MOLECULAR INTERACTION AND MECHANISM OF CELLULOSE AS THICKENING AND REINFORCEMENT AGENT IN CARRAGEENAN BIOCOMPOSITE FOR HARD CAPSULE

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SUPERVISOR'S DECLARATION

We hereby declare that We have checked this thesis and in our opinion, this thesis is adequate in terms of scope and quality for the award of the degree of Doctor of Philosophy.

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STUDENT'S DECLARATION

I hereby declare that the work in this thesis is based on my original work except for quotations and citations which have been duly acknowledged. I also declare that it has not been previously or concurrently submitted for any other degree at Universiti Malaysia Pahang or any other institutions.

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ABSTRAK

Kapsul keras berasaskan tumbuhan mempunyai permintaan pasaran yang tinggi kerana berkeupayaan sebagai pembawa ubat untuk vegetarian. Jumlah pengeluaran kapsul keras berasaskan tumbuhan adalah terhad dan masih berkembang untuk pasaran yang lebih luas. Karagenan adalah bahan yang kos efektif dan mempunyai potensi untuk dibangunkan sebagai kapsul keras. Walau bagaimanapun, ia telah mempunyai kekuatan mekanikal yang rendah, yang mengehadkan potensi penggunaannya. Pengisi seperti hidroksipropil metilselulosa (HPMC), kanji sagu karboksimetil (CMSS) dan nanokristal selulosa (CNC) digunakan dalam matriks karagenan sebagai bahan pemekat dan penguat. Ia dihipotesiskan untuk meningkatkan sifat biokomposit dengan pembentukan interaksi antara molekul. Kerja ini bertujuan untuk membangunkan dan mencirikan sifat mekanikal dan termal bagi biokomposit karagenan yang digabungkan dengan HPMC dan CMSS pada kepekatan yang berbeza; untuk mengenal pasti interaksi molekul antara karagenan, HPMC dan CMSS melalui ^{1}H NMR dan simulasi mekanik kuantum menggunakan perisian Gaussian 09W; untuk menyiasat pembentukan CNC di dalam pelarut eutektik (CNCDES) sebagai agen penguat pada keamatan ultrasonik dan kepekatan suapan yang berbeza dan menilai sifat biokomposit karagenan yang telah diperkuatkan; dan untuk menjelaskan mekanisme ikatan hidrogen di sebalik pembentukan DES dan interaksi molekul dengan CNC sebagai agen penguat dalam biokomposit karagenan dan kapsul keras. Biokomposit karagenan yang mengandungi 0.8 w/v% HPMC memberikan kesan ketara pada kekuatan tegangan dan kekuatan gelung kapsul dengan peningkatan masing-masing sebanyak 59.1 dan 46.9%. Suhu peralihan kaca filem Carra-HPMC meningkat daripada 37.8 kepada 65.3 °C, menunjukkan kestabilan termal yang lebih tinggi. Penambahbaikan ini boleh dijelaskan oleh pembentukan ikatan hidrogen antara kumpulan sulfat (karageenan) – hidroksil (HPMC) pada jarak 1.36 Å, seperti yang disimulasikan dalam perisian mekanik kuantum. Ini adalah selaras dengan pergerakan proton karagenan sekitar 3.20 kepada 3.33 ppm dalam spektrum ^{1}H NMR, yang mencadangkan interaksi molekul antara karagenan dan HPMC. Penggabungan CMSS, bagaimanapun, meningkatkan kandungan lembapan biokomposit. CNC yang telah disediakan dalam DES meningkatkan kebolehserakan dan kestabilannya dalam keadaan larutan. Potensi zeta CNC ialah −48.1 mV dan ianya mencukupi untuk mengelakkan penggumpalan partikel. Kehabluran CNC yang tinggi dalam DES menghasilkan peningkatan ketara dalam kekuatan tegangan filem dan kekuatan gelung kapsul masing-masing kepada 84.7 MPa dan 43.7 N. Ikatan hidrogen antara molekul telah terbentuk antara kumpulan hidroksil (CNC) – ion klorida (DES) dengan panjang ikatan 2.47 Å. Pembentukan ikatan antara CNC dan DES mengubah struktur molekul CNC menjadi lebih longgar, yang boleh menjelaskan mekanisme pengembangan CNC apabila tersebar dalam DES. Topografi filem biokomposit diperkuat dengan CNC dalam DES memperlihatkan permukaan yang lebih licin dengan kekasaran purata 19.4 nm, berbanding dengan filem yang diperkuat dengan CNC dalam air ternyahion. Kapsul keras Carra-HPMC/CNCDES juga mempamerkan prestasi yang baik pada peleraian dan pelarutan ubat mengikut peraturan Farmakope AS. Hasil kajian yang dinyatakan di atas menunjukkan bahawa HPMC dan CNC berupaya menjadi bahan pemekat dan penguat yang bagus dalam menghasilkan kapsul keras karagenan.

ABSTRACT

Plant-based hard capsule has a high market demand due to its versatility as a drug delivery carrier for vegetarians. The production volume of plant-based hard capsules is limited and still growing for a broader over-the-counter market. Carrageenan is a cost-effective and promising material to be developed as a hard capsule. However, it was demonstrated to have low mechanical strength, which limits its potential application. Fillers of hydroxypropyl methylcellulose (HPMC), carboxymethyl sago starch (CMSS) and cellulose nanocrystals (CNC) were employed in the carrageenan matrix as thickening and reinforcement agents. It is hypothesized to increase the biocomposite properties by the formation of intermolecular interaction. This work aims to characterize and evaluate the mechanical and thermal properties of carrageenan biocomposite incorporated with HPMC and CMSS at different concentrations; to recognize the molecular interaction between carrageenan, HPMC and CMSS via ^{1}H NMR and quantum mechanics simulation using Gaussian 09W software; to investigate the formation of CNC in deep eutectic solvent (CNCDES) as reinforcement agent at different ultrasonication intensity and feed concentration and evaluate the properties of the reinforced carrageenan biocomposite; and to elucidate the hydrogen bonding mechanism behind the formation of DES and the molecular interaction with CNC as the reinforcement agent in carrageenan biocomposite and hard capsules. Carrageenan biocomposite with the incorporation of 0.8 w/v% HPMC presented significant effects on the tensile and capsule loop strength with an improvement of 59.1 and 46.9%, respectively. The glass transition of the Carra-HPMC film increased from 37.8 to 65.3 °C, implying higher thermal stability. These improvements could be elucidated by the hydrogen bond formation between sulphate (carrageenan) – hydroxyl (HPMC) groups at a distance of 1.36 Å, as simulated in quantum mechanics software. This is in agreement with the downfield movement around 3.20 ppm of the carrageenan proton to 3.33 ppm in the ^{1}H NMR spectrum, which suggests the intermolecular interaction between carrageenan and HPMC. The incorporation of CMSS, however, increased the moisture content of the biocomposite. CNC was prepared in DES, which improved its dispersibility and stability in the solution state. The zeta potential of CNC was −48.1 mV, which was sufficient to avoid particle agglomeration. The high crystallinity of CNCDES resulted in a significant improvement in the film tensile and the capsule loop strength to 84.7 MPa and 43.7 N, respectively. The intermolecular hydrogen bond was formed between hydroxyl group (CNC) – chloride ion (DES) at a bond length of 2.47 Å. The bond formation between CNC and DES modified the molecular structure of CNC becoming looser, which could explain the swelling mechanism of CNC when dispersed in DES. The topography of the biocomposite film with CNC prepared in DES presented a smoother surface with an average roughness of 19.4 nm, compared to the film with CNC in deionized water. Carra-HPMC/CNCDES hard capsule also presented a good performance on disintegration and drug dissolution following the regulation of US Pharmacopoeia. These aforementioned results indicate that HPMC and CNC would be ingenious thickening and reinforcement agents in producing carrageenan hard capsules.

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