

Advancing SMART Reactors through NMR Relaxometry: Characterization of Stimuli-Responsive Lyogels

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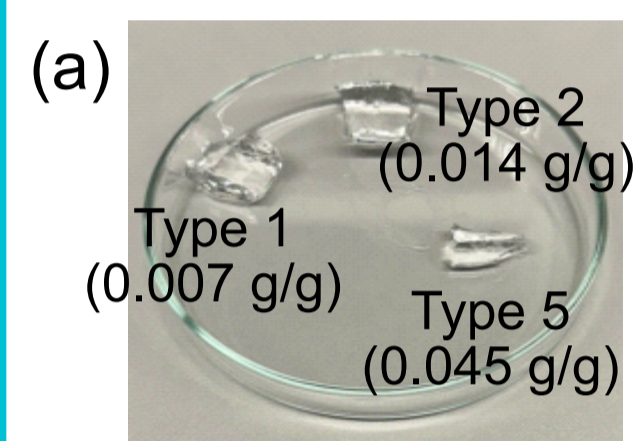
Introduction

Stimuli-responsive lyogels undergo significant macroscopic changes in response to external stimuli. These properties make lyogels ideal for flow control mechanism or as catalyst carriers in SMART reactors [1]. Detailed microstructural analysis during swelling and shrinking phases is crucial for optimizing their performance in these applications.

In our study, we use NMR relaxometry and diffusion measurement to investigate the microstructural changes of lyogels in different solvent polarity at equilibrium state and during the solvent exchange process.

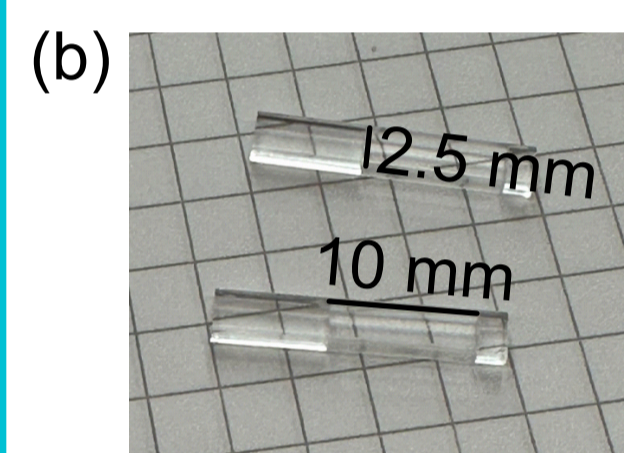
Methods

• Samples:



Lyogels with different crosslink concentration initially in ethanol (EtOH) subsequently exchanging to butyl acetate (BuAc) were examined at equilibrium and during the solvent exchange.

• Sample holder:



Samples were placed on 3D-printed glass holder and then inserted into NMR tubes for measurement.

• Measurements:



Samples were measured on a benchtop NMR in batch sequences included CPMG, PGSTE, and T_1 - T_2 (IR-CPMG), with an echo time of 300 microseconds.

Figure 1. (a) Lyogels in EtOH, (b) 3D-printed glass sample holder, and (c) benchtop NMR.

Results

Measurements of T_2 , self-diffusion coefficient and T_1 - T_2 provide valuable insights into the molecular dynamics and structural properties of the lyogels. A significant effect of solvent polarity, both at equilibrium and during solvent exchange can be observed, revealing the complex interaction between varying solvent polarity and physical characteristics of lyogels.

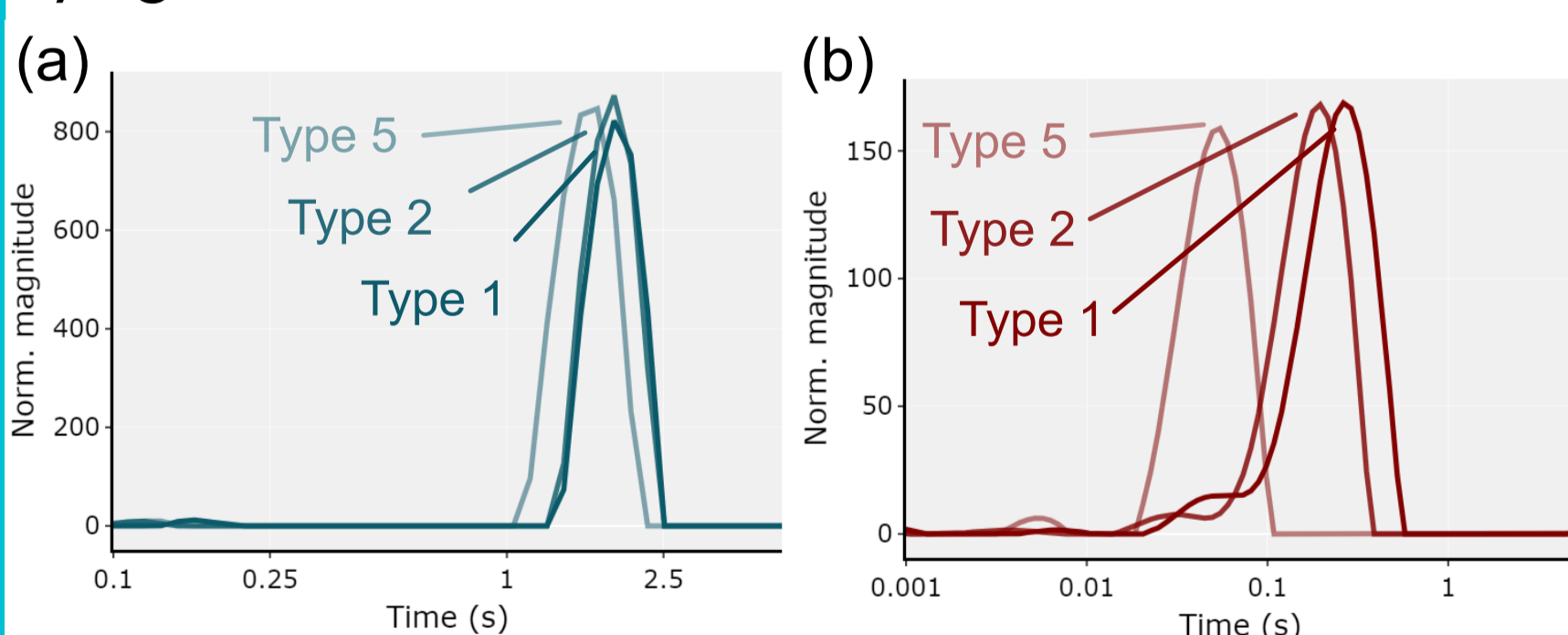


Figure 2. Laplace inversion of CPMG measurement of lyogels (a) in EtOH and (b) in BuAc.

Conclusions

Our study demonstrates the remarkable capabilities of NMR relaxometry in investigating the microstructural changes of stimuli-responsive lyogels with different solvent polarities, both at equilibrium and during solvent exchange process, allowing for the analysis of lyogel kinetics during this process.

Influence of solvent polarity: at equilibrium

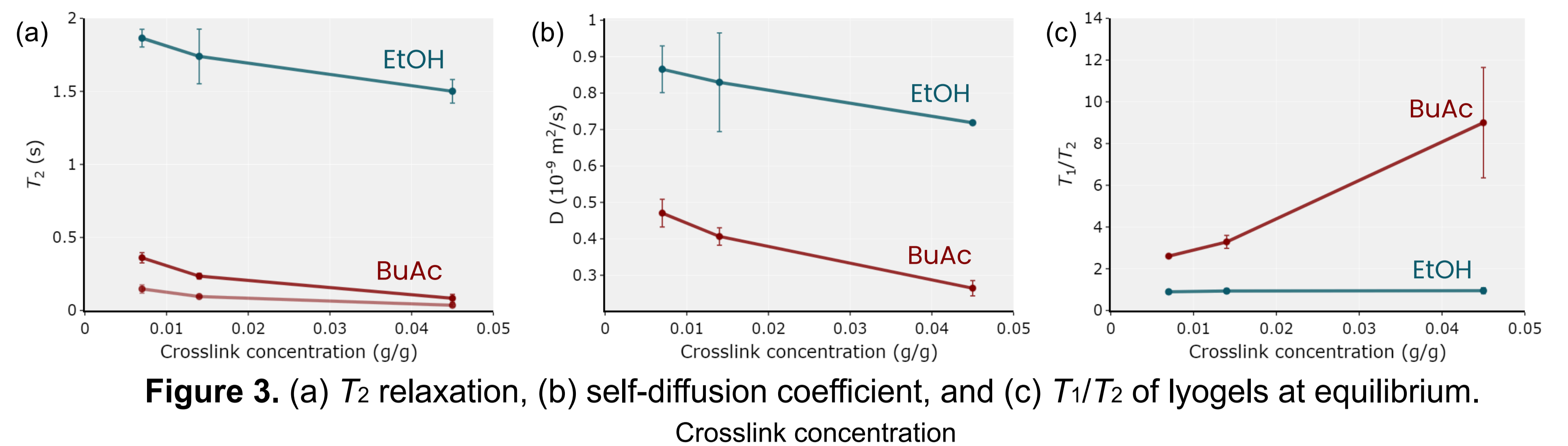


Figure 3. (a) T_2 relaxation, (b) self-diffusion coefficient, and (c) T_1/T_2 of lyogels at equilibrium.

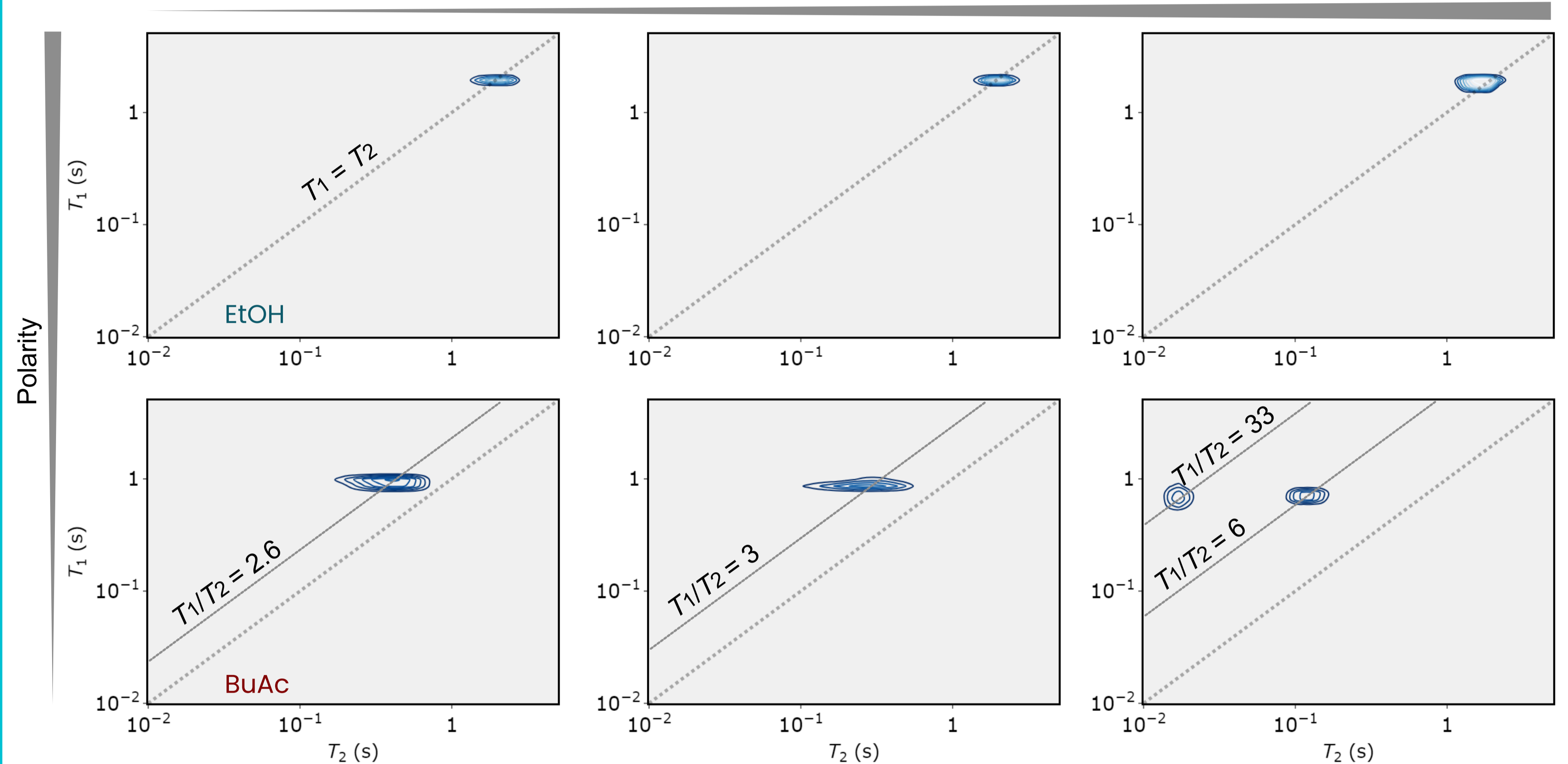


Figure 4. T_1 - T_2 of lyogels at equilibrium.

Influence of solvent polarity: during solvent exchange

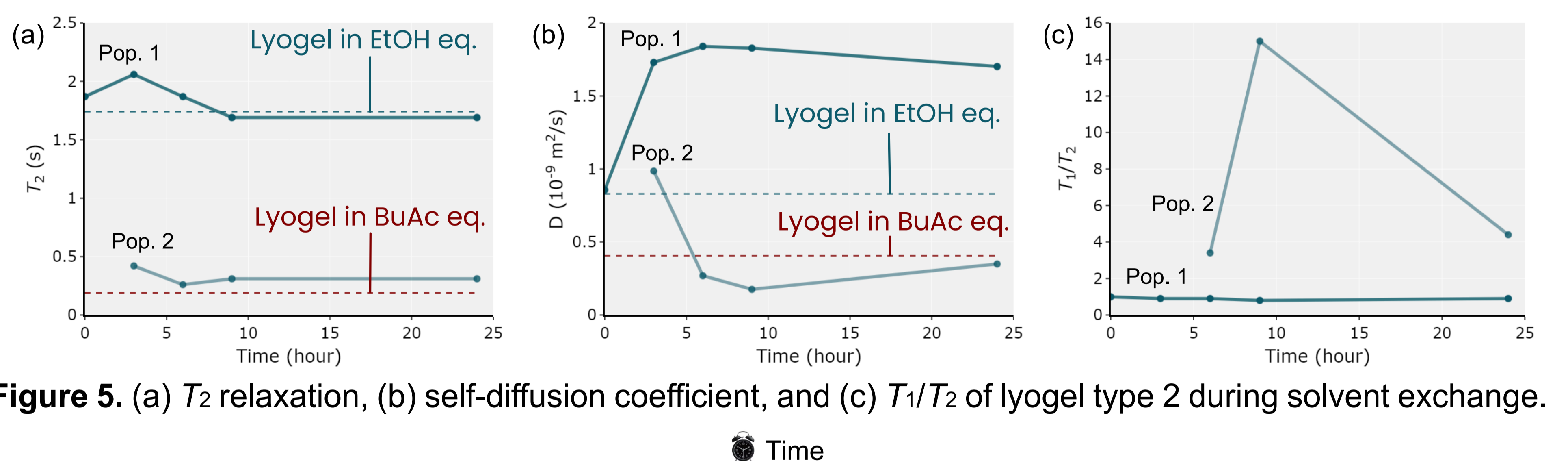


Figure 5. (a) T_2 relaxation, (b) self-diffusion coefficient, and (c) T_1/T_2 of lyogel type 2 during solvent exchange.

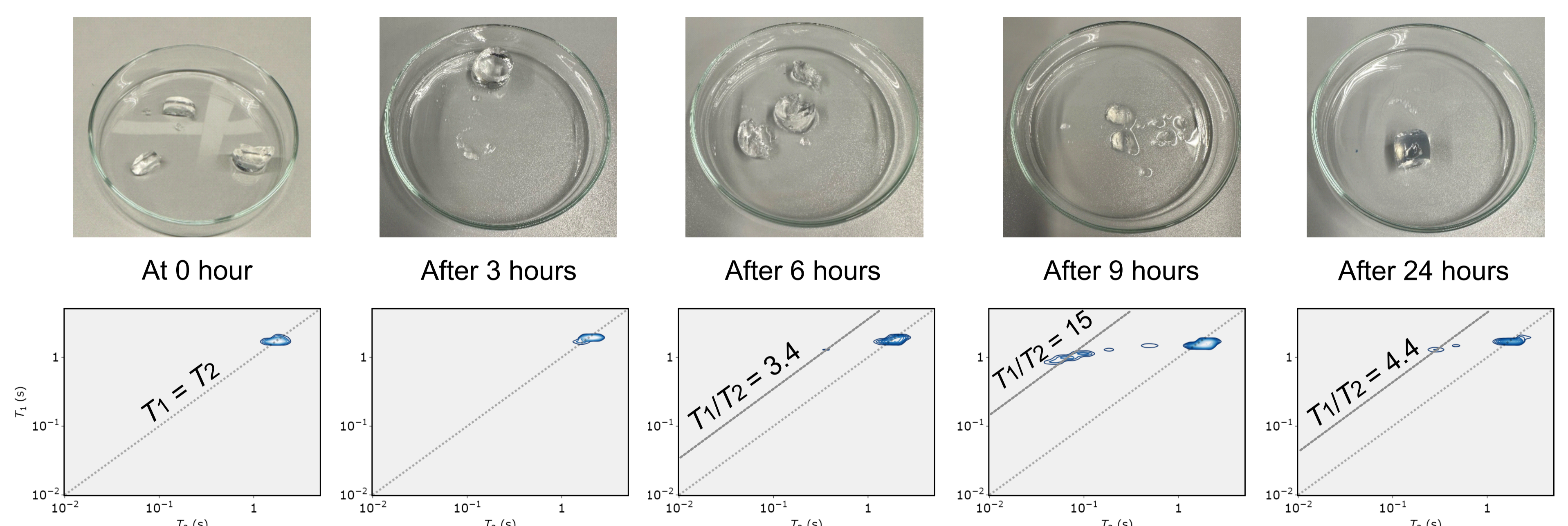


Figure 6. T_1 - T_2 of lyogel type 2 during solvent exchange.

Acknowledgement

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Reference

[1] K. M. Eckert, et al., Fluid Phase Equilibria (2024): 114182.



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