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AMORPHOUS LACTOSE CRYSTALLISATION KINETICS

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Abstract

The crystallisation kinetics of amorphous lactose were investigated at different relative humidity and temperature combinations. Relative humidity was controlled by placing the amorphous lactose in sealed pans containing saturated salt solutions. These pans were stored, for defined time periods, at temperatures ranging from 10-40°C above the glass transition temperature (T_g). The degree to which crystallisation had occurred was measured using both dynamic vapour sorption and isothermal microcalorimetry. The results showed crystallisation to be an all or nothing event, such that a direct measurement of the kinetics could not be obtained. This is not well accounted for by Avrami type models. It is proposed that the rapid crystallisation could be an autocatalytic effect as moisture is released during crystallisation, or a showering event as is seen in highly supersaturated lactose solutions. The latter is supported by the observation that experiments using Supertab (a blend of crystalline and amorphous lactose) show crystallisation at lower T_g conditions than is required for the crystallisation of 100% amorphous lactose.

As part of confirming the equilibrium time for amorphous lactose particles the diffusion rates were investigated. The diffusivity of water through lactose was estimated by fitting a model to the DVS results. Diffusivities of $3.4 \cdot 10^{-13}$, $1.4 \cdot 10^{-14}$, $7.6 \cdot 10^{-12}$ and $3.6 \cdot 10^{-13} \text{ m}^2\text{s}^{-1}$ were found for Supertab, spray dried, freeze dried and milled lactose, respectively.

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