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# Mechanical and microwave absorbing properties of *in situ* prepared hydrogenated acrylonitrile-butadiene rubber/rare earth acrylate composites

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#### ABSTRACT

Lanthanum oxide (La<sub>2</sub>O<sub>3</sub>), samarium oxide (Sm<sub>2</sub>O<sub>3</sub>) and acrylic acid (AA) were *in situ* reacted in hydrogenated acrylonitrile–butadiene rubber (HNBR) to obtain HNBR/acrylate and HNBR/acrylate/multi-walled carbon nanotubes (MWNTs) composites. Fourier transform infrared spectrum analysis showed the absorption peaks of carbon–carbon double bonds of the acrylates became weak after curing at elevated temperatures, indicating the self-polymerization of acrylates and their participation in crosslinking reactions of HNBR. The morphology of composites was characterized by scanning electron microscopy and Xray diffraction analysis, and the well dispersion of acrylates was attributed to the *in situ* reactions during curing. The tensile strength of HNBR/La<sub>2</sub>O<sub>3</sub>/AA/MWNTs and HNBR/Sm<sub>2</sub>O<sub>3</sub>/AA/MWNTs composites reached 25.2 and 17.6 MPa, and their stress at 100% extension reached 8.4 and 7.4 MPa, indicating the good reinforcement of *in situ* formed acrylates and MWNTs on HNBR. The glass transition temperature of HNBR increased to a limited extent because of the addition of AA and La<sub>2</sub>O<sub>3</sub>. The HNBR/La<sub>2</sub>O<sub>3</sub>/AA/ MWNTs and HNBR/Sm<sub>2</sub>O<sub>3</sub>/AA/MWNTs composites had good microwave absorbing properties, with the maximum reflection loss of -9.0 and -13.5 dB, respectively, and broad absorption bands.

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#### 1. Introduction

Rare earth (RE) compounds are used to improve mechanical, electrical, light and magnetic properties of polymers. Generally, RE compounds are incorporated into polymers after modification. A soluble compound of lanthanum (La) was used to modify the surface of nanocalcium carbonate (nCaCO<sub>3</sub>) particles via chemical deposition method to prepare polypropylene (PP)/nCaCO<sub>3</sub>-La composites with enhanced impact strength and improved filler dispersion [1]. RE modified nCaCO<sub>3</sub>/natural rubber (NR) composites were obtained via chemical deposition and rubber latex/filler coagulation, with good mechanical properties that was attributed to the improved interaction between nCaCO<sub>3</sub> and NR by the addition of RE compounds [2]. RE compounds can improve the dispersion of carbon black [3]. Three rare earth oxides (Sm<sub>2</sub>O<sub>3</sub>, La<sub>2</sub>O<sub>3</sub> and Pr<sub>6</sub>O<sub>11</sub>) modified high-abrasion furnace could impart high mechanical strength to NR [4]. RE compound doped polymethyl methacryalumina-siloxane late-wrapped microcapsule powder/NR vulcanizates with good mechanical properties and good resistance to fatigue [5].

Styrene-butadiene rubber/NR composites had improved mechanical properties through incorporation of RE compounds

[6]. Acrylates can be *in situ* prepared in rubber matrix, self-polymerize, and participate in crosslinking reactions of rubbers, contributing to fine dispersion with small particle size.  $Sm(AA)_3/acrylonitrile-butadiene$  rubber (NBR) composites were prepared using both *in situ* reaction method and direct blending method. Low loading of  $Sm(AA)_3$  (less than 30 wt%) imparted good fluorescent properties to the composites, and their fluorescent intensity depended on the preparation methods [7]. The *in situ* polymerization of  $Gd(AA)_3$  in NR during vulcanization with peroxide greatly improved the dispersion of the shielding phase and improved the X-ray shielding properties of the composites [8].

However, the improvement of RE to electromagnetic characteristics and microwave absorbing properties of rubbers has rarely been reported. RE compounds can be used as dopants or modifiers for carbon nanotubes and ferrite to improve the electromagnetic and microwave absorbing properties of nano-materials. Zhang et al. [9,10] studied the electromagnetic characteristics and microwave absorbing properties of Sm<sub>2</sub>O<sub>3</sub>-filled and Er<sub>2</sub>O<sub>3</sub>-filled multiwalled carbon nanotubes (MWNTs). They found Sm<sub>2</sub>O<sub>3</sub> and Er<sub>2</sub>O<sub>3</sub> enhanced the magnetic loss of MWNTs, increased the microwave absorbing peak intensity, and broadened the absorption frequency bandwidth. In this paper, the acrylates of La and Sm were prepared in hydrogenated acrylonitrile–butadiene rubber (HNBR) via *in situ* reaction method to obtain HNBR/acrylate/MWNTs composites. The







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effect of RE acrylates on the mechanical and microwave absorbing properties of HNBR composites was studied.

#### 2. Experimental

#### 2.1. Materials

HNBR (Therban<sup>®</sup> C 3446) with 34% acrylonitrile content and 4% residual double bonds was kindly provided by Lanxess Deutschland GmbH, with Mooney viscosity  $ML_{1+4}^{100}$  °C of 63. MWNTs (NC7000) was produced by Nanocyl, Belgium, with average diameter of about 9.5 nm, the length higher than 1.5 µm, and the purity higher than 90%. Dicumyl peroxide (DCP) and acrylic acid (AA), analytical grade, were purchased from Sinopharm Group Chemical Reagent Co., Ltd., China. La<sub>2</sub>O<sub>3</sub> and Sm<sub>2</sub>O<sub>3</sub> were purchased from Conghua Jianfeng Rare Earth Co., Ltd., Guangdong, China, with the purity of 99.99%.

#### 2.2. Sample preparation

Composites were prepared in the mixing chamber of a Haake Rheometer at a rotor speed of 32 rpm at the initial temperature of 40 °C. The formation was HNBR 100 phr, rare earth oxide 30 phr, acrylic acid 39.8 phr for  $La_2O_3$  or 37.2 phr for  $Sm_2O_3$ . The rare earth oxide/acrylic acid ratio followed the stoicheiometry ratio of 1/6 (mol/mol). HNBR was first mixed in the mixing chamber for 1 min, followed by the addition of rare earth oxide, mixed for 3 min, and then acrylic acid was added and kept mixing for

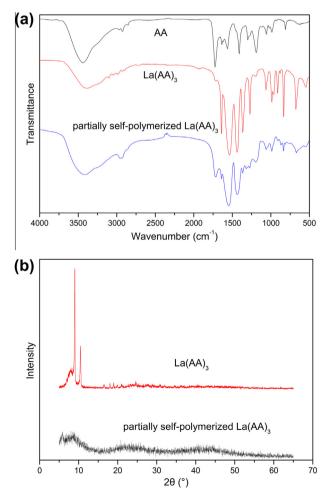


Fig. 1. FTIR spectra (a) and XRD patterns (b) of La(AA)<sub>3</sub>.

8 min. 5 phr of MWNTs and 3 phr of DCP were added to the compounds on a two-roll mill. The compounds obtained were cured under pressure at 170 °C for 20 min for mechanical, dynamic mechanical and microwave absorbing testing.  $La(AA)_3$  was prepared as Ref. [8].

#### 2.3. Characterization

Scanning electron microscopy (SEM) images of cryogenically fractured surface of HNBR composites were taken on a JSM-

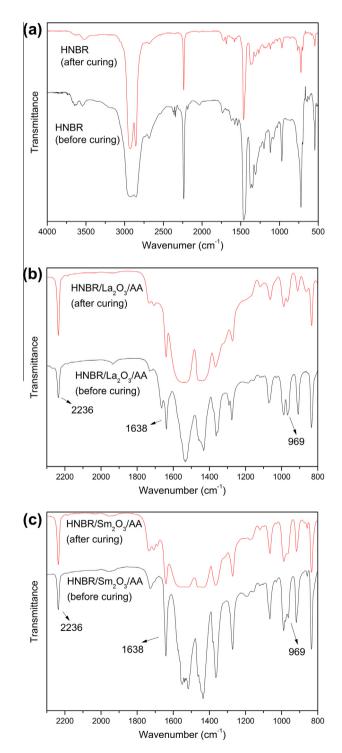


Fig. 2. FTIR spectra of HNBR (a), HNBR/La $_2O_3/AA$  (b) and HNBR/Sm $_2O_3/AA$  (c) composites.

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