



Preparation and properties of styrene butadiene rubber/natural rubber/organo-bentonite nanocomposites prepared from latex dispersions

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ABSTRACT

Styrene butadiene rubber (SBR)/natural rubber (NR)/organo-bentonite nanocomposites were prepared from emulsions. The structure of the nanocomposites was characterized by TEM and XRD. The effect of organo-bentonite on the mechanical properties, thermal stability and swelling behavior was investigated. TEM and XRD confirmed that rubber chains were intercalated into the organo-montmorillonite particles. When the organo-bentonite content was lower than 12 mass%, the nanocomposites showed excellent mechanical properties. The addition of a small amount of OMMT greatly improved thermal stability and swelling behavior, which was attributed to the good barrier properties of the dispersed and partially exfoliated organo-montmorillonite particles. Organo-bentonite addition had little effect on the score and cure times of the nanocomposites, while increased the maximum and minimum torque.

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1. Introduction

Polymer/clay nanocomposites are of great interest for both scientific challenges and industrial applications (Kojima et al., 1993; Messersmith and Giannelis, 1995; Giannelis, 1996). Polymer/clay nanocomposites with a relatively low loading of the filler have superior mechanical properties, thermal stability, flame retardancy and gas barrier properties (Gao et al., 2008; Alexander et al., 2002; Nah et al., 2002; Gu et al., 2009). These improvements in the properties are the result of the nanometer scale dispersion of clay minerals in the polymer matrix. The polymer/clay nanocomposites are generally classified into three groups according to their structure, i.e., nanocomposites with intercalated, exfoliated or both of intercalated and exfoliated structure. Nanocomposites with completely exfoliated clay mineral particles are desired as the exfoliated particles exhibit the greatest reinforcement (Wu et al., 2005). To make rubber compatible with polymers and obtain a high degree of dispersion, the clay minerals are modified with organic compounds.

Natural and synthetic rubbers are not used very often in an isolated form. For practical application, several agents are incorporated, the choice of which is based on the set of properties required for that particular use. An elastomer is mixed with another one for three main reasons: to improve properties of the original material, to improve processibility, or to lower costs (Visconte et al., 2004). The

most common technique of preparing polymer mixtures is mechanical mixing. The complete miscibility of polymers requires that the free energy of the mixing be negative, which implies an exothermic mixing or large entropy of mixing (Hess et al., 1993). Therefore, most blends of rubbers are immiscible due to the differences in solubility of the polymers, which leads to nonuniform dispersion of the components in the rubber phase.

Polymer/clay nanocomposites can be prepared by many methods, including in situ polymerization accompanied by intercalation, intercalation from solution or melts or even using emulsions. Compared to other methods, emulsion intercalation is a promising method for industrialization due to the simplicity of preparations, low cost and superior performance (Wu et al., 2005). The SBR/NR composite is widely used in tyre industry. Blending SBR with NR can improve the tensile strength because NR crystallizes on stretching. NR and SBR can be obtained in the form of latex dispersions which do not flocculate when mixed together. In this study, SBR/NR/OMMT nanocomposites in a 7:3 (m/m) ratio were prepared by emulsion intercalation.

2. Experimental

2.1. Materials

Bentonite was obtained from the clay mine in Shandong Province of China. It had a cation exchange capacity (CEC) of 1.19 meq/g. The SBR latex (23 mass%) was obtained by Qilu Petrochemical Company (China) and the NR latex (60 mass%) was purchased from Double Butterfly Group (China). Other materials are commercial products (AR grade).

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2.1.1. Preparation of organo-bentonite

The bentonite was dispersed in water and purified by sedimentation and washing. The dried bentonite (50 g) was dispersed into 5000 mL of distilled water at 65 °C under vigorous stirring. A solution of 40 g dimethyl ditallow-ammonium chloride, 200 g distilled water and 6 mL concentrated sulfuric acid, prepared at 65 °C was slowly added to the bentonite dispersion. The reaction was maintained for 4 h. The sediment was collected by filtration and washed ten times with hot water, dried at 90 °C in vacuum, ground and sieved to 30 μm .

2.2. Preparation of the nanocomposites

The aqueous suspension of organo-bentonite in water (organo-bentonite content 10 mass%) was added to the mixture of SBR and NR latex in 7:3 (m/m). The mixture was vigorously stirred for 20 min at 60 °C. The amounts of organo-bentonite added were 0, 3, 6, 9, 12 and 15 mass% of the mass of rubber. The mixture was coagulated with acetic acid, washed with water and dried at 60 °C. The obtained nanocomposites were mixed on a two-roll mill with other additives at ambient temperature for about 15 min in the order ZnO, SA, DM, CZ and sulfur. Finally, the compounds were compression-molded at 143 °C under 15 MPa for the optimum cure time (t_{90}).

Recipes: SBR/NR 100 phr, ZnO 5 phr, SA 1 phr, DM 0.5 phr, CZ 1.5 phr, sulfur 1.5 phr; phr in mass per 100 g of rubber.

2.3. Characterization

2.3.1. Transmission electron microscope

TEM observation was performed on ultra-thin films prepared by cryoultramicrotomy using a JEM-1200EX (JOEL, Japan) at an acceleration voltage of 80 kV.

2.3.2. X-ray diffraction pattern

XRD experiments were performed with a D/max-RB diffractometer (Rigaku, Japan), graphite monochromator, Cu Ka radiation. The samples were scanned with 5°/min between 2θ of 3°–25°.

2.3.3. Mechanical properties

Tensile and tear tests were measured on a DXLL-50000 (Dirs Company, China) universal testing machine at a crosshead speed of 500 mm/min. Measurements of mechanical properties were con-

ducted at 25 ± 2 °C according to ISO standards (ISO 37, and ISO 7619). The reported values are the average of five measurements.

2.3.4. TGA analysis

Thermo-gravimetric analysis (TGA) of the samples was performed using a thermo-gravimetric analyzer (TGA/STDA851, Mettler Toledo, Shanghai, China) attached to an automatic programmer from ambient temperature to 700 °C at a heating rate of 10 °C/min in an air atmosphere. Approximately 15 mg samples were used. The temperature reproducibility of the TGA instrument was ± 3 °C.

2.3.5. Swelling behavior

Samples of $20 \times 10 \times 2$ mm³ were used to determine by gravimetry the swelling behavior of vulcanized rubber according to ISO1817-198 in ASTM 1# oil. The samples were periodically removed from the test bottles, the adhering solvent was cleaned from the surface, and the samples were weighed immediately. The values are the average of two parallel samples.

2.3.6. Curing characteristics

The curing behavior was investigated at 143 °C using a rotorless rheometer MDR2000 (D and G Company, China) according to ISO 6502-1999 with a frequency of 1.66 Hz.

3. Results and discussion

3.1. Morphology and structure

The black lines and area in the TEM patterns (Fig. 1) are organo-montmorillonite layers, white region indicates the rubber matrix. Layers are irregularly dispersed. The interface between the matrix and the organo-montmorillonite layers is a little blurry, and the aggregates of layers are in a contorted condition, which indicates that there is a strong interfacial force between rubber and organo-montmorillonite (Liao et al., 2006). The thickness of most aggregates is 20–30 nm and the length about 100–250 nm. The high magnification TEM photograph reveals that the rubber chains are intercalated into the organo-montmorillonite and some particles are exfoliated into single layers.

The bentonite showed a basal spacing of the montmorillonite of 1.55 nm (Fig. 2), the organo-montmorillonite a spacing of 1.92 nm. The reflection (001) of the nanocomposite with 3 mass% organo-

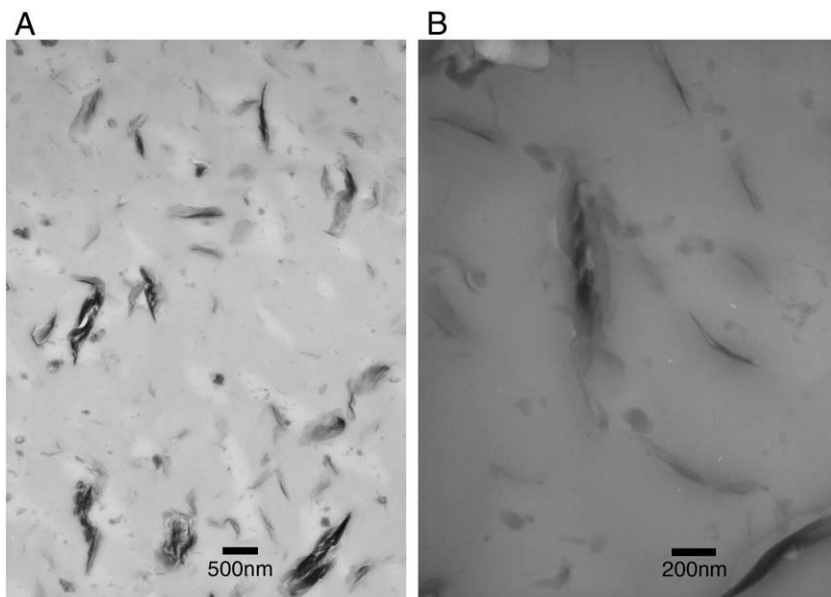


Fig. 1. TEM patterns of SBR/NR nanocomposites with 3 mass% organo-bentonite at two magnifications.

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