



The effect of silane coupling agent on iron sand for use in magnetorheological elastomers Part 1: Surface chemical modification and characterization



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ARTICLE INFO

Article history:

Received 4 May 2014

Received in revised form 18 September 2014

Accepted 5 October 2014

Available online 12 October 2014

Keywords:

A. Particle-reinforcement

B. Interface/interphase

D. Surface analysis

E. Surface treatments

ABSTRACT

Bis-(3-triethoxysilylpropyl) tetrasulphane (TESPT) was employed for surface modification of iron sand for use in magnetorheological elastomers (MREs). The amount of TESPT was varied at five levels (2, 4, 6, 8 and 10 wt%) relative to iron sand content to assess the optimum amount of coupling agent for interfacial bonding and damping performance. Evidence that coupling had occurred between iron sand and TESPT was identified by Raman Spectroscopy and the grafting percentage was determined by thermogravimetric analysis. Subsequently, isotropic MREs containing unmodified and modified iron sand particles and natural rubber were prepared. Crosslink density assessment by swelling testing provided evidence that the tetrasulphane group of TESPT formed crosslinks with the rubber chains. The results exhibited the advantages of TESPT as a coupling agent between iron sand particles and rubber and also revealed that 6% TESPT content produced the highest crosslink density. The effects of the amount of TESPT on dynamic mechanical properties the morphological characteristics of the MREs were also investigated.

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1. Introduction

Magnetorheological elastomers (MREs) are a new group of damping materials which consist of a non-magnetic matrix (normally an elastomer) containing a suspension of magnetically permeable particles. The most commonly used magnetic particles for MREs are carbonyl iron particles and suitable matrix materials include natural rubber, silicone rubber, polybutadiene, polyisobutylene, polyisoprene, and polyurethane rubber [1–7]. The main advantage of MREs is that the damping and stiffness can be varied by application of an applied magnetic field during fabrication or in service. Damping occurs by the viscous flow of the rubber matrix and inclusion of magnetic particles in rubber enables additional damping through magnetic particle interaction and interfacial damping. MREs can be classified into two kinds: isotropic MREs and anisotropic MREs. Isotropic MREs can be characterized by having a uniform magnetic particle distribution in the matrix. Anisotropic MREs have a special chain-like structure of magnetic particles in a matrix resulting from curing the matrix under an applied magnetic field. Over the past few years, MREs have attracted increasing attention and have been considered for

applications such as adaptive tuned vibration absorbers [8], automotive engine mounts [4] and semi active seismic dampers [9].

New Zealand iron sand possesses physical and magnetic properties that make it suitable for use in MREs. Compared to commonly use magnetic particles, such as pure iron and carbonyl iron, iron sand has a number of advantages, including high permeability and saturation magnetisation, low cost and it is readily available in New Zealand. It is derived from erosion of andesitic and rhyolitic volcanic rocks which are the main types of iron ore deposits in New Zealand. Iron sand is a dark, high-density sand that occurs along the west coast of the North Island from Wanganui to Kaipara Harbour near Auckland, over a distance of 480 km. It contains titanomagnetite, a mineral containing iron and titanium, which is highly magnetic [19,20].

Similarly for all MREs, the damping of MREs depends not only on the types of rubber matrix and magnetic particles, but also on the level of adhesion between the particles and the rubber matrix such that the strength of interaction between the particles and matrix has to be sufficiently strong to obtain efficient interfacial damping. For MREs this sets a challenge. Here, as for other MRE components, the incompatibility of the inorganic magnetic fillers and the matrix can actually lead to poor wettability and adhesion between the filler and matrix as well as non-uniformity of filler dispersion leading to low energy absorption [10–12]. Therefore,

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it is sensible to modify the surface of the iron sand in order to improve ease of adhesion with and dispersion within the rubber matrix in order to realise the full potential of MREs.

Surface modification of inorganic particles can be achieved using a number of approaches [13]; most commonly, the filler surface is chemically modified to become more compatible with the matrix using methods such as polymer coating [14], surfactant absorption [15] and bifunctional coupling agent treatments [16,17]. Among various modification approaches, the bifunctional coupling agent treatment using silane based coupling agents is the most successful and cost effective treatment for improving the adhesion between inorganic particle and rubber matrix. These chemicals are silicon-based chemicals that contain hydrolysable groups (such as methoxy, ethoxy or acetoxy) at one end that will interact with inorganic materials and organofunctional groups (such as amino, vinyl or sulphide) at the other end that can react with the rubber matrix. Therefore, inorganic and organic materials can be coupled together with the silane coupling agent acting as a bridge between them. Although this type of surface modification is well established for enabling different types of fillers such as silica [18,19], aluminium powder [20], halloysite nanotubes [21], wood flour [22] and natural fibre [23,24] to be used as reinforcement fillers in rubber compounds, its effect on inorganic magnetic particles for use in MREs has not been extensively studied. Some research has been reported on modification of carbonyl iron particles using silane coupling agents used as a filler in silicon rubber [25,26] and polyurathene rubber [16] based MREs; the results showed that the mechanical and damping performance of MREs increased due to increased dispersion and interaction between particles and the matrix. Although it is accepted that the effectiveness of silane to interact with the substrate is dependent on a number of factors including hydrolysis time, presence of solvent, temperature and pH [27], none have assessed the optimum silane content coupled to the particles to allow for improved subsequent bonding. Furthermore, as far as the authors are aware, there is no work carried out on surface modification of inorganic magnetic particles for use in natural rubber based MREs.

In this study, isotropic MREs based on iron sand and natural rubber were prepared. Bis-(3-triethoxysilylpropyl) tetrasulphane (known as TESPT or Si 69) which is the most popular and effective silane coupling agent for hydrocarbon rubber (see Fig. 1) was used for surface modification of iron sand particles [13]. TESPT contains ethoxy hydrolysable groups at both ends that enable the silicon groups to bond with iron sand particles and the tetrasulphane group of its centre which is capable of bonding with the rubber matrix. Iron sand contains stable oxides that potentially have reactive sites including Fe–O bonds and OH groups on their surface [28,29]. Therefore, there is potential for improvement of iron sand and natural rubber interaction by using TESPT as a coupling agent. The effects of TESPT content on dynamic mechanical properties were investigated using two methods. The loss tangent, commonly called $\tan \delta$, is considered as the fundamental parameter to assess damping. $\tan \delta$ gives a comparison of the energy lost to that stored; it is obtained by dividing the loss modulus (G'' or E'') by the storage modulus (G' or E') [30]. The other estimate of damping used was the amount of energy dissipated during cyclic deformation, which

can be calculated from the area of the hysteresis loop (hysteresis loss).

2. Experimental

2.1. Materials

Natural rubber (SMR L grade) and other chemicals including zinc oxide, stearic acid, *n*-cyclohexyl-2-benzothiazole sulfenamide (CBS), tetramethylthiuram disulphide (TMTD), paraffin oil, and naphthenic oil were all purchased from Field Rubber Limited, Auckland. Bis-(3-triethoxysilylpropyl) tetrasulphane (TESPT) was purchased from Leap LabChem Co., Limited, China. Iron sand was collected from Ngarunui Beach, Raglan. The iron sand was milled using a planetary mono mill (Pulverisette 6) produced by Fristech GmbH and subsequently sieved to obtain a 45–56 μm particle size.

2.2. Surface modification of iron sand particles

The surface modification of iron sand particles was carried out by an aqueous alcohol solution method. The particles were subjected to surface treatment with TESPT at 2%, 4%, 6%, 8%, 10% by weight (wt%) of the particles. An aqueous alcohol solution of 95% ethanol was used and the pH of the solution was adjusted with acetic acid to 4.0–4.5. The TESPT of predetermined quantity was dispersed in the ethanol solution at a ratio of 1:100 and the mixed solution was stirred for 5 min to assure the hydrolyzation of the silane coupling agent. The iron sand particles were then added and stirred for an additional 30 min at room temperature to ensure a uniform distribution of the coupling agent on the surface of iron sand particles. The mixture was filtered and washed three times with ethanol to remove unreacted coupling agent. The treated iron sand particles were then dried at 80 °C in an oven until a constant weight was achieved.

2.3. Preparation of iron sand-natural rubber isotropic MREs

The compound formulation used in this study is given in Table 1. Formulations were compounded using a conventional laboratory two roll mill (model XK150) according to ASTM designation D3184-80. The front roller speed was 23.86 rpm and the rear roller speed was 32.81 rpm, diameter of rolls was 150 mm, friction ratio of two rolls was 1:1.4 and the roller temperature was set at 80 °C. The nip gap (distance between front and back roller) was maintained at 2 mm during compounding. The compounding began with softening the rubber on its own in the two roll mill (mastication). Mastication reduces the viscosity and increase the plasticity of natural rubber which is brought about by heat generated in two roll mill through conduction from the heated roller and shearing of rubber during milling. After mastication, during which the rubber had become invested on the hot roll (2–3 min), additives (other than accelerators and sulphur) were then added followed by iron sand; addition of accelerators and sulphur were delayed to the last part of the process to prevent premature vulcanization during compounding. The mixing time was approximately 40 min. The cure time at 150 °C was then determined according to the procedure as described in reference [31] and the results are shown in Table 1. Compounded rubber samples weighing 13 g were placed in a mould 60 × 50 × 3 mm and were cured in a compression moulder at 150 °C under a pressure of approximately 12 MPa.

2.4. Raman Spectroscopy

Raman spectra were acquired with a Ramanstation 400R (PerkinElmer) spectrometer equipped with an air cooled charged

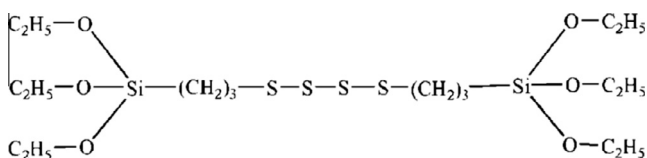


Fig. 1. Molecular structure of TESPT.

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