The Coupling of Synchrotron X-ray Diffraction and DSC to Build Solid-Liquid Triacylglycerol Phase Behavior vs. Temperature Diagrams

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Triacylglycerols (TG; esters of fatty acids and glycerol) are the main components of natural fats. Improving the knowledge about the crystallization properties of TG is of primary importance regarding their technological, functional and sensory properties.
High-melting point (HMP) TG and low melting point (LMP) TG were obtained by dry fractionation (21°C) from the same batch of anhydrous milk fat. TG compositions were determined using HPLC. The crystallographic and thermal properties of TG were investigated using the coupling of time-resolved synchrotron radiation X-ray diffraction (XRD) and differential scanning calorimetry (DSC) using Microcalix [1]. The crystals were observed using polarized light microscopy. The samples revealed different TG compositions, structural and thermal properties. The HMP TG were enriched in long-chain fatty acids, the LMP TG were 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 characterized in the HMP-TG: α $2L_1$ (47.5 Å) and β' $2L_2$ (41.7Å) [2]. A triple-chain length structure was formed in the LMP-TG: α 3L (72.1 Å). Structural models of the 2L and 3L stackings are proposed to explain how a wide diversity of TG can accommodate to form these lamellar structures [2]. Spherulites were observed for HMP-TG whereas needles were formed for LMP-TG. The melting properties of TG were investigated on subsequent heating at 2°C/min [3]. Recrystallisations and polymorphic evolutions of TG were characterized as a function of temperature and related to the thermal events recorded simultaneously by DSC. By creating a quantified solid-liquid phase behavior vs. temperature diagram, the amount of the solid and liquid phases and the relative proportion of each of the crystalline structures within the solid phase were determined [3]. Such a study is important as fundamental knowledge for a better understanding of the phase behavior and polymorphism of TG as a function of their fatty acid composition, and for technological applications in the food, cosmetic and pharmaceutical industry.

References cited: [1] Ollivon et al. (2006) J. Therm. Anal. Cal., 85, 219-224; [2] Lopez et al. (2006) Chem. Phys. Lipids, 144, 17-33; [3] Lopez et al. (2009) Chem. Phys. Lipids, in press online.