

Optimization of synthesis and thermoresponsive characteristics of Hydroxypropyl Cellulose nanogels

Mónica Ledesma-Motolinía¹, Fernando Soto-Bustamante¹, Gavino Bassu¹, Jacopo Vialetto¹, Marco Laurati¹

¹Dipartimento di Chimica 'Ugo Schiff' (DICUS) (University of Florence, Sesto Fiorentino, Florence, 50019, Italy, monica.ledesmamotolinia@unifi.it)

INTRODUCTION & AIM

Hydroxypropyl cellulose (HPC) is a biocompatible polysaccharide and neutral polymer, its **the lower critical solution temperature (LCST)** is between **41 and 44°C** [1,2]. Protocols for the synthesizing of HPC nanogels are few and vary considerably [3–5]. However, HPC nanogel synthesis protocols present high polydispersity, it is not possible to control the particle size, or the synthesis temperature depends on the molecular weight.

In **our approach**, the **optimal synthesis conditions** for **different molecular weights of HPC** were determined by correlating the surfactant concentration and the reaction temperature. Furthermore, **the morphology of HPC nanogels** was characterized as a **function of temperature** using **small-angle neutron scattering (SANS)**.

METHOD

The protocol starts with for one week, at **0.1 wt%** aqueous solution of HPC (**M_w=1 MDa**) was allowed to dissolve with gentle stirring at room temperature. After carefully optimizing surfactant concentration and synthesis temperature, the **HPC nanogels** are **monodisperse** at **1.5 cmc DTAB** and **78 °C**.

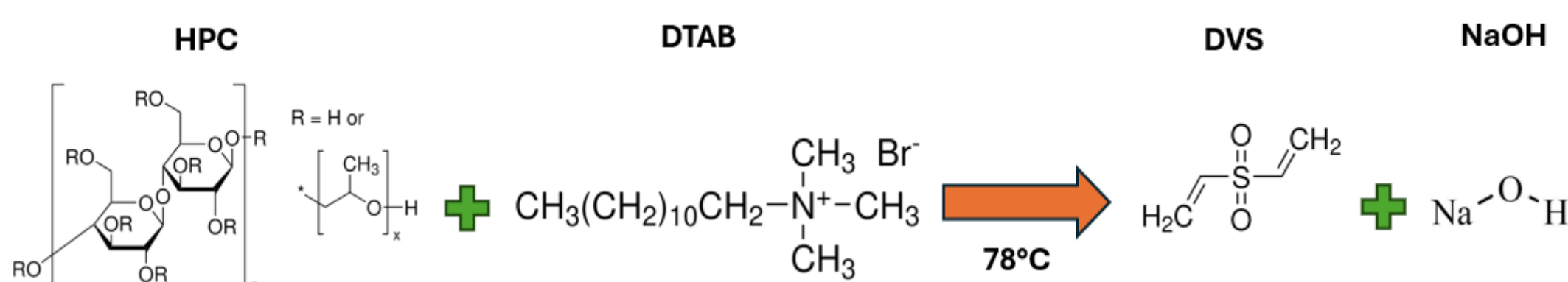


Figure 1. Synthesis of HPC nanogels

RESULTS & DISCUSSION

The **hydrodynamic size (D_h)** was measured by dynamic light scattering (DLS) as a **function of temperature** for 0.1, 0.2, 0.3, 0.5 wt% DVS. **LCST=(44.0±0.1)°C**

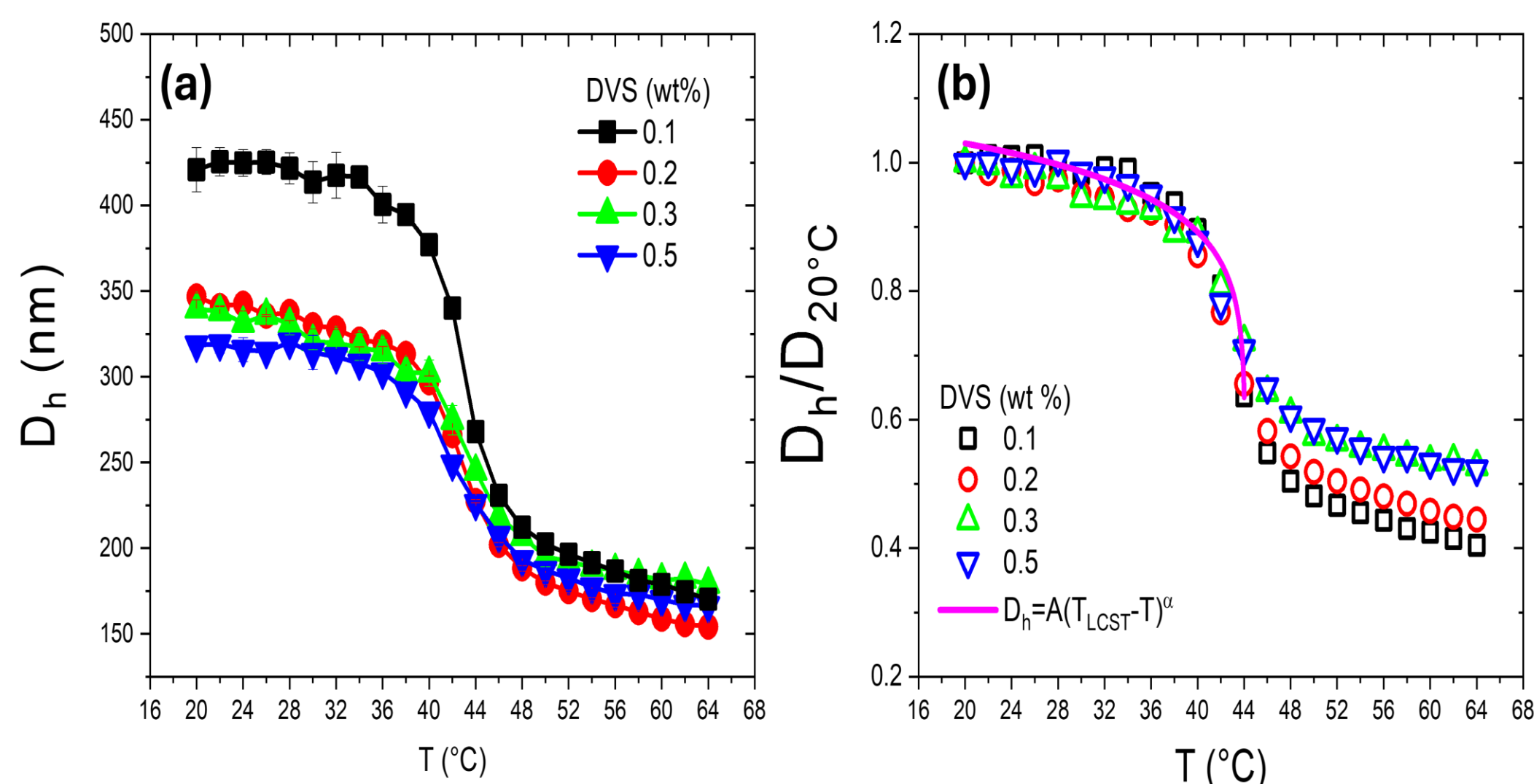


Figure 2. (a) Average hydrodynamic size (D_h), (b) D_h/D_{20°C} as a function of temperature varying DVS cross-linker concentration (wt%).

The **morphology of HPC nanogels** was characterized by **SANS** as a function of DVS (wt%) at 20°C. SANS profiles were fitted using [6]:

$$I(q) = \phi V_p \Delta \rho^2 P(q) S(q) \quad (1)$$

P(q) is the particle form factor that was modeled using a **fuzzy sphere model plus a polymer network** [7]:

$$P(q) = \left[\left(3 \frac{\sin(qR) - qR \cos(qR)}{(qR)^3} \right) \exp \left(-\frac{(\sigma q)^2}{2} \right) \right]^2 + \frac{B}{1 + (\xi q)^2} \quad (2)$$

where **R** is the radius of the particle core, **σ** is the fuzziness parameter. The total radius of the nanogel, **R_{SANS}=R+2σ**, while **R_{BOX}=R−2σ** is the inner region of the nanogel with homogeneous density.

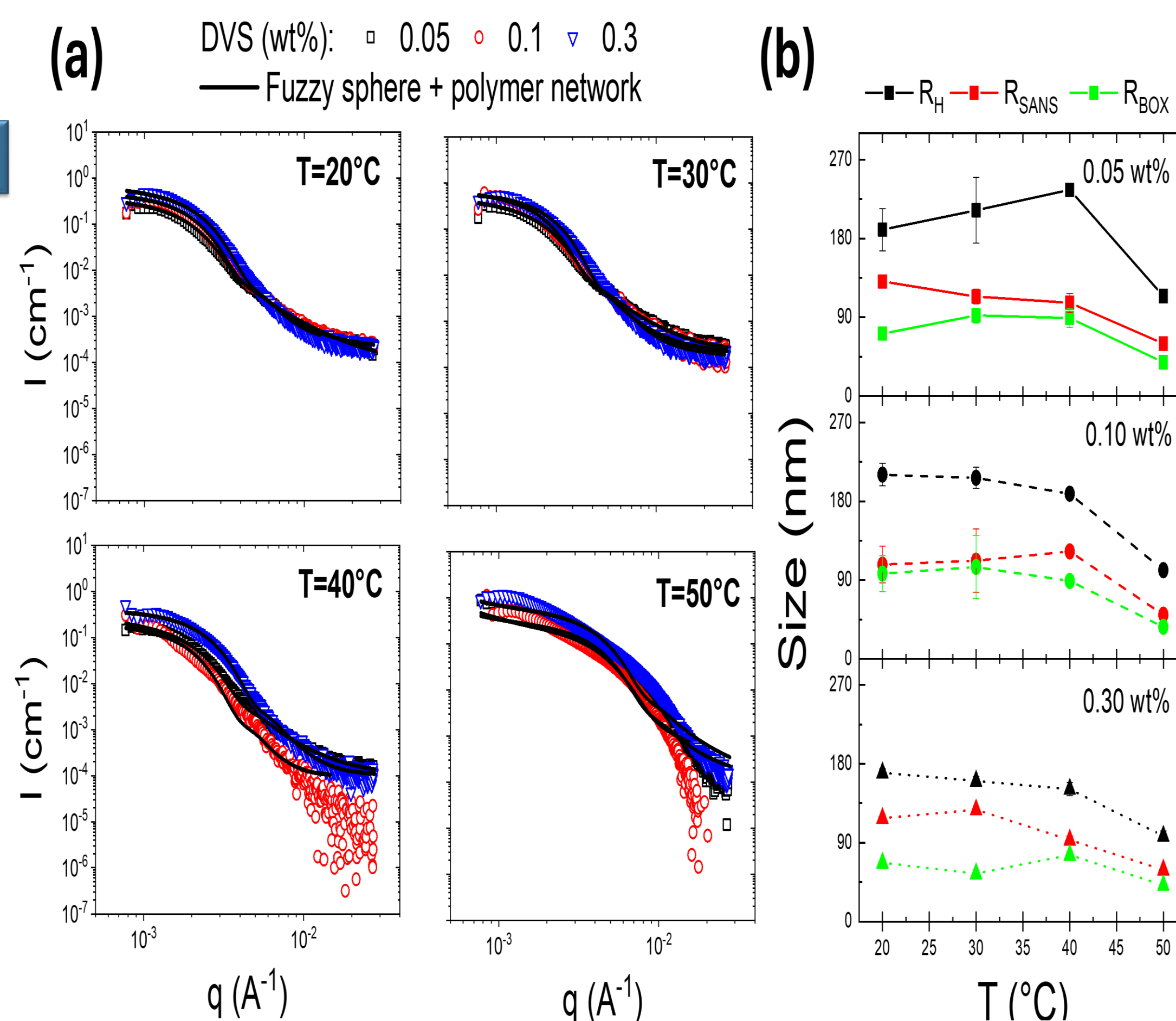


Figure 3. (a) P(q) and black solid lines correspond to fits using Ec. 2. (b) Radii from DLS (R_h) and SANS (R_{SANS}, R_{BOX}) as a function of temperature.

CONCLUSION

The proposed synthesis protocol, it is possible to obtain monodisperse HPC nanogels. Varying the percentage of DVS, the differences between the nanogels occur at 0.05–0.1 wt%. In all systems, the **T_{LCST}=44°C**. The morphology is well represented by a fuzzy sphere polymer network, where **R_{SANS}** does not depend on the DVS concentration, while **R_{BOX}** decreases with increasing DVS.

REFERENCES

- [1]X. Lu, Z. Hu, Z. J. Gao(2000). Macromolecules, 33, 23, 8698–8702. [2]J. Gao, G. Haidar, X. Lu, Z. Hu(2001) Macromolecules, 34, 2242–2247. [3]X. Xia, S. Tang, X. Lu, Z. Hu(2003). Macromolecules 36, 36953698. [4]K. G. Freeman, J. Adamczyk, K. A. Streletsky(2020). Macromolecules, 53, 21, 9244–9253. [5]H. Weng, J. Zhou, L. Tang, Z.Hu(2004).J. Biomater. Sci. Polym. Ed., 15:9, 1167–1180. [6]G. Bassu, J. E. Houston, M. A. Lara-Peña, H. Kriegs, M. Lettinga, L. Porcar, A. Scotti, M. Laurati(2024) Physics of Fluids, 36 (11): 113116. [7]E.Y. Kozhunova, V.Y. Rudyak, X. Li, M. Shibayama, G.S. Peters, O.V. Vyshvannaya, I.R. Nasimova, A.V. Chertovich(2021) J. Colloid Interface Sci., 597, 297–305.