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Deformation of cylinders in the hexagonal phase of sodium decyl sulphate/decanol/water on the introduction of decanol

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X-ray scattering studies show that aggregates of amphiphilic molecules in the hexagonal phase of the binary system sodium decyl sulphate/water are infinite cylinders whose radius is very close to that of the extended molecule. When sodium decyl sulphate molecules are progressively substituted by decanol molecules the phase remains hexagonal, up to a decanol/soap molar ratio of 0·15, but the radius of its cylinders becomes larger than the molecular length. We have developed specific experiments to investigate this growth and see if it is isotropic or anisotropic. We have focused our attention on the evolution of the configuration of soap and decanol molecules within the aggregates. Neutron scattering experiments show that the two molecules do not distribute themselves uniformly and D.M.R. measurements show that they stay anchored at the amphiphile/water interface by their polar heads. The distribution of the two molecules along the interface is not therefore uniform. This induces an inhomogeneity of the interfacial curvature and the growth of the section of the cylinders cannot be isotropic. This anisotropy increases when the decanol/soap molar ratio increases. It provides a clue to understand the transformation of the hexagonal phase into a recangular one whose aggregates are infinite ribbons with non-circular section, above the decanol/soap molar ratio of 0·15.

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1. Introduction

Hexagonal phases of amphiphilic molecules in the presence of water are currently encountered in binary and ternary phase diagrams of lyotropic liquid crystals [1–3]. Their structure has been determined by X-ray diffraction studies for a long time [1, 2]. The diffraction patterns show a set of narrow Bragg reflections whose positions are in the ratio 1, $\sqrt{3}$, $\sqrt{4}$, ... , which corresponds to the packing of infinite cylinders on a two-dimensional hexagonal lattice. These cylinders are generally described as having circular sections. Two arguments are in favour of this description, when the cylinders are aggregates of amphiphilic molecules. One is that the area of the section determined from X-ray scattering experiments is that of a circle having a radius equal to the length of an extended amphiphilic molecule. The second is that sections with symmetries higher than six are compatible with the *p6m* symmetry of the lattice. We present and discuss here the case of a hexagonal phase where the area of the section of the cylinders becomes larger than that of a circle with a radius equal to the extended molecular length.

This case has been observed when sodium decyl sulphate molecules have been progressively substituted by decanol molecules in the sodium decyl sulphate/water hexagonal phase of the ternary system sodium decyl sulphate/decanol/water, keeping the water content constant. The question which appears now is that of the nature of that growth. Is it isotropic, the cylinders keeping its circular shape, or anisotropic, with a deformation of the cross section? This question is of particular importance in understanding the polymorphism of the sodium decyl sulphate/decanol/water system, as it has been shown that the hexagonal phase transforms into a rectangular phase of *cmm* symmetry, with infinitely long ribbon-like aggregates, when the decanol/soap molar ratio becomes larger than 0·15 [4–6]. Unfortunately this question cannot be answered with X-ray scattering experiments alone. As the *p6m* symmetry of the phase imposes at least a six-fold symmetry to the distribution of any section, we cannot discriminate between a section growing with a six-fold symmetry, a nearly isotropic section, and section growing anisotropically with a symmetry lower than six. We have therefore applied an approach similar to that used to detect a local biaxiality in a uniaxial lyotropic nematic phase [7].

The general idea of this approach is that the knowledge of the internal structure of the aggregate, or the way the two amphiphilic molecules are organized within it, may provide information about its shape. For instance: if the decanol molecules just take the place of soap molecules at the interface, without any other change, there should be no growth; if they take this place but are not uniformly distributed along the interface, there should be a modulation of mean area per chain at the interface, hence modulation of interfacial curvature, and the growth should be anisotropic; if they occupy the cores of cylinders of soap molecules rather than the interface, the growth might be isotropic. We therefore need complementary experiments, particularly one giving access to the relative locations of the two molecules within an aggregate (are they uniformly distributed or not?) and a second giving access to their environment (are they at the interface or in the core?). This is why we have chosen neutron scattering experiments with contrast variation [5, 7, 8] and N.M.R. measurements [9].

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The application of these two methods to the problem which we have introduced shows that the increase of the radius of the aggregates with increase of the decanol/soap molar ratio is associated with an inhomogeneous distribution of soap and decanol molecules, with their polar heads along the interface and their paraffinic chains in similar conformational states. This is a configuration which implies a modulation of the interfacial curvature. We shall conclude from this that the cross section of the cylindrical aggregates is anisotropic. This result will be discussed in relation to the transformation of the hexagonal phase into the neighbouring rectangular phase on the phase diagram [4–6].

2. Experimental

2.1. *Samples*

The compositions of the samples in mole percent, their decanol (C_{10}–OH)/sodium decyl sulphate (SdS) molar ratio are given in table 1. Their textures were observed with a polarizing microscope; they correspond to the classical textures of middle phases made of infinite cylinders organized in a two-dimensional lattice, most often of hexagonal symmetry [10]. Their structures were obtained by X-ray diffraction experiments using a Guinier camera with monochromatic Cu, $K\alpha_1$ radiation of wavelength $\lambda = 1{\cdot}54$ Å. The X-ray powder patterns all show two or three narrow Bragg reflections whose positions are in the ratio $1, \sqrt{3}, \ldots$. The position of the first order reflection is $d\sqrt{3}/2$, where d is the lattice parameter given in table 1. From d we can deduce the diameter, d_a, of the cylinders according to the relation [1]

$$d_a = d\left[\frac{2\sqrt{3}}{\pi}\,\frac{1}{1 + (\bar{v}_e/\bar{v}_a)(1 - c)/c}\right]^{1/2},$$

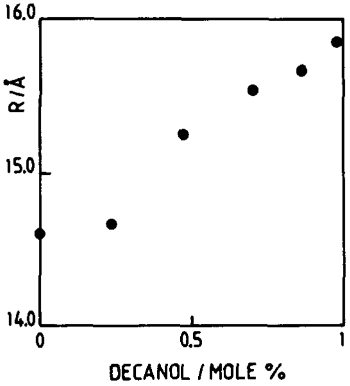

Figure 1. Radius of the aggregates, R, as a function of decanol in mole per cent.

Table 1. Composition of the samples and lattice parameter of the phase.

Sample	Mole per cent			C_{10}-OH/SdS	
	SdS	C_{10}-OH	H_2O	(molar ratio)	d/Å
1	7·09	0	92·91	–	40·04
2	6·95	0·23	92·82	0·03	40·15
3	6·79	0·47	92·74	0·07	41·64
4	6·65	0·70	92·75	0·11	42·32
5	6·55	0·86	92·59	0·13	42·63
6	6·47	0·98	92·55	0·15	43·11

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where c is the weight concentration of amphiphile for 1 g of the mixture, $\bar{v}_e$ and $\bar{v}_a$ are respectively the partial specific volumes of the solvent and amphiphile. The experiments were carried out at 23°C. The radius of the aggregates, R, is plotted versus the decanol concentration, in mole per cent, in figure 1. This radius increases when the C_{10}-OH/SdS molar ratio increases.

2.2. *Neutron scattering experiments: homogeneity of the distribution of amphiphiles*

Coherent scattering of neutrons is much stronger for deuterons than for protons and this can be used to study the relative distribution of two molecules when one is deuteriated while the other is kept protonated [11]. We already took advantage of this to describe the distribution of amphiphilic molecules in disc-like or ribbon-like aggregates with inhomogeneous interfacial amphiphile/water curvatures, of ternary soap/alcohol/water systems. We showed that the inhomogeneous interfacial curvatures are correlated with inhomogeneous distributions of both amphilphilic species within the aggregates [5, 7, 8]. We briefly recall the principle of the method. If the distribution of the species within the aggregates is uniform, the aggregates will have a uniform scattering length density corresponding to the average value of the scattering length densities of the deuteriated and protonated aliphatic chains. If, on the other hand, the distribution of the species is not uniform, the scattering length density of the aggregate will be modulated according to the distribution of the molecules, the amplitude of this modulation being limited to the difference between the scattering length densities of the deuteriated and protonated chains. The form factor of the aggregates will therefore be different in the two cases, i.e. the intensities of the Bragg reflections of the hexagonal lattice should be modulated differently. We looked for an eventual inhomogeneous distribution of the amphiphiles as a function of the C_{10}–OH/SdS molar ratio by comparing the spectra obtained with several pairs of samples prepared and characterized along the lines described in the preceding paragraph. The C_{10}–OH/SdS molar ratio of the samples of each pair is the same but the aggregates of one sample are made of protonated SdS and protonated C_{10}–OH while those of the other are made of protonated SdS and perdeuteriated C_{10}–OH.

The interfacial region of the aggregates, where the polar heads are localized, constitutes an heterogeneity which might be the source of a spurious phenomenon similar to the one just discussed. To remove it, the samples were prepared with a mixture of light and heavy water whose scattering length density, $\varrho_s = 4{\cdot}59\,\mathrm{cm}^{-2} \times 10^{10}$, matches those of the polar head groups of the amphiphilic molecules which are respectively $4{\cdot}33\,\mathrm{cm}^{-2} \times 10^{10}$ for the OSO_3Na group and $4{\cdot}62\,\mathrm{cm}^{-2} \times 10^{10}$ for the hydroxyl group, taking into account the isotropic exchange OH/OD [11]. We controlled, by X-ray scattering studies for each pair of samples with identical molar composition but with either totally or partially protonated aggregates, that the structure is not perturbed by the use of deuteriated molecules. We worked with polycrystalline samples at 23°C.

The scattering length density of the samples with fully protonated aggregate cannot but be uniform. On the other hand the scattering length density of the samples with protonated SdS and perdeuteriated C_{10}–OH can either be uniform, if the distribution of both amphiphilic molecules within the aggregates is homogeneous, or non-uniform, if the distribution is not homogeneous. The form factors of the aggregates are different in these two last situations. In the first situation, when the distribution is homogeneous, the intensity of the Bragg reflections of a sample with perdeuteriated C_{10}–OH and that of the sample with the same C_{10}–OH/SdS ratio with protonated amphiphiles will simply scale the difference between the scattering length density of the protonated aggregates and that of the partially deuteriated ones, i.e. if ϱ_1 is the scattering length density of fully protonated aggregates, $\bar{\varrho}_2$, the average scattering length density of partially deuteriated aggregates, ϱ_s being that of the solvent, the scattered intensity, $I(\mathbf{q})$, will be proportional to $(\varrho_1 - \varrho_s)^2$ and to $(\bar{\varrho}_2 - \varrho_s)^2$ respectively ($\varrho_1, \bar{\varrho}_2, \varrho_s$ can be calculated from the compositions of the samples and the tables of scattering length densities [11]). In the second situation when the distribution is inhomogeneous, the intensity of the Bragg reflection of a sample with perdeuteriated C_{10}–OH and that of the sample with the same C_{10}–OH/SdS molar ratio with protonated amphiphiles will not simply scale the difference between $(\varrho_1 - \varrho_s)^2$ and $(\bar{\varrho}_2 - \varrho_s)^2$.

The experiments were performed in Laboratoire Leon Brillouin (LLB) in CEN-Saclay on the spectrometer PAXY provided with an *XY* multidetector. The sample–detector distance was 194·4 cm, the wavelength $\lambda = 3{\cdot}25$ Å and the wavelength spread, $\Delta\lambda/\lambda = 5$ per cent. The data were treated according to standard LLB procedures for small angle isotropic scattering. They were normalized for unit incident beam flux. The spectra were corrected for background, absorption and detector efficiency. Only the first Bragg reflections can be analysed as the signal-to-noise ratio of the second is too low.

2.3. *D.M.R. experiments: conformations of amphiphiles*

In addition to neutron scattering experiments, D.M.R. measurements were performed. The magnitudes of the quadrupolar splittings of deuteriated methylene

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and methyl groups along the hydrocarbon chain of an amphiphilic molecule depend on the order parameters of the chain links, or the conformational state of the chain, and therefore reflect the situation of the molecule relative to the interface and its environment [9]. Here we have recorded the order parameter curves for each amphiphilic species by measuring the deuterium quadrupolar splittings of SdS perdeuteriated chains and those of C_{10}–OH perdeuteriated chains in samples with the same molar composition but with protonated C_{10}–OH in the first case and protonated SdS in the second. The measurements were made with a Bruker spectrometer CXP 100 equipped with a Drusch electromagnet of 2·1 T.

3. Results and discussion

We recall that we worked with protonated SdS and C_{10}–OH or protonated SdS and perdeuteriated C_{10}–OH in a solvent whose scattering length density matches that of the polar head groups so that the scattered intensity depends only on the contrast of the paraffinic chains with respect to the solvent.

If the soap and the decanol are homogeneously distributed within the aggregates the form factor of fully protonated aggregates is the same as that of partially deuteriated ones with the same molar composition. The scattering length density of fully protonated aggregates, ϱ_1, is equal to $-0{\cdot}41\,\mathrm{cm}^{-2} \times 10^{10}$ so that if ϱ_s is the scattering length density of the solvent, $\Delta\varrho_1 = \varrho_1 - \varrho_s = -5{\cdot}0\,\mathrm{cm}^{-2} \times 10^{10}$ and the scattered intensity $I_1(\mathbf{q}) \propto \Delta\varrho_1^2$. The average scattering length density of the partially deuteriated aggregates, $\bar{\varrho}_2$, function of the percentages of the protonated and perdeuteriated chains can be calculated, $\Delta\bar{\varrho}_2 = \bar{\varrho}_2 - \varrho_s$ and $I_2(\mathbf{q}) \propto \Delta\bar{\varrho}_2^2$ so that the scattered intensities for corresponding Bragg reflections will be in the ratio $I_1(\mathbf{q})/I_2(\mathbf{q}) = \Delta\varrho_1^2/\Delta\bar{\varrho}_2^2$, $\Delta\varrho_1^2 = 25\,\mathrm{cm}^{-4} \times 10^{20}$. $\bar{\varrho}_2$, $\bar{\varrho}_2 - \varrho_s$, $\Delta\bar{\varrho}_2^2$ and the calculated $I_1(\mathbf{q})/I_2(\mathbf{q})$ for the samples investigated are given in table 2.

Table 2. Average scattering length density of partially deuteriated aggregates, $\bar{\varrho}_2$; $\bar{\varrho}_2 - \varrho_s$ and $\Delta\bar{\varrho}_2^2$. Calculated and experimental intensities ratio, $I_1(\mathbf{q})/I_2(\mathbf{q})$†.

Sample	$\bar{\varrho}_2$ $\mathrm{cm}^{-2} \times 10^{10}$	$(\bar{\varrho}_2 - \varrho_s)$ $\mathrm{cm}^{-2} \times 10^{10}$	$\Delta\bar{\varrho}_2^2$ $\mathrm{cm}^{-4} \times 10^{20}$	$I_1(\mathbf{q})/I_2(\mathbf{q})$ (Calculated)	$I_1(\mathbf{q})/I_2(\mathbf{q})$ (Experimental)
3	0·07	−4·52	20·43	1·22	1·42
4	0·28	−4·31	18·58	1·35	1·62
5	0·45	−4·14	17·14	1·46	1·78
6	0·56	−4·03	16·24	1·54	2·05

† $I_1(\mathbf{q})$, scattered intensity for fully protonated aggregates; $I_2(\mathbf{q})$, scattered intensity for partially deuteriated aggregates.

If, on the other hand, the distribution of both amphiphilic molecules within the aggregates is not homogeneous, the scattered intensity for the fully protonated aggregates will still be proportional to $\Delta\varrho_1^2$ but the form factor of the aggregates with perdeuteriated C_{10}–OH will change, $I_2(\mathbf{q})$ is no longer proportional to $\Delta\bar{\varrho}_2^2$ and the scattered intensities for corresponding Bragg reflections will no longer follow the $I_1(\mathbf{q})/I_2(\mathbf{q})$ calculated ratios.

The spectra for the pairs of sample 3, 4, 5 and 6 were recorded. The spectra obtained with fully protonated aggregates and partially protonated aggregates whose C_{10}–OH/SdS molar ratio is 0·11 (sample 4) and those obtained with aggregates whose C_{10}–OH/SdS molar ratio is 0·15 (sample 6), are shown in figures 2(*a*) and (*b*)

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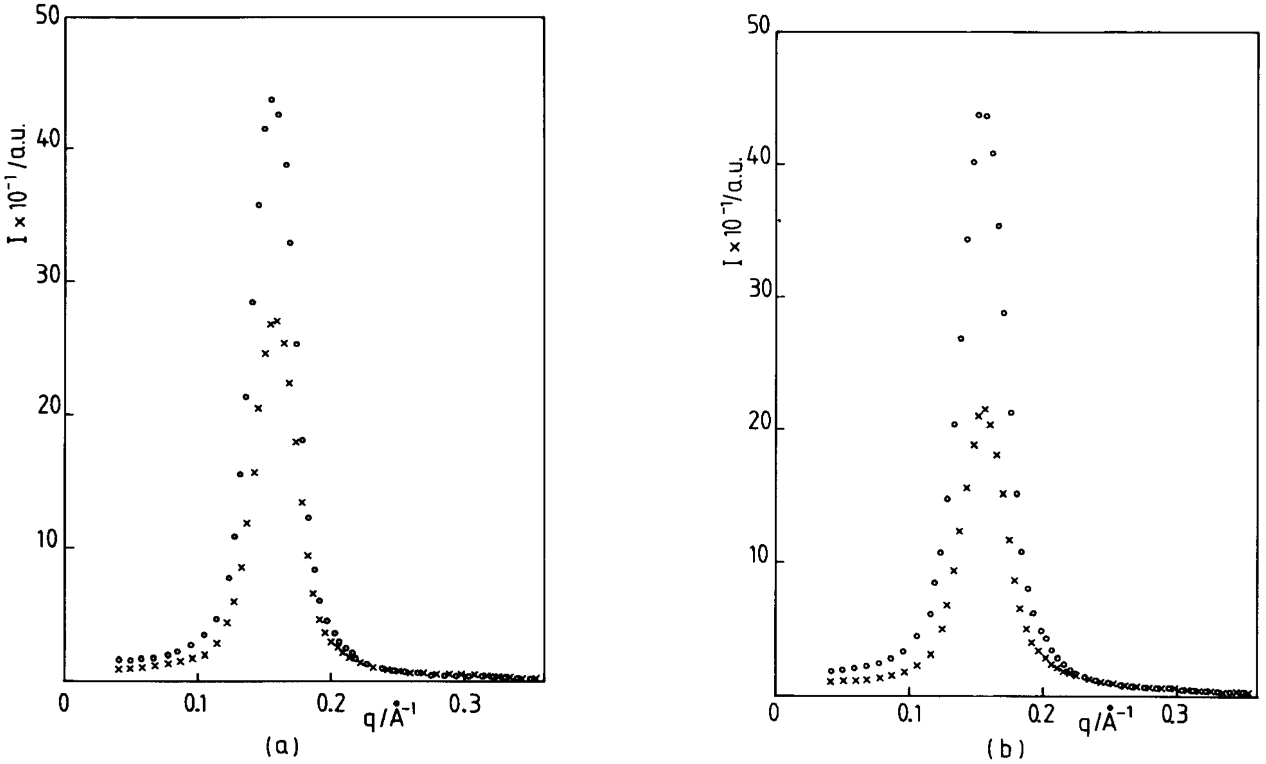

Figure 2. Neutron scattering curves. (*a*) Intensity curves, $I(\mathbf{q})$, recorded with sample 4; C_{10}–OH/SdS molar ratio = 0·11; ○, aggregates with protonated SdS and protonated C_{10}–OH; ×, aggregates with protonated SdS and perdeuteriated C_{10}–OH. (*b*) Intensity curves, $I(\mathbf{q})$ recorded with sample 6; C_{10}–OH/SdS molar ratio = 0·15; ○, aggregates with protonated SdS and protonated C_{10}–OH; ×, aggregates with protonated SdS and perdeuteriated C_{10}–OH. The values $I(\mathbf{q})$ are in arbitrary units.

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respectively. If the protonated and perdeuteriated chains were uniformly distributed within the aggregates we should expect the intensities of the first Bragg reflection of the two samples of each pair should be according to their $\Delta\varrho_1^2$ and $\Delta\bar{\varrho}_2^2$ values, in the ratio 1·35 and 1·54, respectively reported as $I_1(\mathbf{q})/I_2(\mathbf{q})$ Calculated) in table 2. Experimentally the intensity ratios of the first Bragg reflection differs from those expected; they are equal to 1·62 (sample 4) and 2·05 (sample 6) respectively, reported as $I_1(\mathbf{q})/I_2(\mathbf{q})$ (Experimental) in table 2. The form factor of the partially deuteriated aggregates is therefore different from that of the fully protonated ones and we may conclude that the protonated and perdeuteriated chains are not uniformly distributed within the aggregates. The same phenomenon is verified for the pairs of sample 3 and 5. The deviation from the calculated intensity ratios is more pronounced, i.e. the form factor is more affected as the decanol concentration increases.

This inhomogeneous distribution of the soap and decanol molecules within the aggregate might be viewed in two ways. It might result either from a solubilization of the decanol molecules within the core of a cylinder of SdS molecules, as is the case for alkanes [12, 13], or from an inhomogeneous distribution of the two amphiphilic species along the interface. As they correspond to different environments of the molecules, they may be discriminated by a D.M.R. experiment.

We may infer from D.M.R. studies of sample 4 (C_{10}–OH/SdS = 0·11), with either perdeuteriated SdS molecules and protonated C_{10}–OH molecules or protonated SdS molecules and perdeuteriated C_{10}–OH molecules, that the conformational state of the paraffinic chains of both amphiphilic molecules is the same. The measured quadrupolar splittings, $\Delta\nu$, of the CD_2 and CD_3 groups as a function of the carbon number of the methylenes along the chains are shown in figure 3. The quadrupolar splittings for the paraffinic chains of both amphiphiles are very close. If the decanol molecules behaved as alkane molecules, i.e. if they were solubilized within the core of the cylinder, the quadrupolar splittings should be about a factor ten lower [13]. The fact that the paraffinic chains of the decanol molecules are in a conformational state similar to that

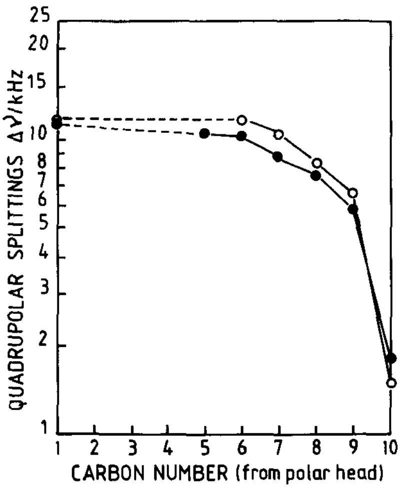

Figure 3. D.M.R. measurements. Sample 4, C_{10}–OH/SdS molar ratio = 0·11. Quadrupolar splittings, $\Delta\nu$, of the CD_2 and CD_3 groups as a function of the carbon number of the methylene groups along the chains. ●, perdeuteriated SdS (protonated C_{10}–OH); ○, perdeuteriated C_{10}–OH (protonated SdS) at T = 25°C.

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of soap, reveals a similar organization of the two molecules relative to the normal to the interface.

At this stage we might argue that the decanol molecules might be similarly disordered relative to the normal to the interface but with their hydroxyl group either on the interface or in the centre of the aggregate. The last situation is highly improbable. Indeed it would be at the origin of another structural heterogeneity, in the core of the aggregates, which we could not detect in the case of contrast variation studies performed to determine the internal structure of micelles in the potassium laurate/decanol/water system, quite similar to the one studied here [7]. We may dismiss this situation and consider only that where decanol molecules are organized relative to the interface as soap molecules, with their hydroxyl group at the interface.

From our set of experiments it follows that when decanol molecules are substituted by soap molecules they are arranged similarly relative to the interface (D.M.R.), but not uniformly distributed along it (neutron scattering). The relative concentrations of hydroxyl and sulphate groups are then modulated along the interface, hence the mean area per paraffinic chain of amphiphile at the interface. As it has been suggested [14] and shown experimentally [5, 6], such a modulation is associated with a modulation of the interfacial curvature: a relative higher concentration of soap molecules being associated with a higher curvature. The cross section of the cylinder has therefore an inhomogeneous curvature and cannot be circular. Whatever this deformation, the orientational distribution of the sections on the hexagonal lattice must preserve its *p6m* symmetry.

4. Conclusion

We have demonstrated the existence of a modulation of the relative concentrations of soap and decanol molecules along the interfaces of cylindrical aggregates of the hexagonal phase of the SdS/C_{10}–OH/water system. From this observation we have deduced information about the evolution of the shape of these cylinders with the C_{10}–OH/SdS molar ratio. The section of the cylinder is, on average, circular at low C_{10}–OH/SdS molar ratios, and becomes non-circular as this ratio increases. This must be discussed in the general context of the SdS/C_{10}–OH/water phase diagram [4, 5] and other ternary phase diagrams [3]. Their examination shows that the introduction of a long chain alcohol into a soap/water system transforms its hexagonal, or micellar phases, into a lamellar phase. In more general terms, the introduction of decanol changes the interfacial curvature. Our observation shows that this change starts at low decanol content, in the hexagonal phase itself. It proceeds inhomogeneously as the section of the cylinder presents regions of different curvatures. This is an interesting example where the change of shape of the aggregates is not immediately related to the change of symmetry of the structure [15]. This deformation increases with the decanol content. Most likely, when it is pronounced enough, it becomes a driving force for the transformation of the hexagonal phase into a rectangular phase with *cmm* symmetry when the C_{10}–OH/SdS molar ratio becomes higher than 0·15 [4–6]. The non-circular sections of the cylinders, which are orientationally disordered with at least a six-fold symmetry on the hexagonal lattice, become organized with a lower symmetry on a rectangular lattice.

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