Synchrotron Radiation X-ray Diffraction Study of Crystallization Processes of Cocoa Butter under Temperature Variation and Shear

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Tempering processes of chocolate is a prerequisite for controlling polymorphic crystallization of cocoa butter in chocolate from metastable Form III or IV to stable Form V which exhibits optimal melting and textural properties. In actual chocolate production processes, a tempering machine is usually employed and dynamic temperature variation under shear is applied. This study aims at observing the crystallization processes of cocoa butter in a growth cell which is subjected to synchrotron radiation X-ray diffraction (SR-XRD) during the variation of temperature of cooling-heating-cooling cycle (40→23→30→15 °C) under shear (shear rates, 800 sec^{-1} ~ 2400 sec^{-1}). In this growth cell, cocoa butter liquid was placed between two tubes having a clearance of 1.25 mm. The inner tube was rotating and temperature control was performed by temperature-controlled water which ran through the outer tube. Synchrotron radiation X-ray beam of a width of 1x1 mm^2 was inserted into the clearance domain of the growth cell, and the diffraction patterns from cocoa butter crystals were monitored with two two-dimensional detectors (sample-detector distances, 50 cm for small-angle diffraction, and 30 cm for wide angle diffraction) so that the subcell and long spacing structures were observed simultaneously.

Our preliminary experiments revealed that, during the temperature variation of 40→23 °C, cocoa butter crystallization without shear occurred quite slowly compared with shear-applied crystallization. However, it was observed that higher shear rate (2400 sec^{-1}) caused retardation of crystallization compared with that under lower shear rate (800 and 1600 sec^{-1}). In the three cases, Form V was observed by SR-XRD study.