Abstract

Silica foams with varying porosity content and distributions have been prepared through steam-heating and freeze-casting routes using slurries containing ovalbumin as organic binder, as well as sucrose and colloidal silica as additives. The pore structure and size distribution in the foam structure is controlled through optimization of the process parameters for achieving the desirable properties. On the basis of rheological studies, the slurry with optimum combination of viscosity, flowability, and foam-stability in wet-condition for both steam-heated (SH) and freeze-cast (FC) foams has been found to contain 38 vol.% solid (SiO_2 + 5 wt.% Al_2O_3) loading, 30 vol. % ovalbumin, and 15 wt.% sucrose. In the steam-heating process, the wet foams cast into aluminum mold were exposed for 15 minutes to steam, and then dried in an oven at 100 ^oC for 1 h. Unlike the drying schedule required in conventional processes for foam preparation, the duration of drying for SH foams is drastically reduced, and formation of crack-free green bodies is observed, because non-uniform shrinkage is inhibited. During steam-heating, the cell-walls are not only restrained from shrinking by intra-cellular steam pressure, but are also simultaneously strengthened by coagulation of protein present in ovalbumin (binder). Sucrose addition aides in both uniform wetting of the cell-walls and moisture retention during steam-heating. In the freeze-casting route, the silica slurries with the aforementioned optimized composition were first subjected to direct foaming by tumbling, followed by casting in vaseline-coated aluminum molds and freezing using liquid nitrogen. The frozen samples were dried at low temperature (-5°C to -10°C) for 24 h in vacuum (10^{-2} Torr) to facilitate sublimation of ice crystals without melting, leaving behind a rigid porous structure. The FC foam was kept at 100 °C for an hour in an oven to remove the residual liquid wetting the pore-walls, and crack-free green bodies were obtained. The amount of final porosity in the FC foams is related to both air-entrapment during tumbling (foaming) and aqueous content of the slurry. The green bodies of both SH and FC foams were heated at the rate of 1°C /minute to 1150 °C, and sintered at this temperature for 1 h.

Densities have been estimated on the basis of both Archimedes principle and dimensional measurements, whereas absence of cracks in the sintered foams has been confirmed by both X-ray radiography and X-Ray micro-Computed Tomography (CT). Quantitative analyses of microstructures recorded using scanning electron microscopy have shown the presence of near-spherical shaped pores with size distributions peaking at ≈250–300 μm and 50-100 μm in sintered SH and FC foams, respectively. The pores in both types of foams appear interconnected through finer pores of ≈15-25 μm size along their walls. The pore distribution and interconnected or closed nature of porosity in silica foam as examined with the help of X-Ray micro-CT is found to be in close agreement with the results of microstructural studies. Comparison of pore sizes of SH and FC foams has shown the pore size obtained in the latter foam to be much finer and more uniform. For increase in porosity content of the sintered SH foam from 55 to 90 vol.%, the static Young′s modulus decreases from 544 MPa to 14.4 MPa, whereas compressive strength falls from 8.6 MPa to 0.3 MPa. On the other hand, static Young's modulus and compressive strengths are found to be in the ranges of 25.4-683 MPa and 0.3-12.7 MPa, respectively for the sintered FC foams having ≈ 60-88 vol.% porosity content. The compressive strength depends on pore sizes, pore distribution, strut thickness, and the meso-porosity present inside the cell walls of the porous structure. It is also possible to predict both static Young's modulus and compressive strength using power-law relations suitable for cellular solids.

***Keywords*:** Silica, Foams, Steam-heating, Freeze-casting, Mechanical Properties.