VISUALIZATION OF SPRAY AND PARTICLE CHARACTERISTICS OF SUPERCRITICAL ANTISOLVENT PRECIPITATION OF PMMA, PVP, AND COPOLYMERS

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Abstract

Supercritical antisolvent (SAS) precipitation process experiments were performed to directly relate spray properties to particle charateristics for a variety of operating conditions and for several polymers and copolymers. A high-resolution imaging system was used to study the spray characteristics of polymer/ethanol solutions in pressurized carbon dioxide. The visualizations provide high magnification video of the jet emerging from the spray nozzle and of the spray at various distances away from the nozzle tip. The precipitated polymer particles were characterized using a scanning electron microscope. Image processing and analyses were developed to measure jet breakup lengths, drop properties, and particle characteristics. Studies were conducted with poly methyl methacrylate (PMMA), poly vinyl pyrrolidone (PVP), and copolymers from the monomers methyl methacrylate and vinyl pyrrolidone. Polymers and copolymers were processed through SAS to observe the effects of their solubility in ethanol on the SAS spray characteristics and the resulting particles. The solubilities of the copolymers were influenced by varying the methyl methacrylate to vinyl pyrrolidone ratio, which reduces the solubility in ethanol. Cloud point experiments were used to evaluate the saturation concentrations in ethanol for the polymers and copolymers. Process visualizations revealed that the spray of each polymer or copolymer solution into carbon dioxide had similar jet breakup lengths and droplet diameter distributions, and that these spray characteristics appear to be closely associated with the solvent (ethanol) properties. Distinct spray regimes were observed by varying system pressure. The SEM images showed variation in particle size distributions, and the presence of large particles and microballoons, with significant influence identified relative to the solubility of the polymer or copolymer in ethanol and to certain processing conditions.