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ACCEPTED MANUSCRIPT

Interconnectivity imaged in three dimensions: nano-particulate silicahydrogel structure revealed using electron tomography

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Abstract

We have used Electron Tomography (ET) to reveal the detailed three-dimensional structure of particulate hydrogels, a material category common in e.g. controlled release, food science, battery and biomedical applications. A full understanding of the transport properties of these gels requires knowledge about the pore structure and in particular the interconnectivity in three dimensions, since the transport takes the path of lowest resistance. The image series for ET were recorded using High-Angle Annular Dark Field Scanning Transmission Electron Microscopy (HAADF-STEM). We have studied three different particulate silica hydrogels based on primary particles with sizes ranging from 22 nm to 3.6 nm and with pore-size averages from 310 nm to 18 nm. Here, we highlight the nanostructure of the particle network and the interpenetrating pore network in two and three dimensions. The interconnectivity and distribution of width of the porous channels were obtained from the three-dimensional tomography studies while they cannot unambiguously be obtained from the two-dimensional data. Using ET, we compared the interconnectivity and accessible pore volume fraction of pore size, based on direct images on the nanoscale of three different hydrogels. From this comparison, it was clear that the finest of the gels differentiated from the other two. Despite the almost identical flow properties of the two finer gels, they showed large differences concerning the accessible pore volume fraction for probes corresponding to their (two-dimensional) mean pore size. Using 2D pore size data, the finest gel provided an accessible pore volume fraction of over 90%, but for the other two gels the equivalent was only 10-20%. However, all the gels provided an accessible pore volume fraction of 30-40% when taking the third dimension into account.

Keywords: electron tomography; silica nanoparticle gel; colloidal silica gel; porous soft materials; accessible volume fraction; interconnectivity.

Highlights:

- Three-dimensional nanostructure of particulate silica hydrogels revealed using electron tomography
- New insights into how to determine the pore network interconnectivity and accessible pore volume fraction as a function of probe size
- The pore connectivity information provides information about the degree of pore throats along the pore network
- The accessible pore volume fraction, interconnectivity and degree of pore throats were determined for three different gels illustrating the ability of to perform 3D reconstruction and evaluation of the pore network using electron tomography and also

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