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# Major Reduction in Chemical Curatives for Rubber Articles

**Ali Ansarifar* and Saad H Sheikh**

*Department of Materials, Loughborough University, UK*

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***Corresponding author:** Ali Ansarifar, Department of Materials, Loughborough University, Leicestershire LE11 3TU UK.

## Opinion

The sulfur cure system in the ethylene-propylene-diene (EPDM)-based Curtain Wall Seal (CWS) has two accelerators, adding up to 2.75 parts per hundred rubber (phr) by weight, and two activators (ZnO: 5phr, stearic acid:1phr) [1]. In total, 8.75phr chemicals are used to fully cure the article with 1phr elemental sulfur. In rubber formulations, chemical curatives are indispensable and once reacted with sulfur at elevated temperature, they produce crosslinks between the rubber chains and provide shape stability, which is essential for the performance, durability and life of the final product in service. Excessive use of chemical curatives is harmful to health, safety, and the environment. According to the European Directive 67/548/EEC, chemicals such as sulfenamide accelerators, zinc oxide and stearic acid are very toxic to aquatic organisms [2]. Stearic acid causes skin and eye irritation in human and is classified as highly flammable [3]. These chemicals are used extensively in the sulfur vulcanization of a wide range of EPDM-based rubber articles.

**Keywords:** Ethylene-Propylene-Diene rubber; N-tert-butyl-2-benzothiazole sulfenamide; Zinc oxide; Stearic Acid; Vulcanization; Oscillating Disc Rheometer

## Aims & Objectives

The aim of this study was to significantly reduce use of N-tert-butyl-2-benzothiazole sulfenamide (a fast curing delayed action accelerator, Santocure TBBS) and zinc oxide (primary activator) in the sulfur cure systems of EPDM-based Curtain Wall Seal and eliminate stearic acid (secondary activator) entirely from the cure system. A new method for measuring the exact optimum amount of the chemical curatives required in the sulfur vulcanization of EPDM rubber was used [4]. All the cure tests were performed at 160°C in an oscillating disc rheometer (ODR) curemeter to produce cure traces from which scorch and optimum cure times, cure rate index and minimum and maximum torques were measured.

## Results

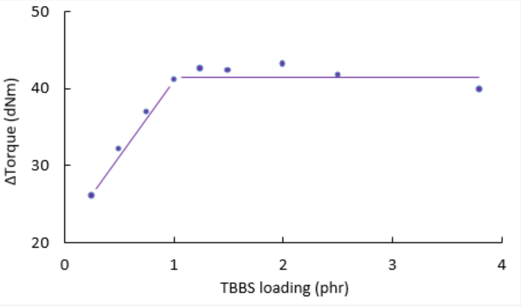


**Figure 1:** ΔTorque versus TBBS loading for the rubber with 1phr sulfur.

Figure 1 demonstrates Δtorque versus TBBS loading for the EPDM rubber with 1phr sulfur. ΔTorque, which is the difference between the maximum and minimum torques on the cure trace of the rubber and is an indirect indication of crosslink density changes in the rubber, increased steeply from 26 to 42 dNm as the loading of TBBS was boosted from 0.25 to 1phr. Subsequently, there was no improvement in Δtorque once the amount of TBBS reached 3.8phr. Apparently, 1phr TBBS was enough to react the sulfur with the rubber. Zinc oxide was then added to improve the efficiency of TBBS. ΔTorque rose noticeably from 41 dNm at 0 phr ZnO to 56 dNm at 0.075phr ZnO and the rate of increase slowed down significantly thereafter. Δtorque then reached to about 67 dNm when the loading of ZnO was raised by an additional 0.325phr (Figure 2). It is interesting that a small amount of ZnO, i.e. as low as 0.075phr, had such a major influence on the performance of TBBS in the cure system as indicated by a significant rise in Δtorque. When 0.5phr stearic acid was mixed with the EPDM rubber with 1phr sulfur, 1phr TBBS & 0.075phr ZnO, Δtorque decreased from 56 to 47 dNm. Δtorque subsequently continued decreasing slowly to about 42 dNm when the loading of stearic acid was raised to 2.5phr (Figure 3). Evidently, the crosslink density as indicated by Δtorque did not benefit from the addition of stearic acid to the rubber. Moreover, the scorch and optimum cure times increased, and the rate of cure as indicated by the cure rate index declined noticeably when stearic added was added [4]. Consequently, stearic acid was eliminated from the cure system entirely. In the absence of stearic acid, no zinc stearate was formed in the rubber. Hence, zinc stearate is not an essential ingredient in the curing of rubber as has been claimed [5]. The scorch time ($t_{s2}$) and the optimum cure time ($t_{95}$) were 6.2 & 21.5 min, respectively. The rate of cure as indicated by the cure rate index (CRI) was 6.5 $min^{-1}$. Clearly, requirement for the accelerator and primary and secondary activators to fully cure the rubber at 1phr loading of sulfur is much lower, i.e. TBBS by 64wt%, zinc oxide by 98.5wt% and stearic acid by 100wt%, than the amounts currently used in the cure system of the CWS at the same loading of sulfur. All the indications are that a significant decrease in the loading of the chemical curatives in the cure system has no adverse effect on the vulcanization of the article. In fact, cure efficiency improves when less accelerators and activators are used with sulfur. This method can be applied to reduce excessive amount of the chemical curatives in other industrial rubber articles.

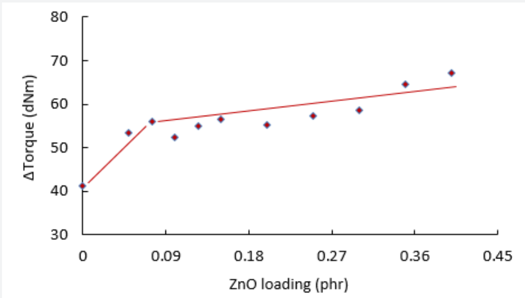


**Figure 2:** ΔTorque versus ZnO loading for the rubber with 1phr sulfur & 1phr TBBS.

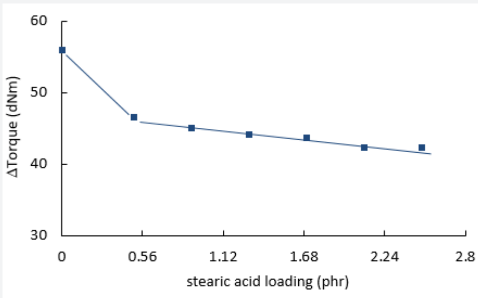


**Figure 3:** ΔTorque versus stearic acid loading for the rubber with 1phr sulfur & 1phr TBBS & 0.075phr ZnO.

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