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Improvements of the processing behaviour of silica compounds

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I. Introduction and scope

The replacement of carbon black by silica in tire tread compounds results in a better performance of the tires in terms of wet-grip, abrasion resistance and rolling resistance; the latter resulting in a lower fuel consumption of the car. These advantages are opposed by the disadvantage of a reduction of the production capacity due to the time- and labor-intensive processing of the compounds.[1] Even though coupling agents are used in order to improve the dispersion of the filler, the silica compounds still have to be mixed in several stages.

This study is focused on adjusting the mixing equipment (fig. 1) in order to enhance the efficiency of the reaction between the coupling agent and the silica filler.

Fig. 1: Internal mixer

II. The silanisation reaction (primary step) [2]

Silica Si-OH + $(H_5C_2\text{-}O)_3Si - C_3H_6 - S_4 - C_3H_6 - Si(OC_2H_5)_3$ ⟷

Bis(triethoxysilylpropyl)tetrasulphide (TESPT)

$$\text{Silica}\; Si - O - \overset{H_5C_2\text{-}O}{\underset{H_5C_2\text{-}O}{Si}} - C_3H_6 - S_4 - C_3H_6 - Si(OC_2H_5)_3 \;+\; H_3C\text{-}CH_2OH$$

III. Measurement of the filler-filler interactions

The Mooney viscosity allows a first estimation of the filler-filler interactions in a rubber compound and indicates pre-scorch of the compound. This measurement does not allow the separation of the two effects.

The Payne effect [3] - the storage modulus G' at low strain - is commonly used to quantify the filler-filler interactions. This effect is inversely related to the hydrophobation of the silica filler by silanisation. The following effects contribute to the storage modulus:

a. the hydrodynamic effect of the filler particles in the polymer matrix

b. the crosslink network connecting the polymer particles;

c. the chemical and physical interactions between the rubber and the filler;

d. the filler-filler interactions

IV. Results

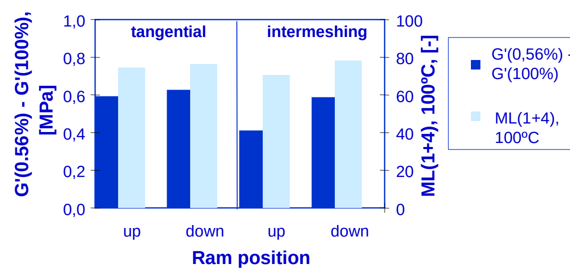

Figure 2: The effect of working in an open mixer (pressure-less silanisation) on the silanisation efficiency (fill factor 60%, cool. temp. 90ºC, sil. temp. 145ºC, sil. time 150")

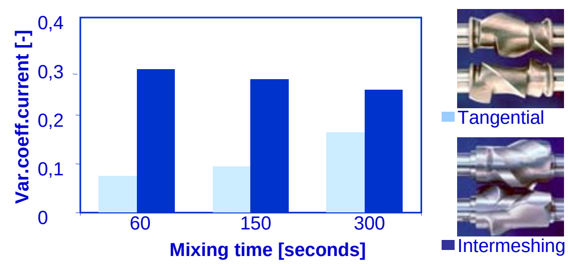

Figure 3: The fluctuation of the current consumption as an indication for the intake behaviour (pressure-less silanisation, cool. temp. 90ºC, fill factor: 60%, sil. temp. 145ºC)

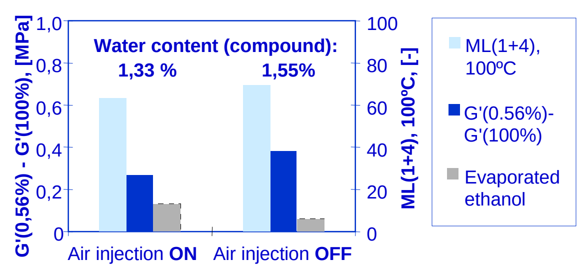

Figure 4: The effect of air injection on the silanisation efficiency (I45, closed mixer, fill factor 40%, cool. temp. 40ºC, sil. temp. 145ºC, sil. time: 150")

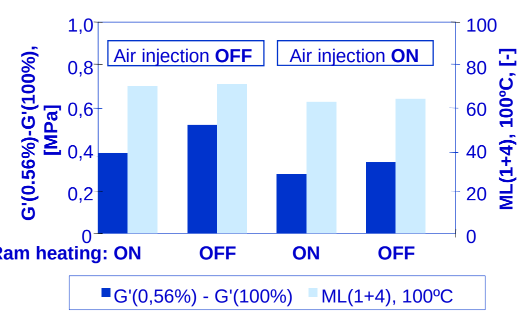

Figure 5: Influence of ram heating on the silanisation efficiency (I45, fill factor 40%, pressure-less silanisation, cool. temp. 40ºC, ram temp. 110ºC, sil. temp. 145ºC, sil.time: 150")

V. Discussion and summary

The efficiency of the silanisation reaction can be improved by adjustments of the mixer design resulting in a more efficient removal of ethanol. This leads to a higher degree of silanisation and hydrophobation reducing the filler-filler interaction and finally allows shorter mixing cycles.

The following measures have been found to improve the efficiency of the silanisation reaction:

- Working pressure-less in an open mixer with an optimized fill factor (fig. 2)
- A rotor design combining the good temperature-control of the intermeshing mixer and the good intake behavior of the tangential mixer (fig. 3)
- Air injection into the mixing chamber for a better ethanol evaporation (fig. 4)
- Heating of condensation-sensitive parts of the mixer (ram) (fig. 5)

VI. Literature:

[1] Thesis L. A. E. M. Reuvekamp: Reactive mixing of silica and rubber for tyres and engine mounts, University Twente, Enschede 2003

[2] U. Görl, A. Hunsche, paper no. 76 presented at the ACS Rubber Division Meeting, October 8-11, 1996, Louisville, Kentucky (USA)

[3] A. R. Payne, Rubber Chem. Technol., 39, 365 (1966)

VII. Acknowledgement: This project is financially supported by the EEC. The technical support from the partner companies (Krupp Elastomertechnik, D; Austrian Automobile-, Motorcycle- and Touring Club, CZ; Tomas Bata University, CZ; Trelleborg Industri AB, SE; University of Paderborn, D; Vredestein Banden BV, NL) is gratefully acknowledged.