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Synthesis of Advanced Hybrid Polymeric Nanomaterials and Characterization of Novel Silsesquioxanes with Desirable Superhydrophobic Coating Properties

Thesis

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Synthesis of Advanced Hybrid Polymeric Nanomaterials and Characterization of Novel Silsesquioxanes with Desirable Superhydrophobic Coating Properties

Abstract

Understanding and controlling the hydrolysis and condensation of trialkoxy and tetraalkoxy silanes in acidic medium (**Figure A**) has not been fully realised. In this research study, both the understanding and control has been achieved using methods developed from the Vitolane™ process invented by TWI Ltd¹ our industrial collaborators. The Vitolane™ process involves the synthesis of 3-methacryloxypropylsilsesquioxane resin from the hydrolytic condensation of 3-methacryloxy-propyltrimethoxysilane (MPTMS) in the presence of methanol, water and an acid catalyst (A-system). The reaction was repeated with two starting materials; 3-methacryloxy-propyltrimethoxysilane (MPTMS) and n-propyltrimethoxysilane (nPTMS) to form the AZ-system. It was found that with certain compositions, the reaction quickly reaches a pseudo equilibrium hence the hydrolysis rate constant could be determined. The instrumental analysis using Maldi-ToF-MS, HPLC, GPC, TGA, GCMS, DLS, DSC, FTIR and CHN analysis of both types of resins gave results that suggested the organic-inorganic hybrid silsesquioxanes obtained had the expected chemical composition and unique physical properties.

This study was further extended to Stöber sphere silica nanoparticles aimed at extending our understanding from the above hydrolysis and condensation mechanistic

study to the synthesis of Stöber silica nanoparticles ² of various sizes (**Figure B**). The synthesis follows a similar pattern as the Vitolane™ process but using TEOS as starting material and ammonium hydroxide base instead of acid as in the original Vitolane™ process.

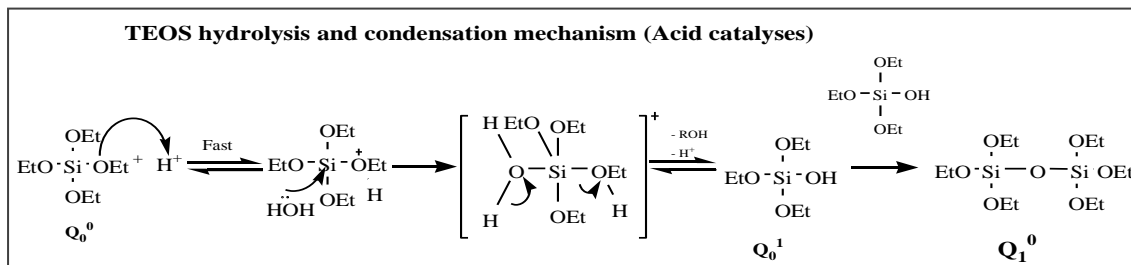


Figure A

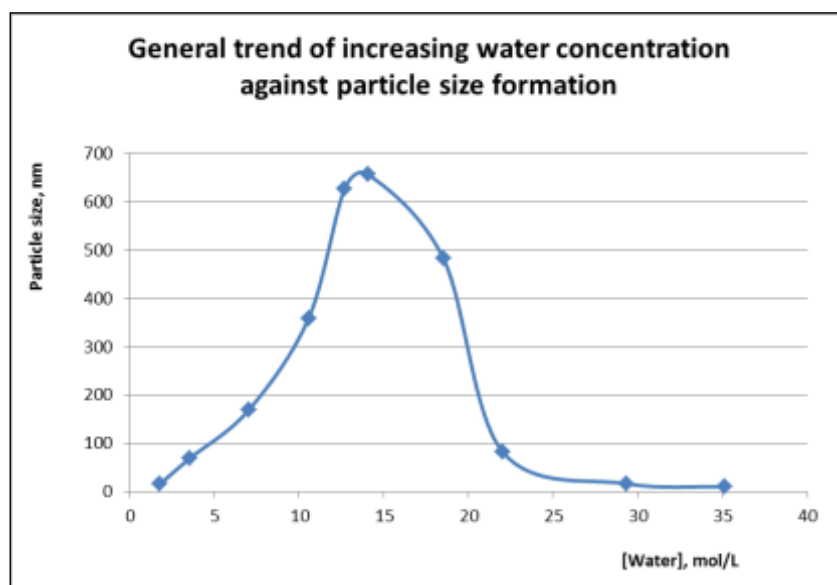


Figure B

The Stöber spheres study was carried out so we could add them to Vitolane in order to give rough (on the nanoscale) surfaces that would be superhydrophobic. The Stöber spheres were characterized using transmission electron microscopy (TEM), X-ray photoelectron spectroscopy (XPS) and dynamic light scattering (DLS) to investigate the particle size formation. The Stöber spheres obtained were of varied sizes depending upon the way they were prepared (**Figure B**).