

Taken from Carroll, Nick J. and Pylypenko, Svitlana and Atanassov, Plamen B. and Petsev, Dimiter N. Microparticles with bimodal nanoporosity derived by microemulsion templating. *Langmuir* 25:13540-13544 (2009).

The silica precursor solution was prepared by dissolving 1.82 g of cetyltrimethylammonium bromide (CTAB) in 20 g of DI water under vigorous stirring at 40 °C until the solution was clear. Next, 5.2 g of tetraethylorthosilicate (TEOS, Purum >98%) and 0.57 g of 1 N hydrochloric acid were added to the mixture under vigorous stirring at room temperature for 30 min to hydrolyze and dissolve the TEOS monomer. The measured acidity of the hydrolyzed sol showed pH  $\approx$  2. The oil phase was prepared by dissolving a modified polyetherpolysiloxane/dimethicone copolyol surfactant with the trade name ABIL EM 90 (Degussa) in hexadecane (3 wt %). The aqueous siliceous precursor solution was then added to the oil phase and subsequently emulsified by brisk shaking of the vial. The emulsion was transferred to a 1000 mL round-bottom flask and heated to 80 °C under a reduced pressure of 70 mTorr for 3 h. The particles were collected and centrifuged, and the supernatant oil removed, followed by calcination in air at 500 °C for 5 h to remove the templating surfactant. The carbon precursor was prepared by dissolving 475 mg of sucrose in 1 mL of 2M H<sub>2</sub>SO<sub>4</sub> and then adding 2.6 mL of acetone. Addition of acetone was necessary to obtain good wetting of silica particles with sucrose solution. The resulting solution was added to 200 mg of silica particles in increments of 100  $\mu$ L and was dried between infusions. After all solution was added to the silica particles, they were dried in the oven at 70 °C overnight. This was followed by pyrolysis at 900 °C in a N<sub>2</sub> atmosphere for 4 h, with the ramp rate at 3 °C per min. After pyrolysis, silica was etched in 6 M KOH for 4 days. The material was then filtered, washed five times with DI water, and dried in the oven at 70 °C.