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Renewable Interfacial Modifier for Silica Filled Natural Rubber Compound

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Abstract

A main problem of using silica as reinforcing filler in natural rubber (NR) compound is a poor dispersion of silica in the rubber matrix due to incompatibility between them. As silica contains high silanol groups on its surface, these polar groups promote strong filler-filler interactions leading to high degree of aggregation and agglomeration of silica particles. To overcome this drawback, many types of modifiers have been applied into silica filled recipes. In this work, a renewable and sustainable interfacial modifier, namely low molecular weight hydroxyl terminated natural rubber (HTNR). HTNR was prepared by controlled epoxidation and cleavage of natural rubber, following by reduction reaction of the obtained carbonyl telechelic natural rubber. Influence of the prepared interfacial modifier on enhancement of rubber-filler interaction was studied by focusing on the cure characteristics and the mechanical properties of silica filled natural rubber at a fixed silica content of 30 phr. In comparison with silica filled compound without silane coupling agent, shorter scorch and cure times, lower Payne effect and higher tensile strength of silica filled natural rubber were achieved with the presence of interfacial modifier at 2.4 parts per hundred parts of rubber (phr). This was attributed to an active reaction of silanol groups with interfacial modifier and compatibility between modifier and natural rubber. For HTNR-treated compound to obtain optimum properties, mixing should be carried out at initial mixing temperature of 30^oC, mixing time of 12 minutes, and rotor speed of 60 rpm. However, TESPT is more effective than HTNR because it imposes better properties than HTNR.

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Nomenclature

2000HTNR	Hydroxyl telechelic natural rubber with molecular weight of 2000 g/mol
T30t12	Mixing temperature at 30°C and mixing time of 12 min

1. Introduction

Natural rubber (NR) is a material widely used in various industries because of its excellent properties such as elasticity, low hysteresis, high resilience, toughness, and *etc.*¹. Normally, NR is an amorphous material, however, when stretched it can crystallize. The strain induced crystallization contributes to good mechanical properties of NR such as tensile strength, tear resistance and abrasion resistance, etc. NR is highly elastic. It can recover almost completely to its original shape when the applied load is removed. Moreover, NR has a very good dynamic mechanical properties¹ and excellent tackiness. In general, rubber products are not made from pure rubbers. Many ingredients such as curing agents, fillers, antidegradants, processing aids etc., are added into rubbers to improve their processing characteristics and mechanical properties². Modulus and strength of rubbers are greatly improved by incorporating reinforcing fillers such as carbon black and silica. Carbon black has been used as reinforcing material in tire industries for long times whereas the use of silica is only limited to coloured rubber products. In recent years, silica has been introduced to tire industries due to low rolling resistance and excellent wet traction and ice grip. The presence of silanol groups on silica surface induces strong particle–particle interaction, giving rise to a high tendency, for filler agglomeration, and low compatibility with non-polar rubbers, which leads to poor dispersion and processability. The structure of silica aggregates in rubbers can be tailored by modifying the filler surface in order to increase the compatibility of silica with hydrocarbon rubbers and to improve the filler dispersion, compound processability and various vulcanizate properties². Surface modification of silica can be carried out by many methods such as admicellar polymerization^{3,4}, core-shell polymerization⁵, plasma polymerization⁶⁻⁸, silane treatment⁹⁻¹¹, and etc. However, silane treatment is more favourable method, TESPT is the most important silane coupling agent for silica reinforced non-polar rubber. Nevertheless, the use of TESPT in silica filled rubbers always encounters problems such as scorching of rubber compounds during the mixing stage and the release of ethanol during vulcanization etc¹⁰. In this study, hydroxyl telechelic natural rubber (HTNR) is used to improve interaction between NR and silica. Moreover, the mixing condition of silica reinforced NR with HTNR is studied in order to determine the optimum mixing condition for desired mechanical properties.

2. Experiment**2.1. Materials**

High ammonia concentrated latex (60%DRC) was purchased from Yala latex industry Co.,LTD, Yala Thailand), Hydrogen peroxide and formic acid were supplied by Acros Organics, Belgium. Periodic acid is a product of Merck, Germany. Sodium borohydride (NaBH₄) was purchased from LobaChemie Pvt. Ltd. All chemicals and solvents for HTNR preparation are of analytical grade. STR 5L was obtained from Yala latex industry Co.,LTD, Yala Thailand. ULTRASIL VN 3, precipitated silica, and bis-(3-triethoxysilylpropyl) tetrasulfide silane (TESPT) were obtained from Evonic, Germany. 2,2,4-Trimethyl-1,2-dihydroquinoline polymer, (TMQ), diphenylguanidine (DPG), N-tert-Butyl-2-benzothiazole sulfonamide (TBBS) are products of Flexsys America L.P., U.S.A. Zinc oxide (ZnO) stearic acid, and sulphur were supplied by Petchthai Chemicals.

2.2. Preparation and structural characterization of HTNR.**2.2.1. Preparation of epoxidized natural rubber (ENR).**

Preparation of ENR was started with diluting concentrated NR latex (60% dried rubber content, DRC) to 20%DRC and preserving this latex with 3.0 phr of 10% non-ionic stabilizer (Teric N30) solution along with mechanical stirring at rotor speed of 75 rpm for 16 hrs. Then, 90% formic acid was introduced drop wise into the

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