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European Polymer Journal

journal homepage: www.elsevier.com/locate/europolj

Macromolecular Nanotechnology

Selective wetting of carbon nanotubes in rubber compounds – Effect of the ionic liquid as dispersing and coupling agent



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ARTICLE INFO

Article history:

Received 9 September 2015

Received in revised form 24 November 2015

Accepted 27 November 2015

Available online 7 December 2015

Keywords:

Carbon nanotubes

Ionic liquid

Filler dispersion

Selective wetting

ABSTRACT

The role of the room temperature ionic liquid, 1-decyl 3-methyl imidazolium chloride (DMIC) as dispersing and coupling agent in multi-walled carbon nanotubes (CNTs)/styrene butadiene rubber (SBR) compounds was investigated. The kinetics of the selective wetting and dispersion of CNTs, which were pre-treated by DMIC, in SBR compounds was studied by means of two methods: (i) *the online measured electrical conductance* and, (ii) *the modified wetting concept*. It was found that DMIC shows a good compatibility to SBR and improves the CNT dispersion significantly. A DMIC layer, which is physically pre-bound to the CNT surface, is partly or completely replaced by the SBR molecules during mixing as a result of thermodynamic driving forces. The filler network formation of CNTs in SBR and its effect on the electrical and mechanical properties of the compounds were characterized and discussed by taking into consideration the selective wetting of CNT surface.

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1. Introduction

Thanks to the impressive mechanical, thermal and electrical properties carbon nanotubes (CNTs) as novel functional fillers in polymers can offer attractive potential applications. However, the results often lie under expectation, because CNTs are difficult to process due to their low dispersibility and their tendency to form aggregates [1,2]. Several strategies have been proposed to improve the compatibility between polymer matrices and CNTs by surface modification of CNTs with carboxyl groups or silanes [3–10]. CNTs can be modified through *in-situ* polymerisation of polyethylene (PE) oder polypropylene (PP) on CNT surface with help of metallocene based complexe [11–13]. The CNT dispersion and mechanical dynamical properties as well as thermal stability are significantly improved. However, in some cases the composites show lower electrical conductivity. It was discussed that the formation of an thick insulation layer of the bound polymer may be the reason for that. Otherwise, the covalent functionalization may disrupt the π -networks of CNTs and thereby reduce their inherent physical properties. Both strong acids and organic solvents, which were used for CNT functionalization often lead to environmental pollution, equipment corrosion and health hazards. As a alternative, recently, ionic liquids (ILs), a kind of

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molten salt with nearly zero vapour pressure and high thermal stability, have been used for functionalization of CNTs. Ionic liquids could provide a facile and promising method to modify the surface properties of CNTs by means of cation- π interaction. The review article made by Polo-Luque et al. [1] gave an overview on the use of ionic liquids as dispersing agent for CNTs in different thermoplastics like vinylidene fluoride-hexafluoropropylene copolymer [14], polystyrene [15], polycarbonate [16,17], polyurethane (PU) [18] as well as polymethylmethacrylate (PMMA) [19]. Recently, our research group has intensively used ionic liquids as coupling and dispersing agent for CNTs in different rubbers [20–23]. We found that the IL plays a major role in enhancing the level of CNT dispersion and thus, good physical properties can be achieved. However, in contrast to the chemical modification the physical cation- π bondings between IL molecules and CNT surface may be dissolved and replaced by the rubber-CNT bondings under thermodynamic driving forces during the mixing process that leads to a completely different wetting behavior of the CNTs. Thereby, the specific adjustment of the composite performance by controlling the rubber-filler interphase becomes very difficult and has not been investigated so far. In this regard, in the present work the selective wetting behavior of CNTs by IL and rubber along the mixing process will be quantitatively characterized by our *modified wetting concept* and the effectiveness of IL as dispersing and coupling agent for CNTs on the electrical and mechanical properties of the SBR compounds will be characterized and discussed.

2. Experimental

2.1. Materials and composite preparation

Solution styrene butadiene rubber (S-SBR) used was SPRINTAN™ SLR4602 (Styron Deutschland GmbH) with a styrene content of 21% and vinyl content of 63%. Mooney viscosity ML 1 + 4 (100 °C) is 65 MU. Multi-walled carbon nanotubes (CNTs) Nanocyl™ NC7000 (Nanocyl S.A., Belgium) were used as filler. Nanocyl possesses a broad length distribution with several nanotubes up to 10 μm . The amorphous carbon content of CNTs is about 3 wt% according to the thermogravimetric analysis made in the present work.

The ionic liquid, 1-decyl 3-methyl imidazolium chloride (DMIC), used in our study was received from Sigma–Aldrich, Steinheim, Germany. DMIC/CNT mixtures with desirable mass ratio were prepared by sonication with surplus ethanol for two hours. Afterward, ethanol was completely evaporated in oven at 100 °C to receive dried DMIC/CNT mixtures. The filled SBR compounds were prepared in an internal mixer (Rheocord 300p, ThermoHaake) by keeping the following mixing conditions: initial chamber temperature T_A of 80 °C, rotor speed of 75 rpm, fill factor of 0.78. The amount of the filler and additives used in the study was varied in parts per hundred rubber (phr). The curing package consists of 2.5 phr ZnO, 1 phr stearic acid, 1.4 phr sulfur, and 1.4 phr *n*-cyclohexyl-2-benzothiazole-sulfenamide (CBS). The dried DMIC/CNT mixture was admixed into the chamber at 2 min mixing time. The flowchart of composite preparation and characterization are illustrated in Fig. 1. A CNT/SBR compound was prepared without IL but with ethanol as dispersing agent for comparison purpose. The composition of all the compounds investigated summarized in Table 1.

2.2. Characterization

2.2.1. Optical microscopy

Optical microscopy was used to characterize the CNT macrodispersion. The ratio of the surface of non-dispersed agglomerates to that of the image, A/A_0 , is a measure for the filler macrodispersion.

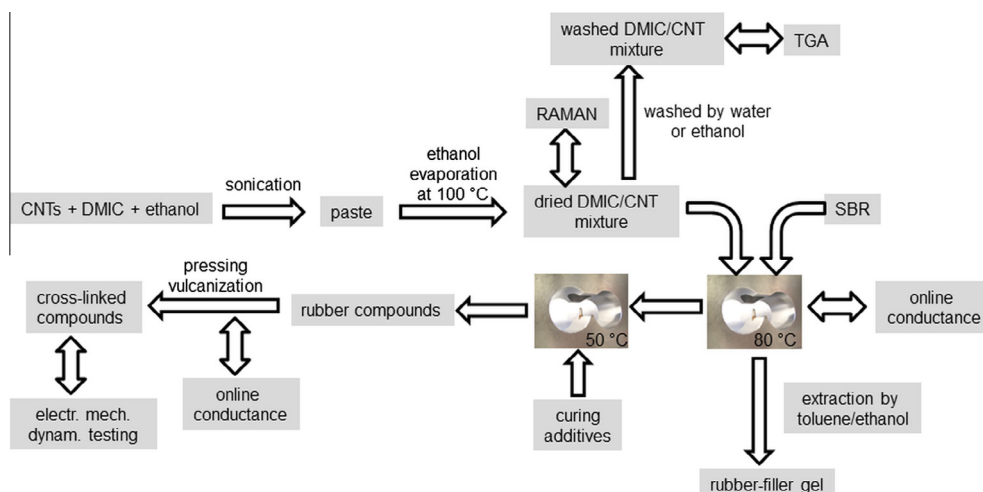


Fig. 1. Schematic flowchart of CNT/SBR composite preparation and characterization.

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