

MONITORING THE EFFECT OF FILLER IN ELASTOMERIC MATERIALS BY TIME DOMAIN NMR SPECTROSCOPY

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In the last decades, many efforts have been dedicated to the improvement of the mechanical properties of elastomeric composite materials, as they are particularly attractive for several industrial applications. As a matter of fact, these properties are mainly related to the motional constraints of the polymer network, which are due to physical entanglements and chemical cross-linking between polymer chains, and may be influenced by the presence of different additives and reinforcement fillers (carbon black, nanosilica, clays) [1,2]. Usually, the mechanical properties of the materials are monitored by rheological measurements, which provide only macroscopic observables; however, also a description of the topology and dynamics of the polymer network at the molecular scale is needed in order to have a more complete comprehension of the factors that influence these properties, with the final aim to guide the design of optimized materials. In this context, low field ¹H time domain NMR (TD-NMR) can give an important contribution [3].

In this work, we studied different elastomeric materials with application in the tyre industry, by TD-NMR spectroscopy, with the aim of investigating the effect of filler particles on polymer structure and dynamics. ¹H Multiple Quantum (MQ) experiments [4] were used to evaluate the residual ¹H-¹H dipolar couplings, which arise from the fast anisotropic motion of the polymer chains and are thus directly related to the amount of topological constraints within the polymer network. Moreover, ¹H relaxation times (T₁, T₂) [5,6] were measured to probe a wide range of motional frequencies of the polymer chains. In particular, ¹H spin-lattice relaxation times (T₁) were evaluated by means of Fast Field Cycling [6] experiments at different temperatures, covering Larmor frequencies from 10 kHz to 35 MHz.

References:

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