Synthesis and Relaxation Study of Poly(dimethylsiloxane) (PDMS)/Polyhedral Oligomeric Silsesquioxane (POSS) Composites

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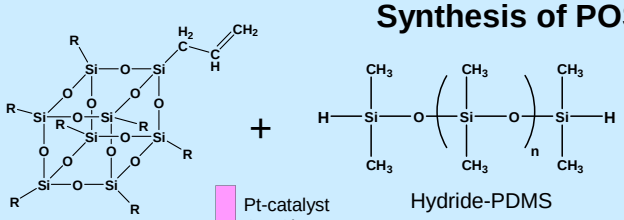
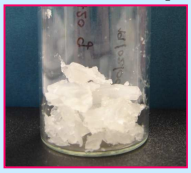
Abstract Three dumbbell shape PDMS/POSS composites were synthesised via a Pt-catalysed hydrosilylation reaction with the molecular weights of the PDMS middle chain ranging from 580 to 24,000 g.mol^{-1}. The reaction products were characterised by ^{1}H NMR, FT-IR spectrometry and gel permeation chromatography (GPC). The impact of POSS on the molecular mobility of the PDMS middle chain was then studied by using the ^{1}H spin-spin (T_2) relaxation NMR technique. Covalent incorporation of POSS into PDMS polymer leads to a change in relaxation showing that the effective PDMS middle chain mobility is restricted when it is end-capped with POSS. In addition, the composites consisting of longer PDMS middle chains show increasing molecular mobility.

Introduction

Polymer nanocomposites have been widely researched because the combination of polymer and nanoparticle provides an opportunity to modify the thermal, electrical, optical, or mechanical properties of the composite materials. Composites of Polydimethylsiloxane polymer (PDMS) and polyhedral oligomeric silsesquioxane (POSS) is a novel nanocomposite systems that has been widely studied. However, most of POSS/PDMS nanocomposites have been synthesised in the form of cross-linked and copolymer materials. Only a few studies of POSS/PDMS triblock polymers have been made. Therefore, the primary aim of this work is to produce the simplest model of the PDMS/POSS nanocomposites and to study this simple system before extending to more complicated systems.

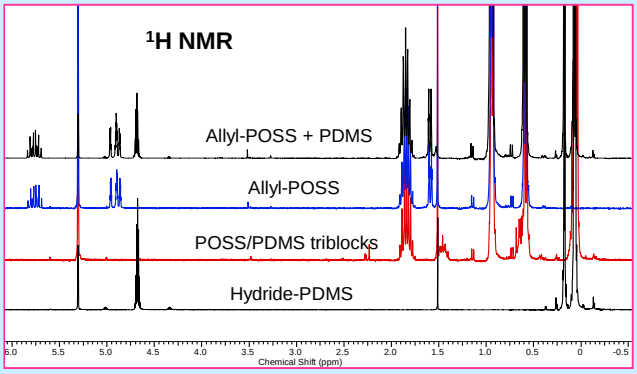
Synthesis of POSS/PDMS triblock polymers




POSS-0.58k PDMS-POSS

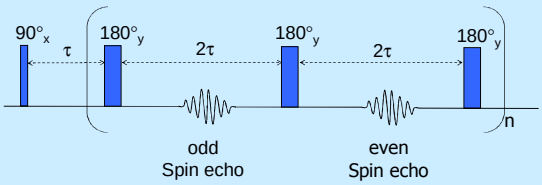

POSS-17.5k PDMS-POSS


POSS-24k PDMS-POSS



Sample Name	Mn	Mw	PDI
Allyl-POSS	821	835	1.02
0.58k hydride-PDMS	777	1040	1.34
17.5k hydride-PDMS	6330	25700	4.06
POSS-0.58k PDMS-POSS	2160	3370	1.56
POSS-17.5k PDMS-POSS	11100	190000	17.1

Spin-spin (T_2) relaxation NMR study

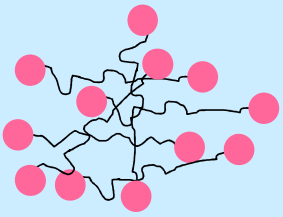


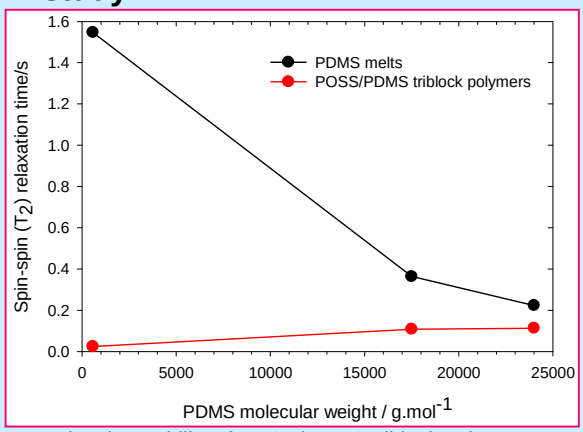
The Carr-Purcell-Meiboom-Gill (CPMG) pulse sequence diagram

The DISCRETE algorithm:

$$y(t) = \sum_{i=1}^{n} y_i^0 \exp\left(-t/T_{2i}\right) + b$$


PDMS melts


POSS/PDMS triblock polymers



Molecular mobility of POSS/PDMS triblock polymers in comparison with PDMS melts.

Conclusions

- ^{1}H NMR data can be used to confirm that POSS/PDMS triblock polymers were successfully synthesised by the Pt-catalysed hydrosilylation reaction.
- Covalent incorporation of POSS into PDMS polymers shows reinforcement of the composite materials.
- Molecular mobility of the POSS/PDMS triblock polymers is directly related to the length of PDMS middle chain.

Future work

- Synthesis of more POSS/PDMS triblock polymers by extending PDMS middle chain
- Rheology measurements of POSS/PDMS triblock polymers
- Analysis of molecular structure of POSS/PDMS triblock polymers using SANS
- Analyse physical property of POSS/PDMS triblock polymers by DSC
- Diffusion NMR measurements of POSS/PDMS triblock polymers

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