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Chitosan/starch-based films modified with alginate dialdehyde

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INTRODUCTION & AIM

Interest in biomaterials based on natural polymers has recently surged. The natural polymers are large molecular compounds, which can be obtained from living organisms or are produced by living organisms. Biopolymers typically exhibit properties highly desirable in tissue engineering, including biocompatibility, biodegradability, and minimal immune responses upon introduction into the human body. They can be classified into polysaccharides, proteins, and nucleic acids. Chitosan (CTS) and starch (ST) belong to the group of polysaccharides and are widely investigated for applications in tissue engineering, including wound healing. However, materials based on single biopolymers or blends often exhibit insufficient physicochemical properties. Therefore, modification strategies, such as chemical cross-linking, are required. Dialdehydes are increasingly explored as natural, non-toxic cross-linking agents. Starch dialdehyde, chitosan dialdehyde, alginate dialdehyde, and other modified polysaccharides have been used for the modification of biopolymer materials.

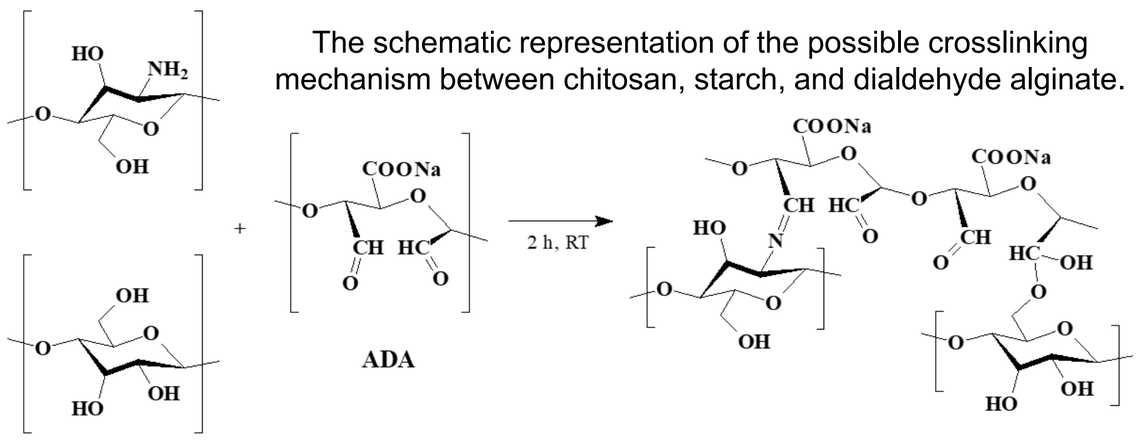
The aim of this study was to obtain and characterize thin films based on chitosan and starch, modified with alginate dialdehyde (ADA).

METHODS

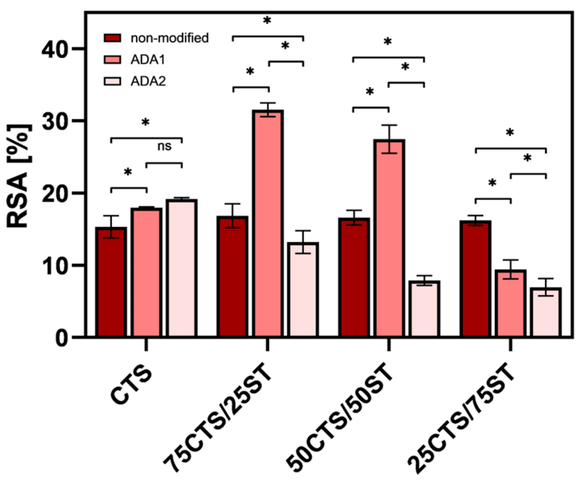
CTS/ST films were prepared using the solvent casting method. A 2% (w/v) chitosan solution and a 2% (w/v) starch solution were mixed in a following mass ratios: 75/25, 50/50 and 25/75, followed by stirring for 2 h. Glycerol was added as a plasticizer. ADA was incorporated as a crosslinking agent at 5% (w/w) relative to the total polymer content. The final solutions were cast onto square polystyrene plates (10 × 10 cm) and left to dry at room temperature under ambient humidity for 72 h to allow solvent evaporation.

Fabrication of Dialdehyde Alginate: Products with different aldehyde group contents were obtained by applying two different concentrations of periodic acid. The oxidation reactions were carried out in aqueous solution at 4 °C for 5 days. Firstly, SA was dissolved in distilled water and stored in a dark bottle. Then, an aqueous solution of sodium periodate was added to the SA solution, and the reaction mixture was magnetically stirred for 5 days at 4 °C in the dark. After completion, the reaction was quenched by adding ethylene glycol and stirring for 1 h at room temperature. The oxidized alginate was isolated by adding NaCl and ethanol. The obtained polymer was redissolved in water and reprecipitated by the addition of ethanol in the presence of NaCl. Finally, the precipitation was filtered, washed with acetone and dried under vacuum at room temperature. The aldehyde group content in dialdehyde alginate was determined by colorimetric titration.

RESULTS



The schematic representation of the possible crosslinking mechanism between chitosan, starch, and dialdehyde alginate.



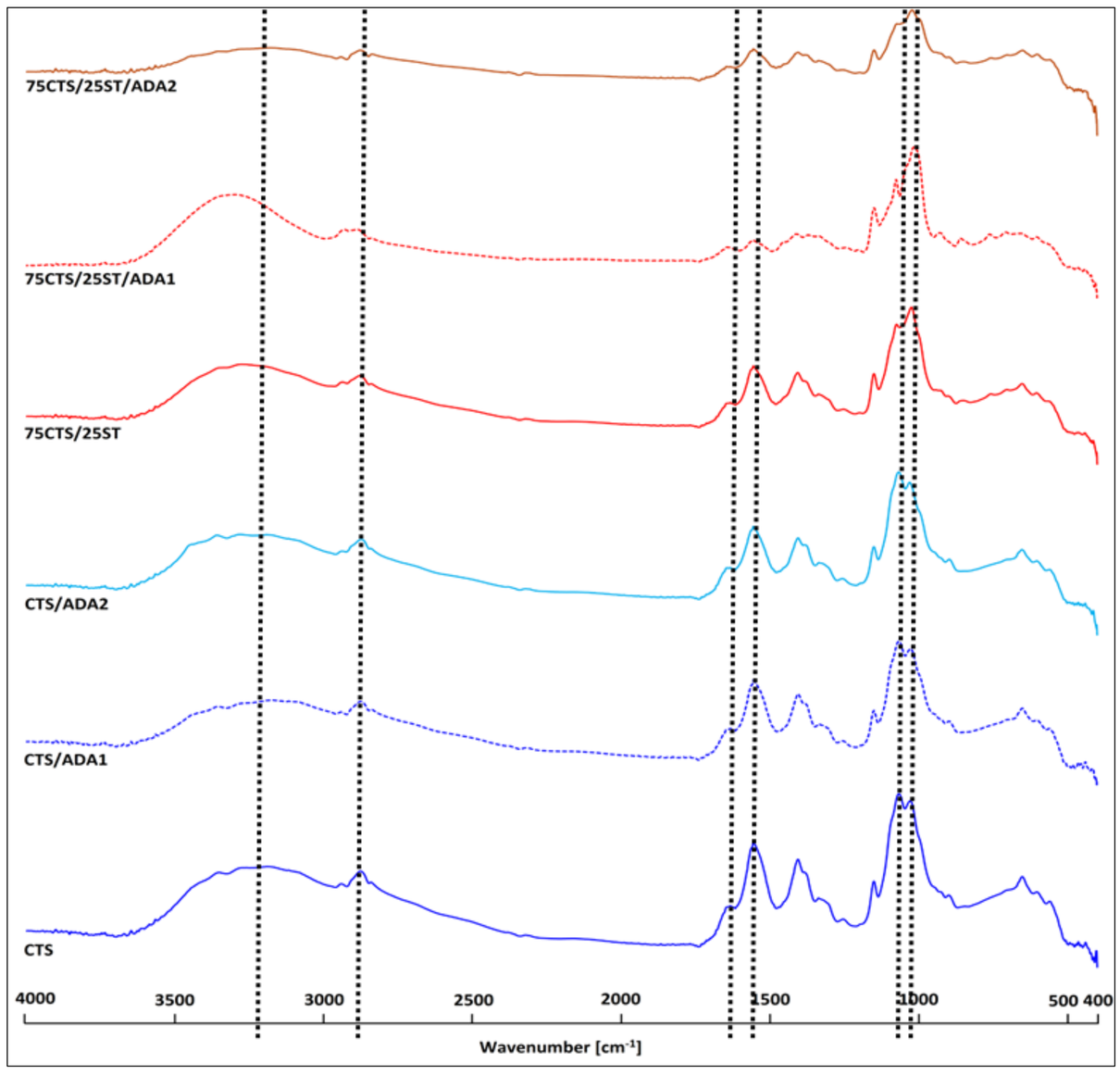
Left – the antioxidant activity, right – the moisture content of tested CTS/ST mixtures modified with dialdehyde alginate; *—significant difference between samples for each group of blends.

Sample	Moisture content [mg/100g of dry sample]
CTS	9.07 ± 0.21
CTS/ADA1	6.87 ± 0.50*
CTS/ADA2	5.43 ± 0.74*
75CTS/25ST	1.67 ± 0.40
75CTS/25ST/ADA1	2.23 ± 0.15^{ns}
75CTS/25ST/ADA2	2.13 ± 0.15^{ns}
50CTS/50ST	2.57 ± 0.38
50CTS/50ST/ADA1	3.13 ± 0.15^{ns}
50CTS/50ST/ADA2	2.23 ± 0.06^{ns}
25CTS/75ST	3.53 ± 0.55
25CTS/75ST/ADA1	2.23 ± 0.06*
25CTS/75ST/ADA2	1.97 ± 0.47*

RESULTS

Sample	Contact angle [°]		IFT(s) [mJ/m^2]	IFT(s,D) [mJ/m^2]	IFT(s,P) [mJ/m^2]
	G	D			
CTS	53.75 ± 1.74	89.97 ± 1.08	42.99 ± 0.48	4.78 ± 0.10	38.22 ± 0.38
CTS/ADA1	54.37 ± 1.89^{ns}	86.22 ± 2.42^{ns}	41.29 ± 0.80	6.16 ± 0.25	35.13 ± 0.55
CTS/ADA2	53.90 ± 1.18^{ns}	83.04 ± 2.19^{ns}	40.94 ± 0.68	7.37 ± 0.25	33.57 ± 0.43
75CTS/25ST	62.50 ± 2.60	72.26 ± 2.16	33.65 ± 0.79	13.61 ± 0.34	20.04 ± 0.45
75CTS/25ST/ADA1	51.40 ± 2.86*	76.63 ± 2.19*	41.86 ± 0.91	9.90 ± 0.30	31.96 ± 0.61
75CTS/25ST/ADA2	51.38 ± 1.48*	93.65 ± 1.79*	46.68 ± 0.60	3.46 ± 0.14	43.22 ± 0.46
50CTS/50ST	59.87 ± 2.02	85.34 ± 1.54	36.32 ± 0.59	7.07 ± 0.18	29.25 ± 0.41
50CTS/50ST/ADA1	53.82 ± 1.73^{ns}	86.53 ± 2.79^{ns}	41.87 ± 0.88	5.99 ± 0.29	35.87 ± 0.59
50CTS/50ST/ADA2	57.92 ± 2.17^{ns}	93.37 ± 2.30*	40.22 ± 0.77	4.04 ± 0.20	36.18 ± 0.57
25CTS/75ST	55.12 ± 2.31	90.14 ± 2.86	41.77 ± 0.94	4.84 ± 0.27	36.94 ± 0.67
25CTS/75ST/ADA1	58.76 ± 2.69^{ns}	88.00 ± 2.47^{ns}	37.85 ± 0.87	5.93 ± 0.26	31.93 ± 0.62
25CTS/75ST/ADA2	55.34 ± 1.87^{ns}	82.90 ± 2.71*	39.69 ± 0.87	7.58 ± 0.31	32.11 ± 0.56

The results of contact angles, surface free energy (IFT (s)), and its dispersive (IFT (s,D)) and polar (IFT (s,P)) components of the tested films (n = 5).



The FTIR-ATR spectra of the examined materials. Chitosan and 75CTS/25ST mixture with ADA1 and ADA2).

CONCLUSIONS

Films based on CTS and ST prepared in different mass ratios (75/25, 50/50, 25/75) were successfully obtained and modified with ADA containing two different contents of aldehyde groups (0.843 and 0.787 mol CHO/mol alginate). FTIR confirmed the presence of characteristic functional groups of the constituent biopolymers, additionally the modification of films by ADA crosslinking, affected bands shifts. The ADA modification influenced the water content of the films, generally reducing moisture uptake due to the additional network formation. Films crosslinked with ADA1 exhibited improved antioxidant activity in the DPPH assay, which may be attributed to the presence of reactive aldehyde-derived structures. Furthermore, the contact angle results showed tunable wettability depending on the CTS/ST ratio and aldehyde content, which may be advantageous for tailoring surface interactions in biomedical applications. Overall, the obtained results suggest that the proposed films represent favorable properties and antioxidant potential, which highlight their suitability for further development toward wound dressing and related biomedical applications.