Synthesis of urethane-norbornene star monomers. Synthesis of aerogels via ring opening metathesis polymerization of those monomers

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ABSTRACT

This study investigates the synthesis and characterization of synthetic polymer aerogels based on star-shaped urethane-norbornene monomers. The core of those monomers is based either on an aromatic/rigid (TIPM/Desmodur RE), or an aliphatic/flexible (Desmodur N3300) triisocyanate. Terminal norbornene (NBE) groups (3 at each of the 3 star branches) were polymerized via ROMP using the 1st generation Grubbs’ catalyst. The polymerization conditions were optimized by varying the amount of catalyst. Different drying conditions (from pentane at 50 °C, freeze-drying from t-butanol, SCF CO₂ drying) were studied. Monomers were characterized with ESI-MS and ¹H NMR. Aerogels were characterized with ATR-FTIR and solid-state ¹³C NMR. The porous network was probed with N₂-sorption and SEM. The thermal stability of monomers and aerogels has been studied with TGA, which also provides evidence for the degree of crosslinking. At low densities (< 0.1 g cm⁻³) all aerogels are highly porous (porosity > 90%) and mostly macroporous. Interestingly, aerogels containing the aliphatic/flexible core are fragile, whereas aerogels containing the aromatic/rigid core are plastic, and at low densities (0.03 g cm⁻³) foamy. At higher densities (0.2–0.7 g cm⁻³) all materials are stiff, strong, and hard. All aerogels of this study consist of aggregates of nanoparticles, the size of which varies with density and depends on the content and nature of the monomer and the rigidity/flexibility of the polymeric backbone.

SUBJECT AREA: Synthetic polymer aerogels

KEYWORDS: aerogels, star monomers, ring opening metathesis polymerization (ROMP), polymeric materials, ruthenium
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NanoHybrids: New generation of nanoporous organic and hybrid aerogels for industrial applications: from laboratory to pilot scale production

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