Orientation of \( n \)-Hexadecane molecules in a single emulsion droplet revealed by microbeam two-dimensional SAXS-WAXS measurement

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Introduction

Alkanes are among the most basic components of soft materials; nevertheless the precise mechanism of crystallization has not been clarified. Metastable phase of \( n \)-alkanes, rotator phase, is a key for the understanding of the crystallization. Recently, the existence of the rotator phase in C16 \([1]\) in emulsion droplets was experimentally certified \([2]\), while the precise mechanism of crystallization through the rotator phase remains unsolved. For the study of the structural inhomogeneity in a single droplet, scanning microbeam X-ray scattering is a promising tool. In this study, we used scanning microbeam 2D-SAXS-WAXS in order to investigate crystal orientations in a single droplet.

Experimental

The sample used was C16 droplets in O/W emulsions. Samples of C16 with > 99 % purity were purchased from Sigma Chemicals and no further purification steps were taken. The O/W emulsion droplets were made by microchannel emulsification technique \([3]\), by which we obtained large droplets with nearly monodisperse size distribution. We added 1 wt.% concentration of emulsifier before emulsification.

Experiments were performed at BL-4A. The X-ray wavelength was 1.54 Å and the beam size was around 5 \( \mu \)m \( \times \) 5 \( \mu \)m. A large aperture X-ray Image Intensifier (270 mmφ) coupled X-ray CCD detector \([4]\) was used as a detector. The sample-to-detector distance was around 160 mm. Various positions in a droplet were irradiated by the microbeam X-ray and 2D-SAXS-WAXS image was simultaneously recorded.

Results & Discussion

Figure 1 shows the result of scanning microbeam SAXS-WAXS. A droplet with a diameter of around 50 \( \mu \)m was scanned by the microbeam X-ray. The SAXS-WAXS image from the center part of the droplet showed isotropic scattering pattern, while the SAXS-WAXS from the upper side oil-water interface and from the lateral side oil-water interface showed anisotropic scattering pattern. There is a decisive difference between the scatterings from the upper and the lateral sides of interface. In the SAXS-WAXS image from the upper interface, SAXS is observed in the vertical direction, and WAXS is observed in the horizontal direction. On the other hand, the SAXS-WAXS image from the lateral side interface was rotated by 90°. These results show that the C16 molecules align their axes parallel to the hydrophobic base of the surfactant. This finding strongly suggests that the surfactant at oil-water interface plays a precursor role for the crystallization.

References

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