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Structural characterization of alkyltrimethylphosphonium surfactants by powder real-time and monocrystal XRD analysis

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Surfactants based on tetraalkylphosphonium salts have recently become the focus of a good number of investigations because they offer superior properties when compared with tetraalkylammonium surfactants [1], such as much higher thermal stability. Since the structure of phosphonium surfactants still remains hardly explored [2], we have undertaken the structural study of a series of alkyltrimethylphosphonium bromides (nATMP·Br, n is the number of carbon atoms in the alkyl chain taking values from 12 to 22). This study is based on the information provided by DSC, XRD and optical polarizing microscopy. WAXS and SAXS patterns were simultaneous recorded from powder samples using synchrotron radiation (ALBA, NCD BL11 line). Monocrystals for selected cases (n = 12, 18) were also analyzed by XRD using conventional cameras.

The DSC traces registered over the 20-280 ºC range revealed the presence of two (for $n \leq 14$) or three (for $n \geq 16$) endothermal peaks arising from reversible thermal transitions involving crystalline, liquid-crystal or liquid phases. These phases were characterized by real-time XRD recorded at variable temperature (Fig.1). SAXS patterns displayed the presence of a long spacing peak in the ~1.8-4.0 nm range corresponding to the nanoperiodicity of the structure. In the WAXS region, the crystal structure that is adopted by the salts in the different phases was reflected as multiple discrete scattering. Conspicuous changes in both WAXS and SAXS patterns were observed upon heating and cooling at temperatures consistent with those estimated by DSC. Results of the monocrystal analysis showed that the unit cell of nATMP·Br salts is monoclinic for n = 12 or triclinic for n = 18, and contains four or two molecules respectively. In both cases molecules are in a fully extended conformation and packed with the long alkyl chains and the phosphonium and bromide ions arranged in alternating non-polar and polar layers (Fig. 2). The alkyl chains are tilted respect to the layer planes and are deeply interpenetrated.

It could be preliminary concluded that nATMP·Br surfactants adopt a layered biphasic structure with features similar to that described for alkyltrimethylammonium surfactants previously studied by us.

References

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