585b Visualization of the Effects of Processing Conditions on the Spray Characteristics in Sas Precipitation

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A high-resolution imaging system is used to study supercritical antisolvent (SAS) precipitation. A 1 wt% solution of poly (L-lactic) acid in methylene chloride was sprayed into carbon dioxide at several bulk fluid conditions. Visualizations of the spray were obtained at various distances from the nozzle. Image processing techniques were developed to provide micrometer resolution measurements of jet breakup lengths and droplet sizes.

Constant density experiments at 0.313 g/cm3 produced radically different spray characteristics. At 83 bar, 313 K and 88 bar, 318 K atomization into droplets was observed. At 93 bar, 323 K, a gas-like plume was observed, and no distinct droplets were visible in the images. However, the particles that were produced from these three conditions had similar size distributions as shown by SEM images. For constant temperature experiments at 323 K, the average droplet diameter was similar when droplets were visible, but the droplet number density (number of droplets in the field of view) varied with the pressure and density. At 83 bar, 0.235 g/cm3 and 88 bar, 0.270 g/cm3 atomization was observed, and gas-like diffusion was observed at 93 bar, 0.313 g/cm3. In each case where atomization was observed, the droplets initially increased in size and then decreased as they traveled further from the nozzle, indicating that the droplets first swell and then dissipate. For increasing temperature and pressure at constant density and increasing pressure and density at constant temperature, (1) jet breakup length decreases, (2) droplets dissipate closer to the nozzle, and (3) mixing shifts from atomization to gas-like diffusion.

Our studies show that spray characteristics can be controlled by changing the bulk fluid temperature and pressure to obtain regimes where mixing occurs by atomization and droplet dissipation or where mixing occurs by gas-like diffusion. We will also discuss how the spray behavior and the particle precipitation process can be affected by varying the solute-solvent interaction. Visualizations of the spray characteristics and resulting particles provide a better understanding of the supercritical antisolvent precipitation process.