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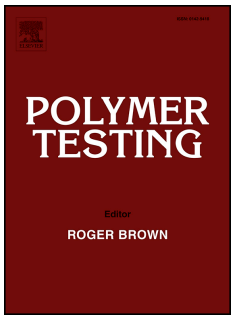

Synthesis and structural studies of an epoxidized natural rubber/titania (ENR-50/TiO_2) hybrid under mild acid conditions

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ACCEPTED MANUSCRIPT

Material Characterisation

Synthesis and Structural Studies of an Epoxidized Natural Rubber/Titania (ENR-50/TiO_2) Hybrid Under Mild Acid Conditions

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Abstract. The ring-opening reaction of epoxide units in an epoxidized natural rubber/titania (ENR-50/TiO_2) hybrid in the presence of acetic acid was studied using NMR techniques as well as FTIR spectroscopy. The thermal characteristics of the hybrid were evaluated using TG/DTG and DSC analyses. ^{1}H NMR results suggest that 86.51% of the epoxide rings had opened in the hybrid, and the ^{13}C NMR results revealed new peaks from the hybrid at δ 22.3, 34.8 and 72.2 ppm. 2D NMR (HMQC, HMBC and COSY) techniques further confirmed these assignments. FTIR spectra showed a characteristic Ti-O-C peak at 1028 cm^{-1}. The TG/DTG results showed four decomposition steps at 30-159, 229-325, 325-466 and 664-825°C due to the presence of Ti moieties along with the mixture of polymer chains *i.e.*, the ring-opened and intact epoxides of ENR-50, which also led to an increase in the T_g value of the hybrid (27.8°C) compared with purified ENR-50 (-17.7°C).

Keywords: epoxidized natural rubber, titania, ring-opening reaction, hybrid, NMR.

1. Introduction

In general, epoxidized natural rubber (ENR)-based hybrids can be divided into two main groups: ENR-based polymer hybrids or ENR-based inorganic hybrids. The formation of hybrids is classified according to the formation of covalent bonds between these moieties. Typically, both natural and synthetic polymers are hybridized with ENR *via* grafting, crosslinking or forming covalent bonds. Typically, ENR-50 is hybridized with other polymers such as bisphenol A epoxy [1], chitosan [2], ethylene-propylene-diene rubber (EPDM) [3], nitrile butadiene (NBR) [4], styrene butadiene

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