

冷冻干燥中升华界面的临界温度实验 Critical Temperature of Sublimation Interface in Freeze-drying

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关键词: 冷冻干燥 塌陷温度 冻干显微镜 差示扫描量热法 玻璃化转变

摘要: 利用冻干显微镜(FDM)研究了二元水溶液冷冻干燥过程中升华界面的临界温度,采用差示扫描量热法(DSC)测量了溶液的共晶熔融温度和最大冻结浓缩溶液的玻璃化转变温度。实验结果表明,对于降温时发生共晶的溶液,临界温度为共晶熔融温度,否则将发生融化;对于降温时发生玻璃化转变的溶液,临界温度为塌陷温度,否则将发生塌陷。对于质量分数为5%蔗糖、10%葡萄糖、10%麦芽糖、10%聚乙烯吡咯烷酮(PVP)二元水溶液,微塌陷温度与塌陷温度的差值在2℃以内,微塌陷温度比玻璃化转变温度高1~3℃。 Freeze-drying microscopy (FDM) was used to investigate the critical temperature (T_{cr}) of sublimation interface during freeze-drying of binary aqueous solutions, and differential scanning calorimetry (DSC) was used to investigate the eutectic melting temperature (T_{em}) and the glass transition temperature of the maximally freeze concentrated solution ($T'g$). The experimental results showed that if a solute crystallized from solution during freezing step, the critical temperature was the eutectic melting temperature, or else a meltback would occur. If the solute remained amorphous during the freezing process, the critical temperature was the collapse temperature, or else the amorphous phase would undergo viscous flow. For 5% Sucrose, 10% glucose, 10% maltose, 10% PVP binary aqueous solutions, the microcollapse temperature (T_{mc}) and the collapse temperature (T_c) differed by less than 2℃, and T_{mc} was 1~3℃ above $T'g$.

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