Rate and Mechanism of Lactose Hydration

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Micronization of powders can lead to high-energy sites that probably are confined to the surface. Spray drying or freeze drying of the same material can lead to a bulk or contiguous high-energy material. We attempt to answer the following questions: Is the energy of the presumed amorphous state the same in both? Do we know with certainty the extent of amorphous content in either? Characterizing the rate and extent of water mediated conversion in the two high-energy materials via isothermal calorimetry and gravimetry has the potential for making this measurement and discrimination. Amorphous lactose (via XPRD) is exposed to 50 % RH in an isothermal microcalorimeter (TAM) and an isothermal microbalance (DVS). Heat flow or mass is measured as a function of time in a sorption region (before any crystallization) at 5 temperatures with n = 8 or 9. Friedman analysis shows the reaction to be more than one step. Formal kinetic analysis via NETZSCH Thermokinetics® suggested several potential mechanisms. XPRD is used to detect crystallinity in the product. Solid-state NMR is used to detect the presence of α and β forms present in reactant and product [1]. A plausible mechanism might be a three step consecutive process: A>B, 1st order (adsorption); B>C, rate limiting 1-dimentional diffusion (spray dried product consists of hollow microspheres with shell thickness of 0.07 – 0.30 μm [2]); C>D, 1st-order with catalysis by D (non-geometrical internal structural collapse [3]).

References

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