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## Abstract

Biological materials are abundant in nature, forming the structural foundation of both plant and animal bodies. With the growing demand for sustainable alternatives to synthetic plastics, natural polymers have attracted increasing attention. Among these, chitosan, which is produced by the deacetylation of chitin, stands out for its excellent biocompatibility, biodegradability, and functional versatility. Although hierarchical structures such as nanofibers and twisted architectures have been extensively explored, achieving controlled anisotropic microstructures at the millimeter scale remains a significant challenge. Overcoming this limitation is essential for expanding the potential applications of chitosan in the development of sustainable materials.

In order to create a three-dimensionally organized microstructure, a chitosan network was recreated from an aqueous solution using the meniscus splitting approach. When deposited at the millimeter scale, the resultant chitosan membrane demonstrated practical anisotropic pH-responsive hydrogel characteristics. Capillary forces caused chitosan to undergo ordered deposition during the evaporation of the aqueous solution from a confined region, creating a membrane with directed microstructures and microlayers. This membrane formed between two air-liquid interfaces, as opposed to solid-liquid and air-liquid interfaces, where cast films form. Consequently, directed swelling in aqueous environments and reversible/irreversible swelling-deswelling transitions were seen in membranes with ordered microstructures, based on the pH range that is controlled. Furthermore, the evaporative interface created a non-equilibrium state for both dissolved molecules and geometrically asymmetric macrostructures, similar to crystal growth. While multiple-nuclei generation has been observed, the relative positioning of nuclei and dominant influencing factors remain unclear. This study investigates meniscus splitting with multiple nucleation events, focusing on symmetry breaking and asynchronous nucleus formation.

**Keywords:** polysaccharides, pH-responsive, sysmmetry breaking, evaporation, synchronous/asynchronous nucleation