CHITOSAN CROSSLINKING STRATEGIES USING VANILLIN: A SUSTAINABLE MICROENCAPSULATION SYSTEM

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Abstract: Currently, to attain the competitiveness and demand of industrial sectors, scientific researches are devoted to the development of innovative products, achieved through technologies such as microencapsulation. Chitosan (CS) is a natural-derived polymer widely employed as an encapsulating material, due to its antimicrobial capacity, biodegradability and non-toxicity. Also, its solubility in acidic solutions could be overcome by physical or chemical crosslinking. In this context, the application of emerging crosslinking strategies to avoid the traditionally used toxic agents, e.g. glutaraldehyde, has gained interest towards more sustainable approaches including non-toxic crosslinking agents such as tripolyphosphate (TPP) or vanillin (VA). Therefore, in this work, CS microparticles were prepared through spray-coagulation technique by the combination of TPP and VA dual crosslinking. Initially, a physical crosslinking of a CS solution of 3% (w/v) in acetic acid medium 3% (w/v) was carried out by atomization into a coagulation bath with different TPP concentrations (10, 5 and 3% (w/v)) at pH 6. Herein, two VA chemical crosslinking strategies were designed, where both VA 1% (w/v) solutions were stirred at 50 ºC for 2 h: (1) the VA solution was added in a controlled flow into the coagulation bath containing the microparticles; (2) the atomized microparticles were filtered and added into the VA solution. The success in the formation of CS microparticles crosslinked with TPP was monitored by optical microscopy (OM) during the spray-coagulation process, while Fourier transform infrared spectroscopy (FTIR) spectra confirmed the structural rearrangement of CS by Schiff base formation (1643 cm^{-1}), evidencing the crosslinking reaction with VA. Scanning electron microscopy (SEM) of dried microsystems indicated the formation of shrunken and rough-surfaced microparticles. In general, the effectiveness of the crosslinking approach was corroborated by thermogravimetric analysis (TGA) revealing an increase of the final weight residue after the crosslinking and by swelling tests in acidified medium, leading to less swollen microparticles with unperceived dissociation degrees. The CS crosslinked microsystem with TPP 5% and VA through strategy (1) exhibited the most potential properties, to be proposed for the microencapsulation of bioactive principles.

Keywords: Microencapsulation; Chitosan; Tripolyphosphate; Vanillin; Green Crosslinking.

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