Recrystallization of Silibinin Using a Supercritical Fluid Antisolvent Process

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Silibinin was recrystallized from its solutions using supercritical carbon dioxide as an antisolvent. Silibinin was dissolved in organic solvents (acetone and alcohols) at various concentrations. Carbon dioxide was injected into these solutions, thereby inducing supersaturation of the solution and particle precipitation. The effect of process parameters such as crystallizing temperature, solvent type, and the carbon dioxide injection rate on the solid state properties of silibinin was investigated. The influence of growth retardants on crystal habit and particle size was also examined. The recrystallized silibinin particles exhibited spherical crystal habit. Smaller crystals were obtained when crystallization took place at higher temperatures. Slower injection rate of antisolvent provided favorable conditions for a more random arrangement of silibinin molecules, and consequently the crystallinity of particles decreased when the antisolvent injection rate decreased. Amorphous silibinin particles were produced when urea was used as a growth retardant.