Fluorinated lamellar phases: structural characterisation and use as templates for highly ordered silica materials

Supplementary Information

1: Effect of coexistence on scattering spectra and fitted quantities obtained:

![Graph showing scattering spectra and fitted quantities](Figure S1)

**Figure S1**: Plot showing the relative scattering intensities of a micellar sample (H29, 10 wt% NaPFO in water, red symbols) and a pure lamellar sample (H37, 10 wt% NaPFO + 10 wt% PFOH in water, blue symbols). These are compared with the scattering spectrum of H29+H37 (green symbols). This simple demonstration does not normalise for volume fractions of the two contributions.

In analysing the SANS spectra of samples in the lamellar+micellar (that is, Lα + L1) coexistence region of the phase diagram, the data were fitted using only the lamellar model. This approximation was based on the assumption that the scattering from the micelles is far less intense than that from the lamellae, and therefore would not affect the overall scattering spectrum obtained for the bulk sample greatly.

However, to demonstrate and test this assumption, the scattering from a purely lamellar sample and purely micellar sample were compared (shown in Figure S1) and added. As can be seen, the lamellar scattering is 10-100 times greater than that
from a micellar sample, due to the low contrast between the NaPFO and D$_2$O when compared to the higher contrast when the alcohol, PFOH is added to make a lamellar phase.

The effect of a coexistence sample was probed by adding the two spectra together (shown as the green data points in Figure S1). This is indistinguishable from the scattering of the pure lamellar phase until higher $q$-values (around 0.09 Å$^{-1}$). Because we did not normalise the contribution of the micellar and lamellar scattering by volume fraction, in fact this deviation is over-estimated, and thus represents the most extreme possible case.

When fitting the added lamellar+micellar spectrum, the values for the inter-lamellar spacing and Caillé parameter remained unchanged from those of the pure lamellar phase (see Table S1), with the bilayer width $\delta$ changing by $\approx 10\%$ due to the slightly higher intensity at higher $q$. From this we conclude that the values for inter-lamellar spacing and Caillé for the samples are likely to be precise for the lamellar contribution in these mixed phases, despite the coexistence. However, the values for delta have an uncertainty of up to 10% for these samples, and thus should be interpreted with some caution.

**Table S1:** Fitted values for pure a lamellar phase, and estimated deviation in their values due to the micellar contribution of a kinetically trapped, mixed phase

<table>
<thead>
<tr>
<th>Sample</th>
<th>Bilayer spacing, $d$ (nm)</th>
<th>Caillé parameter, $\eta_{cp}$</th>
<th>Bilayer thickness, $\delta$ (nm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>H37</td>
<td>17.0</td>
<td>0.14</td>
<td>2.1</td>
</tr>
<tr>
<td>H38+H29</td>
<td>17.0</td>
<td>0.14</td>
<td>2.3</td>
</tr>
</tbody>
</table>