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SYNTHESIS AND CHARACTERIZATION OF COVALENTLY CROSSLINKED SERICIN-DIALDEHYDE INULIN COMPOSITES

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Abstract

Dialdehyde inulin was synthesized by periodate oxidation of inulin with controlled degrees of oxidation ranging from 5% to 35%. The oxidation introduced reactive aldehyde groups through cleavage of vicinal diols, enhancing the reactivity of the polysaccharide. The obtained dialdehyde inulin samples were reacted with sericin at different mass ratios (1:1, 1:2, and 2:1) to form composite materials. The interaction occurred via Schiff base formation between aldehyde and amino groups, resulting in a covalently crosslinked structure. FTIR analysis confirmed the formation of imine -C=N-) linkages and the reduction of aldehyde groups. The properties of the resulting composites were found to depend on the degree of oxidation and component ratio. The results demonstrate an effective approach for the preparation of sericin-dialdehyde inulin composites with potential applications as biodegradable materials.

Keywords: *Dialdehyde inulin; Sericin; Composite materials; Schiff base; FTIR spectroscopy; Biodegradable polymers*

Introduction

Increasing environmental concerns and the limitations of synthetic polymers, such as poor biodegradability and environmental persistence, have driven the development of sustainable materials based on natural polymers (Barclay, T. G., Ginic-Markovic, M., Cooper, P. D., & Petrovsky, N., 2010). Biopolymers derived from renewable resources are attractive due to their biodegradability, biocompatibility, and low toxicity, making them suitable for biomedical and material science applications.

Inulin, a fructan-type polysaccharide composed of β-(2→1)-linked fructose units, is widely recognized for its biocompatibility and chemical versatility (Muhammad, M., Willems, C., Rodríguez-Fernández, J., Gallego-Ferrer, G., & Groth, T., 2020). The presence of vicinal hydroxyl groups enables selective modification via periodate oxidation, which cleaves C–C bonds and introduces reactive aldehyde (–CHO) groups, forming dialdehyde inulin (Wang, P., He, H., Cai, R., Zhang, X., & Chen, J., 2019). The physicochemical properties of oxidized inulin strongly depend on the degree of oxidation, which controls the density of functional groups. Oxidized polysaccharides containing aldehyde groups can form covalent networks with proteins through Schiff base reactions, resulting in imine (–C=N-) linkages that enhance structural stability and functional properties (Aramwit, P., Siritientong, T., Srichana, T., & Ratanavaraporn, J., 2012). Sericin, a natural protein obtained from Bombyx mori cocoons, contains amino and hydroxyl groups that make it suitable for such interactions and has been widely investigated in biomaterial applications (Khusenov, A. S., Kamalova, D. S., Kee, N. K., & Rakhmanberdiev, G., 2024).

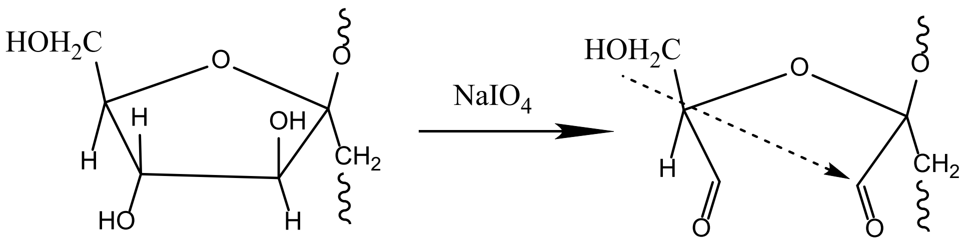
Despite numerous studies on sericin-based composites, most reported systems are based on physical interactions, while covalently crosslinked systems using dialdehyde inulin remain insufficiently explored. Therefore, the present study aims to synthesize a biodegradable composite material by oxidizing inulin to dialdehyde inulin and subsequently reacting it with sericin via Schiff base formation, followed by structural characterization using FTIR spectroscopy.

Materials and Methods

Sericin extracted from *Bombyx mori* cocoons, inulin (CAS 9005–80–5), and sodium periodate ($NaIO_4$, TU 6–09–02–54–74) were used as starting materials.

Preparation of dialdehyde inulin. Inulin was dissolved in distilled water under continuous stirring at room temperature. $NaIO_4$ was added at a molar ratio of 1:1–1:1.5 relative to the repeating units of inulin, and the reaction was carried out in the dark for 4 h. The reaction was quenched with ethylene glycol, followed by dialysis against distilled water for 24 h. The product was then freeze-dried to obtain dialdehyde inulin as a dry powder (Afinjuomo, F., Fouladian, P., Barclay, T. G., Song, Y., Petrovsky, N., & Garg, S., 2020). The oxidation proceeds via cleavage of vicinal diol groups, resulting in the formation of dialdehyde inulin, as shown in **Scheme 1.**

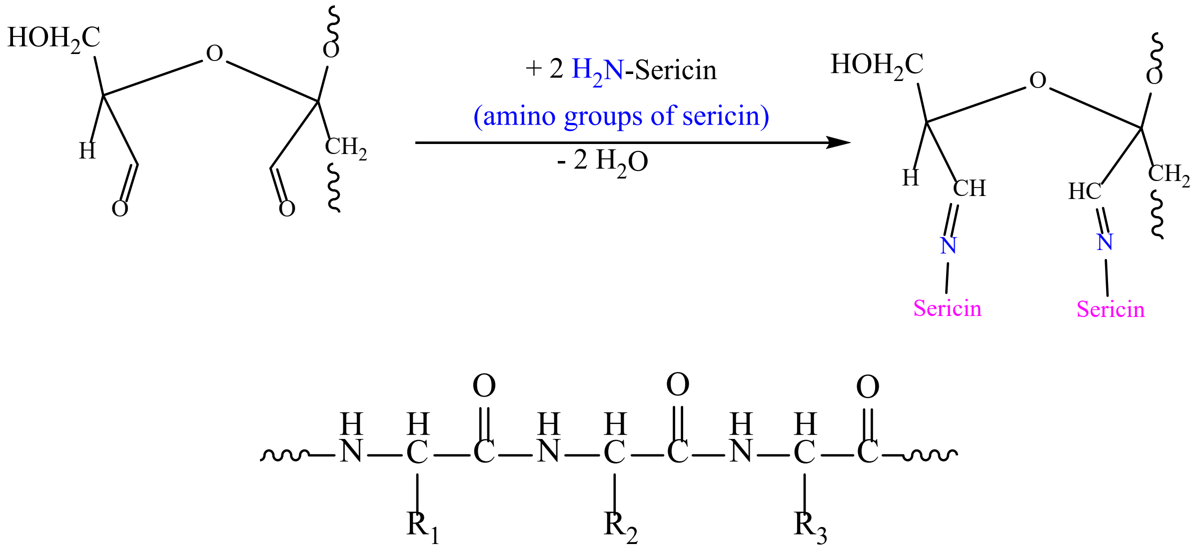
Scheme 1. *Periodate oxidation of inulin*



Preparation of sericin. Sericin was extracted from Bombyx mori cocoons by degumming. The cocoons were cut into small pieces and boiled in 0.02 M Na_2CO_3 solution for 30–60 min. The solution was filtered, cooled, and dialyzed against distilled water for 24 h to remove impurities. Finally, the purified sericin was freeze-dried to obtain a dry powder (Zhang, Y. Q., 2002).

Preparation of Sericin Dialdehyde Inulin Composite. Dialdehyde inulin was reacted with sericin to obtain a covalently crosslinked composite. Briefly, dialdehyde inulin was dissolved in distilled water, and an aqueous sericin solution was added at a predetermined ratio. The mixture was stirred at room temperature for several hours.

Scheme 2. *Formation of Schiff base linkages between dialdehyde inulin and sericin*



Sericin (model structure)

FTIR Spectroscopy. The FTIR spectra of the obtained samples were recorded using a Shimadzu IRSpirit-X FTIR spectrometer (Japan). The samples were prepared as KBr pellets by mixing approximately 2–3 mg of dried sample with 100–300 mg of KBr powder and pressing into pellets. The spectra were collected in the wavenumber range of 400–4000 cm^{-1} with 64 scans.

Results and Discussion

In this study, inulin was oxidized with sodium periodate to obtain dialdehyde inulin with degrees of oxidation ranging from 5–35%. The oxidation involved cleavage of vicinal diol groups, resulting in C–C bond scission and the formation of reactive aldehyde (–CHO) groups. Increasing the oxidation degree led to a higher density of aldehyde functionalities and enhanced polymer reactivity, accompanied by partial depolymerization.

The obtained dialdehyde inulin samples were reacted with sericin at different mass ratios (1:1, 1:2, and 2:1) to form composite materials. The interaction occurred via nucleophilic addition of amino groups to aldehyde groups, followed by dehydration, leading to the formation of imine (–C=N-) linkages and a covalently crosslinked network structure.

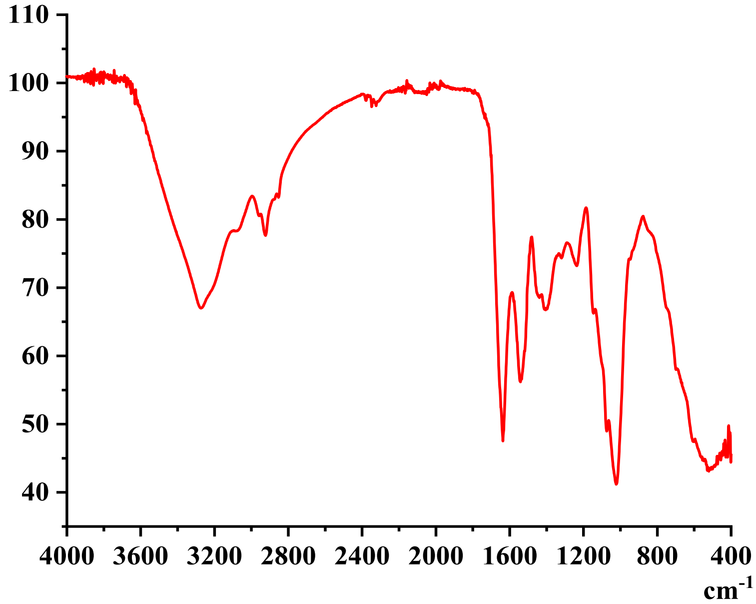
The degree of crosslinking depended on both the oxidation level of inulin and the sericin content, allowing modulation of the composite structure and properties. FTIR spectroscopy was used to confirm covalent bond formation and structural changes by comparing the spectra of inulin, oxidized inulin, sericin, and the resulting composites. The FTIR results are discussed in the following section.

The FTIR spectrum of the obtained sericin-dialdehyde inulin composite is presented in **Figure 1**. A broad absorption band observed in the region of 3200–3400 cm^{-1} can be attributed to the stretching vibrations of hydroxyl (–OH) and amino (–NH) groups, indicating the presence of both polysaccharide and protein components.

The intensity and broadness of this band suggest the formation of intermolecular interactions, such as hydrogen bonding within the composite structure. A characteristic absorption band in the region of 1650–1620 cm^{-1} is assigned to the stretching vibration of imine (–C=N-) groups, confirming the formation of Schiff base linkages between dialdehyde inulin and sericin.

This band is a key indicator of covalent interaction and demonstrates successful crosslinking of the components. The band observed near 1540 cm^{-1} corresponds to the amide II vibrations of sericin, which are associated with -N-H bending and C-N stretching. The presence of this band confirms that the protein structure of sericin is retained in the composite material.

Figure 1. *FTIR spectrum of sericin–dialdehyde inulin composite*



In addition, strong absorption bands in the range of 1000–1150 cm^{-1} are attributed to C-O-C and C-O stretching vibrations typical for polysaccharide structures, indicating the presence of the inulin backbone. The changes in intensity and position of these bands compared to native inulin suggest structural modification after oxidation and subsequent reaction. Notably, the characteristic aldehyde (–CHO) band around 1700 cm^{-1} is significantly reduced or absent in the composite spectrum, indicating the consumption of aldehyde groups during the reaction with amino groups of sericin. This observation further supports the formation of covalent imine bonds.

Overall, the FTIR results confirm the successful formation of a sericin-dialdehyde inulin composite through Schiff base reactions, resulting in a covalently crosslinked network structure.

Conclusion

In this study, inulin was oxidized using sodium periodate to obtain dialdehyde inulin with degrees of oxidation ranging from 5% to 35%. The obtained products were reacted with sericin at different ratios to form covalently crosslinked composite materials. FTIR analysis confirmed the formation of Schiff base (–C=N-) linkages between dialdehyde inulin and sericin. In particular, the appearance of imine bands in the region of 1620–1650 cm^{-1} and the reduction or disappearance of aldehyde bands provided evidence of covalent bonding. The results demonstrate an effective approach for the preparation of sericin-dialdehyde inulin composite materials and indicate their potential application as biodegradable materials.

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