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Mechanical properties and stability towards heat and UV irradiation of natural rubber/nanotitanium dioxide composites

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Abstract

Reactive double bonds on the main chains of natural rubber (NR) lead to a degradation by weather, heat and ultraviolet (UV). Nano-titanium dioxide (n-TiO₂) should be efficient for long term heat and UV protection for rubber compounds. This work desizes a commercial micron size-TiO₂ to n-TiO₂ by using an ultrasonic processor. The unsonicated TiO₂ with a median particle size (d₅₀) of about 1 μm is desized to 123 nm after sonication. The obtained n-TiO₂ is then used in NR compounds at varying loadings in the range of 0-20 parts per hundred parts of rubber (phr). An increase of n-TiO₂ content enhances the tensile modulus and strength of the vulcanizates. The 100% modulus, tensile strength and elongation at break show no significantly changes after heat aging at 70°C and UV irradiation at 50°C, but the heat aging at 100°C drastically reduces the tensile properties. The UV-irradiated NR vulcanizates show only small changes in tensile properties, but displays a significant change in the molecular structures at the surface, as shown in their ATR-FTIR spectra. Thermal stability of the vulcanizates, as analyzed by thermogravimetric analysis (TGA), shows that the decomposition temperatures are increased whereas the rates of weight change are decreased with increasing the TiO₂ loadings. The SEM micrographs indicate that the n-TiO₂ aggregates are uniformly dispersed in the rubber matrix.

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1. Introduction

Natural rubber (NR) possesses good mechanical properties, elasticity and dynamic behavior but also has some drawbacks caused by its reactive double bonds that lead to degradation by weather, heat and ultraviolet (UV). The degradation leads to a decrease of mechanical properties and an occurrence of surface cracks as well as discoloration¹. In general service conditions, photo- and thermal oxidations are the main causes for polymer degradation. The process starts *via* an initiation step that finally leads to an auto-oxidation cycle². Polymer degradation results in the decrease of mechanical properties such as modulus, hardness and tensile strength with either increasing or decreasing ultimate elongation as generally observed in unsaturated rubbers, e.g. NR, synthetic polyisoprene rubber (IR) and isobutylene-isoprene rubber (IIR)³. To extend a service life of rubber products for outdoor application, antioxidant, antiozonant and UV absorber are normally added into a rubber compounds. Use of nano-titanium dioxide (n-TiO₂) should be efficient for long term heat and UV protection for rubber compounds as it does not decompose and migrate during the service. The addition of n-TiO₂ into the polymer matrix increases mechanical properties and degradation temperature of the composites⁴⁻⁶. However, the nanoparticles easily agglomerate due to their large specific surface area and also a direct incorporation of n-TiO₂ into the polymers faces incompatibility problem between the two components. Chemical interactions between n-TiO₂ and polymer can be enhanced by use of coupling agent leading to the improved filler dispersion and mechanical properties^{7,8}.

Based on our preliminary study on comparing the properties of the rubber vulcanizates filled with n-TiO₂ and micron size-TiO₂, it was found that n-TiO₂ could be well dispersed in the rubber matrix and gave better mechanical properties compared to the use of micron sized TiO₂. Therefore, this work employed n-TiO₂ for further study. A commercial micron size-TiO₂ was desized to n-TiO₂ by using an ultrasonic processor at a frequency of 20 kHz. The n-TiO₂ suspension was later mixed with NR latex to prepare NR masterbatch at varying loadings in the range of 0-20 parts per hundred parts of rubber (phr). The dried masterbatches were mixed with other additives to obtain final NR compounds. The influence of n-TiO₂ on the mechanical properties, aging and UV resistance and heat stability of the NR vulcanizates were investigated.

2. Experimental

Materials: High ammonia (HA) concentrated NR latex with 60% of dry rubber content (DRC) was produced from Pan Asia Biotechnology, Thailand. Commercial TiO₂ (anatase grade) was manufactured by Metal Chem, Thailand. Isopropyl trioleyl titanate (ITT) was manufactured by Nanjing Chemical, China. ZnO, stearic acid, N-tert-butyl-2-benzothiazole sulfenamide (TBBS) and sulfur were of commercial grades for rubber industry.

Preparation of TiO₂ nanoparticles: Ultrasonication was carried out by using an ultrasonic processor (UIP1000hd, Hielscher, Germany) with a power of 1000 W and ultrasonic frequency of 20 kHz. The sonotrode was immersed in 10 wt% of commercial micron size TiO₂ suspended in freshly deionized water, and then the suspension was sonicated for 30 min. The particle sizes of TiO₂ suspension, as measured by laser light scattering particle size analyzer (Horiba LA-950, USA), are summarized in Table 1.

Table 1 Particle size of TiO₂ suspension.

TiO ₂ types	Particle size (nm)		
	d10*	d50*	d90*
Before ultrasonication	466	1111	3089
After ultrasonication	73	123	627

Remark: * d10, d50 and d90 are defined as the diameter where 10%, 50% and 90% of the population lies below, respectively.

The TiO₂ suspensions show a bimodal particle size distribution of primary particles and agglomerates/ aggregates, as shown and discussed in our previous work⁹.

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