517d Comparison of the Polymerization Kinetics of Bis(Triethoxysilyl)Ethane and Methyltriethoxysilane under Acidic Conditions

Jyothirmai Ambati and Stephen E. Rankin

Synthesis of organic-inorganic hybrid silica materials using organically modified bridged precursors provides for more structural and functional possibilities than can be achieved by mixtures of non-bridged silanes. Bridged precursors are of particular interest for the synthesis of ordered mesoporous materials with organic groups built into the walls of the materials. To allow rational design of these materials, predicting their properties a priori is important because there is little scope for refining them after their synthesis. Prior knowledge of the reaction kinetics of the precursors would give an idea about the point at which a gel will form, and the distribution of the components in the final product. Comparison of the hydrolysis and condensation kinetics of the bridged and their counterpart non-bridged silanes would help understand why, for instance, a bridged silane will gel and form a solid quickly while the non-bridged counterpart fails to produce a solid material over a much longer time.

In the present study, bis(triethoxysilyl)ethane (BTESE) is compared with methyltriethoxysilane (MTES). Their hydrolysis and condensation reactions are monitored in situ by ^{29}Si NMR and FT-IR spectroscopy. Polarization transfer using the DEPT pulse sequence is used to enhance the ^{29}Si NMR sensitivity without requiring a paramagnetic relaxation agent. A mathematical model is developed and solved to determine the rate constants of MTES and BTESE polymerization. The model is applied under conditions where both monomers remain homogeneous throughout the experiment. However, it is found that the polymerization products of BTESE are prone to the loss of NMR signal during the experiments at certain initial concentrations of the monomer. The loss of signal can be attributed to the precursor entering into an environment where fast tumbling cannot occur, most likely due to microphase separation.