Quantitative determination of grafted silane amount on modified silica nanoparticles

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Extreme weather conditions (ice, abrasive dusts…) constitute a real issue for the durability and the performance of polymer-based composites used in several industrial sectors such as automotive, wind energy and aerospace. Common active ice protection strategies which rely on hot bleed air or chemical fluids generally result in an increase in materials weight and cost and raise environmental concerns. To address this problem, the introduction of innovative multi-functional silica nanoparticles into the composite matrix constitutes an alternative way of improving both its erosion resistance and ice repellency with no loss in stiffness.

Silica nanoparticles have been synthesized by a refined Stöber method and subsequently functionalised by reaction with organosilanes [1]. Thus, nanoparticles have been designed to bear two types of chemical groups: one enables further covalent bonding with the composite matrix (i.e epoxy) to improve interfacial adhesion, the other (of alky or fluorine nature) confers hydrophobicity to the particles and so ice-repellency to the composite surface.

In this contribution, the implementation of quantitative methods to determine both the number of silanol on pristine silica nanoparticles and the functionalisation degree of organically modified ones are described. Depending on the considered organosilane, relevant analytical methods have been selected and optimized: bulk-analysis techniques such as solid-state MAS ^{29}Si NMR and elemental analysis and surface-analysis techniques such as X-Ray Photoelectron spectroscopy (XPS). The combination of these techniques leads to fruitful information about the chemical structure of the organo-modified silica nanoparticles.

[1] A. Taylor, G. G. Durand, M. Alvarez Tirado, N. Sid, S. Mycock, "Functionalisation Method for Metal Oxide Particles", *WO2017/093759A1*. **2017**.