24AG015 BL-12

Monitoring the Self-Assembly of Alginate Induced by Calcium Ions Using Circular Dichroism

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Keywords: Self-assembly, Hydrogel, Circular Dichroism, Phase diagram

Circular dichroism (CD) spectroscopy is a valuable technique for analyzing the secondary structures of proteins and peptides [1]. However, its application to polysaccharide structural studies remains limited. Therefore, evaluating the feasibility of CD spectroscopy for polysaccharide analysis is essential. The alginate (Alg) polysaccharide is known to transition from liquid to gel when exposed to polyvalent ions [2]. This study used CD experiments to investigate the effect of calcium ions (Ca^{2+}) on the structural changes of Alg during self-assembly and hydrogel formation.

In the absence of Ca^{2+}, the CD spectrum of Alg exhibited a peak around 200 nm and a trough near 215 nm (Figure 1a). Both peak intensities and positions gradually changed with increasing Ca^{2+} concentration. Additionally, CD spectra of dried samples closely resembled those of liquid samples, indicating minimal structural changes due to drying. By analyzing the peak intensities and positions in the CD spectra, a phase diagram illustrating self-assembly and aggregation phases was constructed (Figure 1b).

To validate this diagram, ATR and AFM measurements were conducted. ATR spectra showed a peak shift around 1600 cm^{-1}, identifying the concentration range where structural transitions occur, as observed in the CD experiments. AFM measurements were performed at three key concentrations corresponding to the self-assembly, aggregation, and gel states. As Ca^{2+} concentration increased, a densely entangled fibrous structure emerged, correlating with the transition from the self-assembly to aggregation and then to gel state, supporting our findings from CD experiments.

These results highlight the potential of CD spectroscopy for analyzing structural changes in Alg-Ca^{2+} hydrogel system.

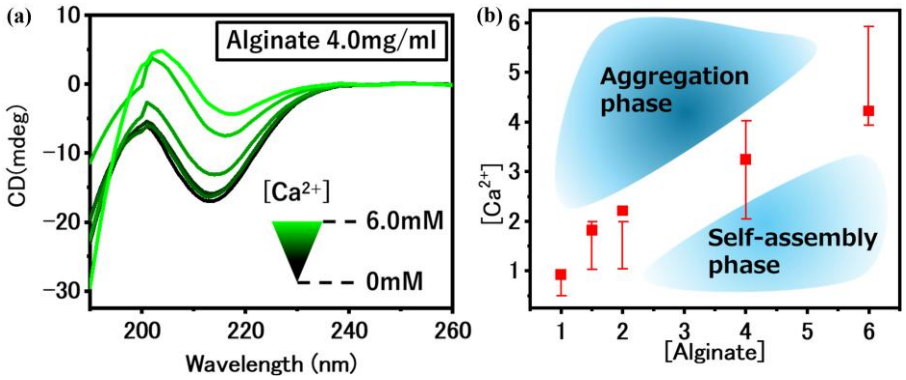

FIGURE 1. CD spectrum of Alg (4.0 mg/ml) as a function of calcium concentration (liquid sample), and (b) proposed phase diagram of Alg-Ca^{2+} system based on CD findings.

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