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Fabrication and characterization of β-cyclodextrin inclusion-based supramolecular hydrogel for potential biomaterials applications

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β-Cyclodextrin (β-CD) inclusion-based supramolecular hydrogels have been developed as potential biomaterials for various biomedical applications including controlled drug delivery. This study focused on the fabrication and characterization of a β-CD inclusion-based novel supramolecular hydrogel with the intention to develop a multi-stimuli responsive hydrogel for controlled and efficient drug delivery applications. Building blocks of the supramolecular hydrogel, namely, BB1, poly (β-cyclodextrin-co-citric acid), and BB2, citric acid ester of phenyl-mono-capped polyethylene glycol-4000 were synthesized using modified literature procedures and characterized. Hydrogel formation conditions of each building block and supramolecular networks with BB1:BB2 (w/w) ratios of 1:1, 1:2, 1:3, 1:4, and 1:5 in water and phosphate-buffered saline (PBS) pH 7.4 were optimized by using several methods. The swelling behavior of the hydrogels in PBS pH 7.4 and water was investigated gravimetrically at ambient conditions over time. The hydrogel with BB1:BB2 (w/w) ratio of 1:2 was selected for further studies and characterized using Fourier transform infrared spectroscopy (FTIR), thermo-gravimetric analysis (TGA), differential scanning calorimetry (DSC), high-performance liquid chromatography (HPLC), and scanning electron microscopy (SEM). Among the different gel formation conditions studied to obtain a stable homogeneous gel network with minimal gelation time, sonication followed by cooling of BB1 and BB2 mixtures to 4 ^{o}C yielded hydrogels with the shortest gelation time of 3-4 days. Hydrogels with BB1:BB2 (w/w) ratios of 1:1 and 1:5 demonstrated the lowest and highest degree of swelling, 356 % and 450 %, respectively. The FTIR spectra of lyophilized hydrogel confirmed the presence of ester bonds. The TGA thermogram of hydrogel demonstrated that hydrogel has thermal stability up to 150 ^{o}C. The DSC thermogram of hydrogel showed an endothermic peak between 400 ^{o}C – 450 ^{o}C. Further, the complete disappearance of the exothermic peak of BB2 in the DSC curve of hydrogel evidenced the molecular encapsulation of the phenyl group of BB2 within the cavity of β-CD in BB1. The HPLC chromatograms of BB1, BB2, and hydrogel demonstrated differences in the retention time of each building block compared with the corresponding building blocks in the hydrogel. This observation further confirmed the inclusion complex formation between BB1 and BB2. SEM image of the hydrogel exhibited the microstructure with pores. In conclusion, a novel, stable, microstructured supramolecular hydrogel was fabricated for potential biomaterials applications. These research findings could facilitate the development of multi-stimuli responsive, supramolecular hydrogel for controlled drug delivery applications.

Keywords: β-Cyclodextrin, Biomaterials, Gelation time, Supramolecular hydrogel, Swelling

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