

From Solution to Interface: Structural Evolution of pH-responsive O-Carboxymethyl-Chitosan Microgels with Varying Cross-linking

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Microgels are cross-linked soft-particle networks with colloidal dimensions that swell in a compatible solvent (Scheffold, 2020). Their high deformability, surface activity, tuneable size and composition, and responsiveness to external stimuli make them attractive stabilisers for stimulus-sensitive emulsions. When the polymer matrix is derived from renewable resources, the microgels gain added value for sustainable applications because of their biocompatibility, low toxicity and biodegradability. Consequently, their behaviour at fluid interfaces has attracted considerable interest in the food and pharmaceutical applications (Cai et al., 2025).

In this study we compare bulk swelling and interfacial responses of pH-responsive O-Carboxymethyl-chitosan (O-CMC) microgels with three cross-linking densities using scattering techniques, Langmuir–Blodgett isotherms, and atomic-force microscopy (AFM). At the air-water interface the particles adopt non-isotropic, partially flattened shapes that agree with the scattering data. Lower cross-linked microgels deform markedly more than highly cross-linked ones, indicating stronger interfacial spreading. Surface pressure-area isotherms show pronounced hysteresis during compression–expansion cycles, suggesting partial loss or rearrangement of the monolayer.

While bulk swelling is clearly pH-dependent, the present interfacial data do not reveal a strong pH effect. Future work will perform systematic pH-variation experiments at the interface to probe possible changes in packing, rigidity and monolayer stability.

Keywords:

Atomic Force Microscopy, Langmuir-Blodgett technique, Carboxymethyl Chitosan, Microgels

References:

- Scheffold, F. (2020). Pathways and challenges towards a complete characterization of microgels. *Nature Communications* , 11 (1).
- Cai, J., Zhang, S., Liu, S., Li, X., Wan, Z., Noskov, B. A., & Yang, X. (2025). pH-responsive microgels constructed from soy protein coacervates: Structure and rheology at the oil-water interface. *Food Hydrocolloids* , 167 .