



Asphalt modified by thermoplastic elastomer based on recycled rubber



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HIGHLIGHTS

- Asphalt modified by TPE based on recycled rubber has been discussed.
- Dynamic devulcanization and dynamic vulcanization improve the dispersion of GTR in the asphalt.
- Asphalt modified by TPE has good processing property and high storage stability.

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ABSTRACT

Thermoplastic elastomers (TPEs) based on ground tire rubbers (GTRs) and polyethylene were used for asphalt modification. TPEs were prepared by dynamic devulcanization (DD) in the presence of desulfurizer and dynamic vulcanization (DV) with sulfur, respectively. Structure and properties of TPEs and their modified asphalts were characterized by morphology, thermal, sol–gel content and mechanical testing. It was found that DD process decreased the physical properties, while the DV process strengthened that of TPE. The results also showed that the compatibility between PE and GTR was improved by DV, which further enhanced physical property and stabilized the TPE in the asphalt.

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1. Introduction

Recycling waste rubbers as asphalt modifiers has been considered as the most potential way from both economic and environmental points. However, the insoluble properties of recycled waste rubbers hindered their wide usage. The chemical crosslink structure of the recycled waste rubbers makes them act as elastic fillers, which causes the high initial cost and uncertain properties of asphalt modified by the rubbers. The uncertain properties arise from the degradation and settlement of ground tire rubber (GTR) during processing GTR modified asphalt.

Thermoplastic elastomers (TPEs) are a class of copolymers or a physical mixture of polymers (usually a plastic and a rubber) which consist of materials with both thermoplastic and elastomeric properties. TPEs have attracts more attentions in industry since they combine the properties of vulcanized rubber with the ease processing of thermoplastics [1–6]. Styrene–butadiene–styrene copolymer (SBS) is a typical synthesized copolymer and the largest TPE being used as asphalt modifier because of its good

compatibility with asphalt, workability and high performance. However, the poor oxidation resistance caused by the presence of double bonds in backbone of SBS and high cost are the main challenges before selecting it as asphalt modifier [7–11]. Therefore, more competitive TPEs are deserved to develop for asphalt modification.

TPEs based on recycled tire rubbers have been widely investigated for the purpose of improving the performance and recycling tire rubbers. Karger-Kocsis et al. [12] have given a thorough review on processing GTR into TPEs. The biggest challenge of using GTR in TPEs is how to obtain favorable properties compared to those traditional TPEs. The use of GTR component leads to the deterioration of the mechanical properties of TPE because of the poor compatibility between the components. Devulcanization, surface activation, compatibilizer and dynamic vulcanization are considered as useful tools to improve the properties of GTR based composites [12–23]. Maleic anhydride and irradiation approach are also used to improve compatibility [12]. Magioli reported using dynamic vulcanization technology to enhance the properties of TPE [1]. Frequently, GTR was reclaimed before the process of dynamic vulcanization to further improve the homogeneous structure and physical properties of TPE.

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The main purpose of above prepared TPEs based on GTR is to improve the physical properties. However, as far as we know, the TPEs based on GTR have not been widely used because of their low properties and odor. Therefore, it is quite necessary to try to use these TPEs as asphalt modifiers for the purpose of ease processing and balanced properties for asphalt pavement.

The objective of this paper is to investigate the basic structure and properties of asphalt modified by TPE based on GTR. Dynamic devulcanization (DD) is firstly applied when GTR and LLDPE are blended in a twin-screw extruder. In this process, both thermo-shearing force and chemical desulfurizer (aryl disulfides) are used to partially de-vulcanize GTR and decrease the particle size of GTR. The blends are further dynamically vulcanized to improve the properties of TPE and its effect on the modified asphalt.

2. Experimental

2.1. Materials

GTR (30 mesh) was obtained by grinding a whole waste truck tire. Linear low density polyethylene (LLDPE) with the melting index of 2.0 g/10 min (load of 2.16 kg, at 190 °C) was used. Desulfurizer was aryl disulfides. Processing oil was the engine oil with a viscosity of 46 centi-Poise (25 °C). The basic properties of asphalt were listed as following: softening point was 45.3 °C, penetration at 25 °C was 70.1 (0.1 mm).

2.2. Preparation of TPEs and their modified asphalts

2.2.1. Preparation of TPEs

Dynamic devulcanization (DD) process: GTR was mixed with linear low density polyethylene (LLDPE) and devulcanized in a twin-screw extruder (L41/D1, ZE 25A). Blends were passed through the extruder at the screw speed of 200 rpm and 180 °C.

Dynamic vulcanization (DV) process: The resulting DD blends were dynamically vulcanized on a two-roll mill at 150 °C for 5 min. Sulfur vulcanization system (S 1.5, ZnO 1.5, Stearic acid 0.3, and DM 0.5 phr) was used as well (see Table 1).

2.2.2. Preparation of TPEs modified asphalts

Asphalt was heated till fluid in an iron container. Then upon reaching at about 180 °C, TPEs were added to the asphalts. The modified asphalts were blended by using a high shear mixer at 180 °C with a shearing speed of 4000 rpm for 30 min.

2.3. Characterization

2.3.1. Mechanical measurements

Tensile and tear measurements were performed on a universal testing machine at room temperature with a crosshead speed of 500 mm/min, according to ASTM D412 and D624 respectively. Sheets of TPE were prepared by compression molding. The

dumbbell-shaped and un-nicked (90 °C) specimens were punched out.

2.3.2. Conventional properties and storage stability of the modified asphalts

The conventional properties of modified asphalt like penetration, softening point and ductility were measured according to ASTM D5-06, D36-06, and D113-07, respectively. The high-temperature storage stability was evaluated through the following procedure. The samples of prepared blends were transferred into an aluminum toothpaste tubes (25.4 mm in diameter, 140 mm in height). The tubes were sealed and stored vertically in an oven at 163 °C for 48 h. After that, the samples were cooled down to room temperature. The aluminum tubes were then cut horizontally into three equal parts. The storage stability of the TPEs modified asphalt was evaluated by measuring the difference in softening points (ΔSP) between the top and bottom sections. If the value is less than 3.5 °C, the blend was considered as stable.

2.3.3. Structure analysis

Sol-gel analysis was carried out by Soxhlet extraction of the samples with toluene at 110 °C. The samples were extracted for 24 h at atmospheric pressure. After that, the toluene was removed from the samples in an oven at 60 °C for 12 h. The gel fraction was calculated as

$$\text{Gel-fraction (\%)} = W_1/W_0 \times 100\% \quad (1)$$

where W_0 is the original weight of the sample before extraction, and W_1 is the weight of dried sample after extraction.

Swelling behavior was determined by immersing 0.1 g blends in 30 ml toluene for 72 h to reach swelling equilibrium. Every 12 h, the samples were taken out from toluene and the solvent was blotted from the surface of the sample then weighed immediately. Then, the samples were dried out at 60 °C after the constant weight was reached. The swelling ratio is calculated as

$$\text{Swelling ratio (\%)} = (W_s - W)/W \times 100\% \quad (2)$$

where W is the original weight of the sample before swelling, and W_s is the weight of sample containing toluene at the equilibrium.

2.3.4. Thermal analysis

Differential scanning calorimetry (DSC) was performed on a differential scanning calorimeter under nitrogen flow. The tests were conducted as follows: the samples were heated from 40 to 200 °C, then cooled from 200 to –80 °C and reheated to 200 °C. The heating and cooling rate was 20 °C/min. To erase the differences in thermal history, the glass transition temperature was measured in the second and third steps.

Thermo gravimetric analysis (TGA) was carried out using thermo gravimetric analyzer with nitrogen atmosphere. The temperature ranged from 40 to 600 °C at a heating rate 10 °C/min.

Dynamic mechanical analysis (DMA) was implemented with a dynamic mechanical analyzer. The experiments were carried out using the rectangular samples and tension mode over the temperature ranging from –80 to 50 °C, at a heating rate of 3 °C/min. The samples were scanned at a frequency of 1 Hz, and a strain of 0.01% was applied.

2.3.5. Morphology

Scanning electron microscopy (SEM) was conducted by observing the topography of the cryogenic fracture surface of the specimens, using a Scanning Electron Microscope. The samples were sputter coated with a fine layer of gold under vacuum for 60 s before scanning.

Optical microscopy was used to observe the morphologies of the modified asphalts. A small drop of asphalt was placed between

Table 1
Recipes of different TPEs.

Code	GTR (phr)	LLDPE (phr)	Oil (phr)	Desulfurizer (phr)	Sulfur (phr)
DD ₀	70	30	15	0	0
DD ₁	70	30	15	0.3	0
DV ₀	70	30	15	0	1.5
DV ₁	70	30	15	0.3	1.5

The dosage of desulfurizer was selected according to literature [21], phr means part per hundred rate.

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