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Fig. 1. Example TGA thermograms of lysozyme (A) crystals (B) dried crystals. Conditions: samples heated from 30 to 210 °C; heating rate: 10 °C/min.

Fig. 2. PXRD patterns of lysozyme powders (A) unprocessed, (B) crystals, (C) dried crystals, (D) dried crystals milled for 3 minutes (3M), (E) dried crystals milled for 10 minutes (10M), and (F) dried crystals milled for 20 minutes (20M).

Fig. 3. Example DSC thermograms of lysozyme (A) unprocessed, (B) crystals, (C) dried crystals, (D) dried crystals milled for 3 minute (3M), (E) dried crystals milled for 10 minutes (10M), (F) dried crystals milled for 20 minutes (20M), (G) dried crystals milled for 30 minutes, (H) dried crystals milled for 45 minutes (45M), and (I) dried crystals milled for 60 minutes (60M). Conditions: samples heated from 30 to 210 °C; heating rate: 10 °C/min.

Fig. 4. Correlation between the milling time and the calorimetric unfolding enthalpies of the milled lysozyme crystals (A) the enthalpy of the crystalline content, (B) the enthalpy of the amorphous content and (C) the enthalpy of the total contents (crystalline and amorphous).

Fig. 5. Expanded DSC thermograms showing the unfolding transition peaks of mixtures of amorphous lysozyme (unprocessed): crystalline lysozyme (3M) samples at different ratios of (A) 3:7, (B) 5:5, and (C) 7:3. Conditions: samples heated from 30 to 210 °C; heating rate: 10 °C/min.

Fig. 6. FT-Raman spectra of dried crystals (line without marker), 3M sample (crystalline lysozyme) (cross), 20M sample (amorphous) (square) and 60M (denatured lysozyme) (triangle). Vibration modes are (B) back bone, (C) amide III and (D) amide I. The spectra were normalized using the methylene deformation mode at 1448 as an internal intensity standard.



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