

NMR Relaxometry for Molecular Structure Analysis of Stimuli-Responsive Lyogels in Varied Solvent Polarities

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Introduction: Stimuli-responsive lyogels exhibit pronounced macroscopic changes in response to external stimuli, positioning them as promising candidates for applications such as flow control valves and catalyst carriers in SMART reactors [1]. For their effective integration in such systems, it is essential to understand their internal pore structure and diffusion behavior, as these factors directly influence functionality. While several techniques exist to quantify macroscopic swelling and shrinking, few methods provide insight into the microstructural transformations occurring during these processes.

Methods: To address this gap, we employed ¹H Nuclear Magnetic Resonance (NMR) relaxometry and diffusometry to study solvent dynamics within lyogels under different environmental conditions. NMR is particularly well-suited for examining molecular mobility within macro-, meso-, and nanoporous systems by detecting changes in relaxation times and self-diffusion coefficients [2, 3]. Using a Spinsolve 60 MHz benchtop NMR spectrometer (Magritek, Germany), we investigated the dynamic swelling and shrinking behavior of lyogels in solvents with differing polarity.

Results and discussion: Our measurements revealed significant variations in the relaxation time profiles depending on solvent polarity, reflecting changes in molecular mobility and pore structure during swelling and shrinking (Fig. 1). These relaxation dynamics provide valuable insights into the solvent-polymer interactions at the microstructural level, which are often inaccessible by traditional swelling ratio measurements. The data suggest that solvent polarity play a critical role in modulating the internal morphology and transport behavior of lyogels.

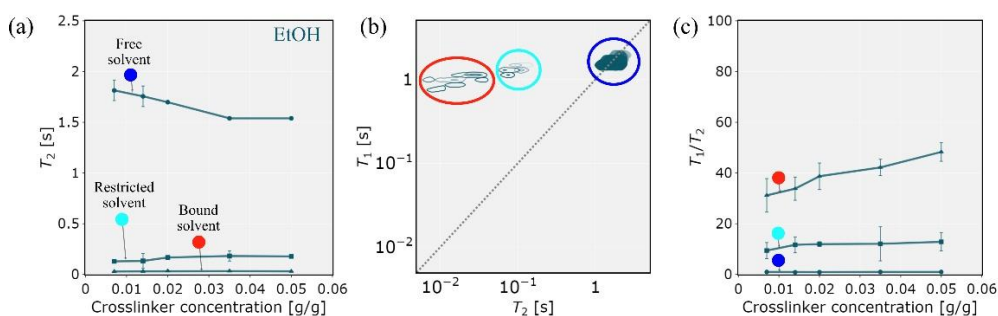


Fig. 1: (a) T_2 relaxation, (b) T_1 - T_2 correlation map, (c) T_1/T_2 ratio of stimuli-responsive lyogels in ethanol (EtOH).

Conclusion: ¹H NMR relaxometry proves to be an effective tool for probing the microstructural evolution of stimuli-responsive lyogels. By offering detailed insights into solvent dynamics and polymer-solvent interactions, this method supports the rational design and integration of lyogels in SMART reactor technologies and other responsive material applications.

References: [1] Eckert, Fluid Phase Equilibria. (2024). [2] Giussi, Soft Matter. (2015). [3] Gruber, Polymer. (2023).