Manufacture of Regularly Shaped Sol-Gel Pellets

For mass production, an extrusion process is superior to a spray process.

John H. Glenn Research Center, Cleveland, Ohio

An extrusion batch process for manufacturing regularly shaped sol-gel pellets has been devised as an improved alternative to a spray process that yields irregularly shaped pellets. The aspect ratio of regularly shaped pellets can be controlled more easily, while regularly shaped pellets pack more efficiently. In the extrusion process, a wet gel is pushed out of a mold and chopped repetitively into short, cylindrical pieces as it emerges from the mold. The pieces are collected and can be either (1) dried at ambient pressure to xerogel, (2) solvent exchanged and dried under ambient pressure to ambigels, or (3) supercritically dried to aerogel. Advantageously, the extruded pellets can be dropped directly in a cross-linking bath, where they develop a conformal polymer coating around the skeletal framework of the wet gel via reaction with the cross linker. These pellets can be dried to mechanically robust X-Aerogel.

The extrusion process begins with the preparation of a suitable sol. This is accomplished online by mixing tow solutions as they flow from two separate containers towards the mold. A typical sol that yields a gel that can be cross-linked easily is made by mixing two cold solutions: One solution consists of 4.5 volume parts of acetonitrile, 0.482 volume parts of aminopropyltriethoxysilane, and 1.45 volume parts of tetramethoxysilane. The other solution consists of 4.5 volume parts of acetonitrile and 1.5 volume parts of water.

The cold sol is equipped with a plunger. As soon as the sol gels in the mold, the newly formed gel is pushed out of the mold and is chopped to the desired length. When the gel mold is exhausted, the plunger is retracted and the process repeated. An important consideration in the process is that the sol needs to have formed a gel, which is stable enough to slice before the extrusion process is begun. The process can be used with nearly any sol-gel formulations, but high throughout requires a fast gelling sol such as the one obtained by using aminopropyltriethoxysilane (APTES)/tetramethoxysilane (TMOS) mixtures. In this formulation, control of the gelation rate requires that the precursor solutions must be refrigerated prior to filling the mold. In an extension of the basic process described here, multiple molds with plungers can be connected to a liquid manifold connected to a sol-preparation apparatus and mounted together to facilitate repeated filling with sol and automatic extrusion.

The most benefit from this process is realized when it is combined with chemical cross linking as pellets emerge from the mold. A suitable cross linker is a diisocynanate (e.g., Desmodour N3200 from Bayer Corporations, or equivalent), which, owing to the small size of the pellets, diffuses and reacts quickly with the internal surfaces of the porous nanoparticle framework of the wet gels. The wet-pellets are subsequently dried from supercritical carbon dioxide, yielding mechanically robust colorless, slightly translucent pellets of X-Aerogel. Other cross-linker systems such as epoxies and polyolefins have been also investigated.

This work was done by Nicholas Leventis, James C. Johnston, and James D. Kinder of **Glenn Research Center**. *Further information is contained in a TSP (see page 1)*

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