Rubber Science, **30**(3): 278-297, 2017 RESEARCH ARTICLE

STABLE FREE RADICAL ASSISTED MECHANICAL DEVULCANISATION OF CARBON BLACK FILLED NR VULCANISATES: DEVULCANISATION OF UN-AGED AND AGED SAMPLES

Anu Mary Joseph, K.N. Madhusoodanan, Rosamma Alex and Benny George

Rubber Research Institute of India, Rubber Board, Kottayam, Kerala-686 009, India

Received: 09 October 2017 Accepted: 13 November 2017

Joseph, A.M., Madhusoodanan, K.N., Alex, R. and George, B. (2017). Stable free radical assisted mechanical devulcanisation of carbon black filled NR vulcanisates: Devulcanisation of un-aged and aged samples. *Rubber Science*, **30**(3): 278-296.

Devulcanisation of filled rubber vulcanisates through mechanical/ mechano-chemical processes in a two-roll mill is a well-established technique. The general mechanism of mechano-chemical devulcanisation suggests blocking of possible reunion of free radicals produced *via* crosslink scission. Hence the efficiency of shear induced devulcanisation processes can be improved significantly by the effective utilization of a chemical /process that can instantaneously block the reunion of the radicals formed by bond scission. A new concept of blocking the reunion free radicals formed by shear breaking of crosslinks using a stable free radical and thereby increasing the efficiency of devulcanisation is presented. A cyclic nitroxide stable free radical *viz.* 4-hydroxy 2, 2, 6, 6-tetramethylpiperidine 1-oxyl free radical (4-Hydroxy TEMPO abbreviated as 4HT) assisted mechanical devulcanisation of carbon black filled NR vulcanisates (un-aged and aged) in which 4HT was used to block the recombination of the radicals formed by bond scission.

Stable free radical increased the per cent devulcanisation and revulcanisate properties of devulcanised rubber in all cases under consideration. The recombining capacity of different type of cleaved crosslinks (mono-, di- and poly- sulphidic) were found to decisively influence the per cent devulcanisation of samples under shear induced devulcanisation. The influence of factors like cure system, crosslink density, the extent of ageing *etc.* of the original sample on the devulcanisation efficiency and the subsequent revulcanisate properties are discussed in detail.

Key words: Mechanical devulcanisation, Mechano-chemical devulcanisation, Revulcanisate properties, Stable free radical

INTRODUCTION

Devulcanisation is a process of rubber recycling aimed to retrieve polymer chains in used rubber products by selective scission of crosslinks (with minimum main chain scission). The process of devulcanisation envisages maximum retention of vulcanisate properties enabling its maximum reuse in the rubber industry itself. Among the various devulcanisation strategies with inherent merits and demerits, mechano-chemical devulcanisation processes like the De Link process (Sekhar *et al.*, 1998; Sekhar, 2014) and the Lev-Gum process (Goldshtein and

Correspondence: Benny George (E-mail: benny@rubberboard.org.in, georgebenni@hotmail.com)