

PHYSICOCHEMICAL APPROACHES FOR THE SYNTHESIS AND CHARACTERISATION OF MESOPOROUS MATERIALS

John Bartlett

School of Natural Sciences, University of Western Sydney Locked Bag 1797
Penrith South DC NSW 1797 Australia

Synthetic chemical strategies provide the “nanostructural engineer” with a range of well defined precursors and techniques for the production of nanoparticles, nanofibres and nanopowders with tailored properties, thus providing an artist’s palette for the construction of materials with controlled nanoarchitectures. However, simple physicochemical approaches for controlling the internal structure and morphology of nanomaterials are often overlooked in modern sol-gel chemistry, even though they provide complementary tools for controlling nanostructural evolution..

In this work, the effect of ionic strength and solids content on the nanostructure and morphology of mesoporous titania gel microspheres obtained by spray-drying nanoparticles has been investigated using SAXS, SANS, electron microscopy and N₂ adsorption. Scattering data were obtained from the as-prepared powders over three decades in q , revealing hierarchical nanostructures that were probed on length scales from 0.5 to > 1000 nm. Absolute scaled measurements in this range show two exponential regimes and a transition region, which were modelled to obtain:

- the surface area of the nanosized pores (high- q Porod region);
- the total porosity of the microspheres (transition region);
- the average size of the microspheres (low- q Porod region).

The macroscopic structure (morphology) of the gel particles was also found to be strongly influenced by the ionic strength. At low ionic strength, fragmentation of the atomised droplets occurs during drying, leading to the formation of fine powder, in addition to gel particles with distorted shapes (*e.g.* donuts). In contrast, at high ionic strength, the formation of well-defined spherical particles was observed. It is shown that these effects can be attributed to the relative magnitudes of the Laplace and osmotic pressures immediately before the sol-to-gel transition. If the Laplace pressure exceeds the osmotic pressure, then well defined spherical particles are obtained. Conversely, distorted particles are formed when the osmotic pressure exceeds the Laplace pressure. For a given droplet with rapidly increasing osmotic pressure during the fast process of drying, the relative magnitudes of the Laplace and osmotic pressures before the sol-to-gel transition thus determine whether compression or fragmentation is favoured.

The relationship between the interaction potential (ionic strength) in the sols and the porosity (nanostructure) and morphology (macro/microstructure) of the resulting hierarchical materials will be discussed, together with prospects for the use of such aqueous sol-gel processing in other multicomponent, hierarchical systems.