Crystallisation, Polymorphism and Melting Properties of Triglycerides obtained by Dry Fractionation of Milk Fat

Elucidated using the Coupling of X-ray diffraction and DSC.

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Modification of milk fat composition to improve its nutritional and functional properties is of considerable interest for expanding its use in the food industry. Dry fractionation of milk fat, which permits to obtain fractions with variable triglyceride (TG) compositions and melting points, is commonly employed in the dairy industry. However, information on the crystallographic properties of the milk fat fractions is scarce.

From the same batch of anhydrous milk fat, one-step dry fractionation was performed at 21°C to obtain the olein (liquid) fraction and the stearin (solid) fraction. The TG compositions were determined using HPLC. The crystallographic and thermal properties of TG were investigated using the coupling of synchrotron radiation X-ray diffraction (XRD) and differential scanning calorimetry (DSC), with Microcalix [1].

The fractions displayed different TG compositions: stearin fraction was enriched in long-chain fatty acids, whereas olein fraction was enriched in short-chain and unsaturated fatty acids. On cooling at |dT/dt| = 1°C/min, two main types of crystals corresponding to double-chain length structures were characterised in the stearin fraction: \( \alpha \) \( 2L_1 \) (47.5 Å) and \( \beta' \) \( 2L_2 \) (41.7 Å) [2]. A triple-chain length structure was formed in the olein fraction: \( \alpha \) \( 3L \) (72.1 Å). Crystallisation of milk fat showed the formation of two 2L (47.3 and 41.6 Å) and one 3L (72.1 Å) lamellar structures of \( \alpha \) form [2]. Schematic representations of the 2L and 3L stackings are proposed to explain how a wide diversity of TG can accommodate to form these lamellar structures [2]. The melting properties of TG were investigated on subsequent heating at 2°C/min. Recrystallisations and polymorphic evolutions were characterised. The structural evolutions of TG molecules were related to the thermal events recorded simultaneously by DSC.

Such a study is important as fundamental knowledge for a better understanding of the phase behaviour and polymorphism of fats as a function of TG composition, and for technological applications in the food, cosmetic and pharmaceutical industry.