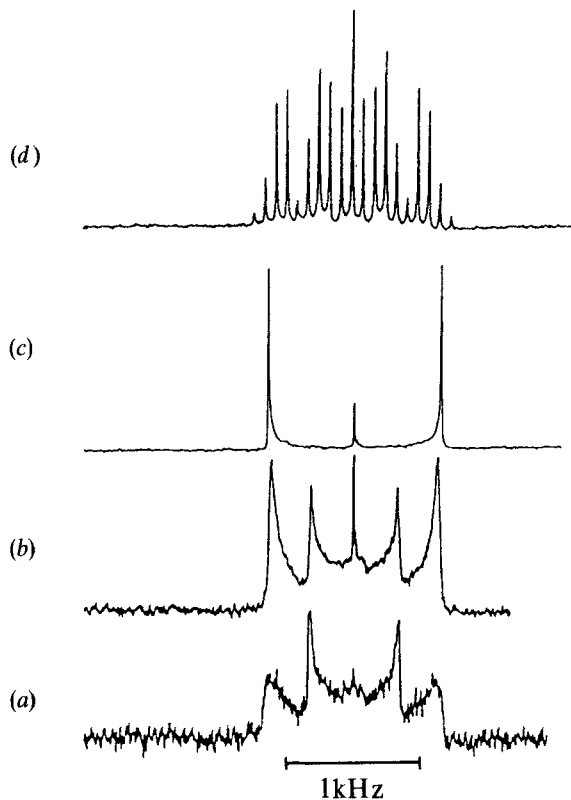


Figure 11.  $^2\text{H}$  NMR spectra of 25 per cent 6SDBS at  $70^\circ\text{C}$ : (a) two nematic doublets recorded immediately after bringing the sample into the magnetic field; (b) the doublets after 5 min, and a central peak which is not an isotropic one; (c) a nematic doublet (only parallel orientation) with the central peak after 10 min; (d) rotation peaks caused by spinning in the magnetic field.

crossed polarizers, it can be concluded that the texture is typical of the disc-like aggregates.

The very fast reorientation of the director parallel to the magnetic field, is characterized by the disappearance of the doublet peak in the  $-1/2$  position. At  $80^\circ\text{C}$  the director orients even faster in the magnetic field (see figure 12). The distance between the two recordings is 60 s; the time of recording started from the upper part of figure 12 to the lower. Initially only the parallel orientation can be seen; the second spectrum at 60 s is recorded after turning the sample by  $90^\circ$  in the magnetic field. After 60 s realignment is so fast that the peaks corresponding to perpendicular orientation are missing. The next three spectra show the growth of the doublet associated with the director parallel to the field.

In the time just before the 360 s recording, the sample has been turned again by  $90^\circ$ . In the spectrum at 360 s two doublets can be seen, which were recorded following realignment.

### 3.5. Emphasis of nematic phases of double-tailed surfactant

The nematic phases formed by small double-chain alkylbenzenesulphonate surfactants have been found to be highly ordered. Very fast magnetic alignment of the

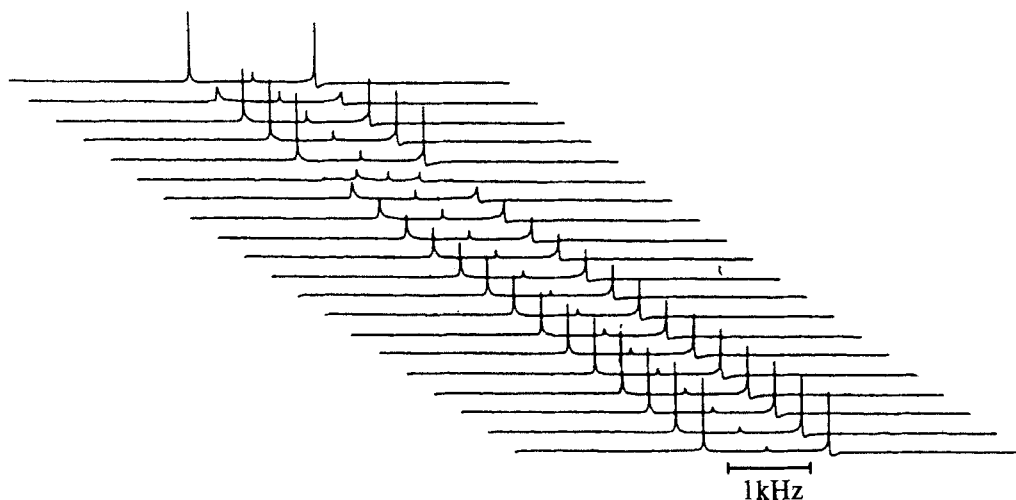


Figure 12. Time-resolved  $^2\text{H}$  NMR spectra of 25 per cent 6SDBS at  $80^\circ\text{C}$ .

nematic phases is due to the size and shape of the micelles as well as the very low viscosity of the micellar solution. The sign of the anisotropy of the diamagnetic susceptibility is positive as in comparable systems [22].

#### 4. Conclusion

The characterization of micellar, solid and liquid-crystalline phases of single and double chain small surfactant molecules in binary mixtures with water has shown:

- (i) the agreement of micellar parameters is in accord with literature data; critical parameters concerning the lengths of surfactant molecules were also obtained;
- (ii) the lamellar phases and anisotropic solid crystal phases show the high ordering in molecular packing, obviously due to the aromatic system in the surfactants;
- (iii) the nematic phases that have been found in a narrow concentration region in binary systems of a small double chain surfactant molecule and water, can be oriented very rapidly in a magnetic field; the nematic phases show characteristic textures under crossed polarizers. In this concentration region disc-like aggregates have been formed. Such aggregates exhibit very fast orientation in the magnetic field, as well as wall alignment between glass surfaces (such an orientation results in the isotropic and pseudotropic texture between crossed polarizers).

Batônnetts appear only in the transition phase regions (as is usual). They are assumed to be the liquid monocrystals [23]; this is confirmed by the fact that they appeared only in the transition regions of any phases; we can talk about a kind of supersaturation condition. Under these conditions the batônnetts are forming and disappearing rapidly. They can change in shape very rapidly, characterizing in such a way the material transition region between phases.

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