



UPO UNIVERSITÀ DEL PIEMONTE ORIENTALE
DIPARTIMENTO DI SCIENZE E INNOVAZIONE TECNOLOGICA

EVENTI DiSIT

Seminario

23-09-2022

11:00-13:00

AULA 203

LOW FIELD TD-NMR FOR THE STUDY OF POLYMERIC NETWORKS

[Roberto Simonutti](#)

Department of Materials Science - University of Milan-Bicocca

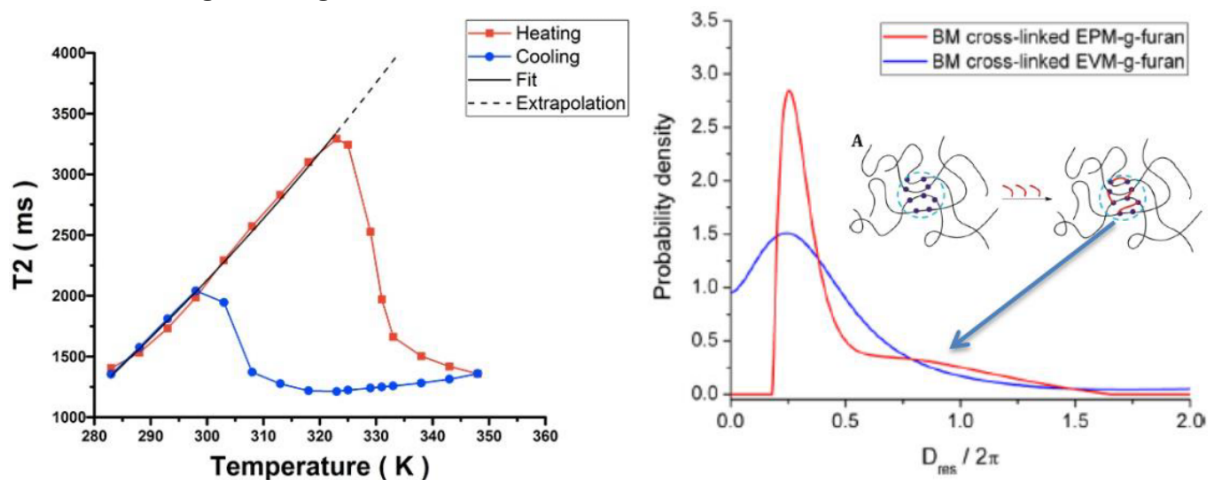


Polymer networks, including rubbers and hydrogels, are ubiquitous materials. The determination of their structure-property relationship is fundamental for their rational improvement.

Low Field (LF) Time Domain (TD) Nuclear Magnetic Resonance (NMR) is a powerful technique to probe molecular-level dynamics, through ^1H relaxation times T_1 e T_2 , and the measure of residual dipolar couplings (D_{res}), which are linearly proportional to crosslinks density (CLD).¹

This technique has been exploited to measure the evolution of crosslinking in conventional rubbers and their blends,² and to prove the formation of polar crosslinks clusters in more innovative modified EPDM rubbers crosslinked via reversible Diels-Alder chemistry³. Figure 1 (right) highlights the bimodal distribution of D_{res} in a heterogeneous system as compared to a more homogeneous one. Access to CLD distribution is the most outstanding advantage of TD-NMR compared to traditional method for the determination of CLD, such as equilibrium swelling measurements.

Important variations in chain dynamics, such as phase transitions, can be easily monitored with the study of T_2 at varying temperatures. In Figure 1, a methylated cellulose thermogelating material⁴ shows increased T_2 upon temperature increase, followed by a sudden drop at the gelation point. The reversibility of the process, the formation of a metastable phase and the associated hysteresis phenomena can be monitored, obtaining results in line with rheological testing.



Left: T_2 relaxation time of methylcellulose gels during a temperature cycle (from 10 to 75°C). Right, distribution of dipolar coupling in a heterogeneous reversibly crosslinked rubber, compared to a more homogeneous sample.

These examples highlight that LF TD-NMR is a comprehensive tool to characterize in details polymeric networks, making it suitable for both academic and industrial applications.

1. D. Besghini, M. Mauri, R. Simonutti, R. Appl. Sci.-Basel 2019, 9 (9).
2. M. K. Dibbanti, M. Mauri, L. Mauri, G. Medaglia and R. Simonutti, Journal of Applied Polymer Science, 2015, 132, 132, 42700.
3. L. M. Polgar, E. Hagting, P. Raffa, M. Mauri, R. Simonutti, F. Picchioni and M. van Duin, Macromolecules, 2017, 50, 8955-8964.
4. D. Besghini, P. Hashemi, M. Knarr, R. Adden, M. Mauri, P. Mischnick, R. Simonutti submitted.

EVENTO APERTO A:

Docenti, Borsisti, Assegnisti, Dottorandi, Studenti, Esterni UNIUPO

