

## **550a Preparation of Polyetherimide Nanoparticles by Electrospray Drying, and Their Use in the Preparation of Nano-Sized Carbon Molecular Sieve (Cms) Adsorbents and Membranes**

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The goal of this research is to prepare nano-sized CMS particles to be utilized as adsorbents, and in the preparation of mixed matrix membranes. These CMS nanoparticles are prepared by either conventional or plasma pyrolysis of polymeric nanoparticle precursors. The latter are prepared using the technique of electrospray drying. In this paper the focus is on the preparation of fine and monodisperse polyetherimide (PEI) particles. The electrospray drying experiments were operated in the cone-jet mode by making use of a strong electric field (metallic nozzle connected to a high voltage source), in which a pendular droplet deforms into a conical shape and then passes through a weaker electric field (shielding electrode) at the same polarity as the first one. The charged particles were neutralized with the aid of corona discharge. Experiments have been carried out in order to find the optimum window of operation for the electrospray system. The effects of three key parameters have been investigated in detail, namely applied voltage, liquid flow rate, and polymer concentration. Four distinct spray modes were observed under different operating conditions, namely, single cone spray, multiple-cone spray, dripping, and micro-dripping. For a given concentration of PEI, a stable and monodisperse electrospray could only be established within certain range of liquid flow rates (defined as the cone-jet domain) and applied voltage differences. Within the cone-jet mode the initial size of the droplets can be controlled through the flow rate, and the concentration of PEI. The PEI solution concentration affects the physical properties of the liquid (viscosity, density, surface tension, and more importantly conductivity), which determine, in turn, the electrospray performance; too high of a concentration causes premature drying of the droplets at the orifice tip, while too low of concentration causes solvent saturation at the collection cup. Our experiments indicate that, for a given voltage, and liquid flowrate, an optimal concentration exists that optimizes both the size and the structure of the resulting nanoparticles. The liquid flow rate has the most important effect in determining the particle size. Again an optimal range of values often exists. The particles obtained with flow rate of 0.05 ml/h, at the voltages of 13.6 and 7 kV (on the capillary and ring respectively), with a 0.05 wt% PEI solution had a narrower size distribution (with an average size of 200nm), and at the same time better morphology (dense spherical) in comparison with those produced with other liquid flow rates. Increasing the flow rate at constant applied voltage and PEI concentration drastically changes the morphology of the agglomerated particles to hollow shells for a given capillary size. The resulting CMS nanoparticles have been characterized by a variety of surface characterization techniques. Their pore size distribution has been measured by adsorption techniques. The preparation of mixed matrix membranes using these CMS nanoparticles is currently ongoing and single and mixed gas experiments will be presented at the meeting.