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

Thesis

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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\(^{TM}\) process but using TEOS as starting material and ammonium hydroxide base instead of acid as in the original Vitolane\(^{TM}\) process.

![TEOS hydrolysis and condensation mechanism (Acid catalyses)](image)

Figure A

![General trend of increasing water concentration against particle size formation](image)

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).