

## **Thixotropic properties of silica-based hydrogels**

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### ***Synthesis of silica-based hydrogels***

Silica sol was prepared by mixing 5 mL of tetraethoxysilane (TEOS), 1 mL of water and 1 mL of HCl aqueous solution (0.25 M). The mixture was stirred at 600 rpm for 1 hour until homogeneity (transparency) was achieved. 0.25 mL of modifier (3-mercaptopropyltrimethoxysilane, 3-aminopropylthiethoxysilane) or TEOS (in case of unmodified silica hydrogel preparation) was added to phosphate buffer solution (pH 7.4), and the resulting mixture was stirred for 8 min at 900 rpm. The transformation of the sol to gel occurred when the strongly acidic silica sol was neutralized with the buffer solution containing modifier or TEOS. The hydrogels were prepared by two methods: by pouring the buffer solution into the silica sol (b/s) or by pouring the sol into the buffer solution (s/b) with stirring at 900 rpm. The pH of the resulting mixture was then measured and the mixture was stirred until gel formation. The obtained hydrogels were aged for 7 days and stored in tightly closed containers.

### ***Investigation of thixotropic properties of hydrogels***

The thixotropic properties of the synthesized hydrogels were studied by two methods: the hysteresis loop method and the three-step method.

#### ***Hysteresis loop method***

In the study of the hydrogels by the hysteresis loop method, the dependences of apparent viscosity on increasing shear rate (from 0 s<sup>-1</sup> to 8.4 s<sup>-1</sup>) and then at decreasing shear rate (from

8.4 s<sup>-1</sup> to 0 s<sup>-1</sup>) were obtained. The measurements were performed on a Brookfield DV2T viscometer (Brookfield, AMETEK, Inc. MA, USA). Thixotropic behavior was evaluated using the index of thixitropy (T), which was defined as

$$T = \frac{S_{up} - S_{down}}{S_{up}} \quad (1),$$

where S<sub>up</sub> and S<sub>down</sub> are the areas under the curve taken at increasing shear rate and under the curve taken at decreasing shear rate, respectively<sup>S1</sup>. The areas were calculated using the trapezoidal method.

#### *Three-step method*

To determine the thixotropic properties using the three-step method, the apparent viscosity relationships of the hydrogels exposed first to a low shear rate (0.01 s<sup>-1</sup>) for 150 seconds, then to an increased shear rate (0.84 s<sup>-1</sup> or 2.1 s<sup>-1</sup>) for 30 seconds, and then again to a low shear rate (0.01 s<sup>-1</sup>) for 150 seconds were obtained. The recovery degree of the hydrogels (R) after exposure to shear forces was calculated as

$$R = \frac{\eta_3}{\eta_1} \times 100 \quad (2),$$

where  $\eta_1$  and  $\eta_3$  are the equilibrium apparent viscosities at the first and third stages, respectively<sup>S2, S3</sup>.

#### **References**

- S1. M. V. Ghica, M. Hîrjău, D. Lupuleasa and C.-E. Dinu-Pîrvu, *Molecules*, 2016, **21**, 786; <https://doi.org/10.3390/molecules21060786>.
- S2. F. K. Rony, G. Kimbell, T. R. Serrano, D. Clay, S. Ilias and M. A. Azad, *Micromachines*, 2025, **16**, 163; <https://doi.org/10.3390/mi16020163>.
- S3. X. Sun, X. Lin, Y. Luo, D. Yu, W. Yan, H. Zhang, Z. Wang, C. Zhang, J. Guo, W. Zhang, W. Gao and S. Huang, *Materials*, 2024, **17**, 4626; <https://doi.org/10.3390/ma17184626>.