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## STRUCTURE AND PROPERTIES OF DIALDEHYDE DEXTRAN-SILK FIBROIN COMPOSITE FILMS

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### Abstract

In this study, composite films based on dialdehyde dextran (DAD) and silk fibroin (SF) were synthesized. DAD was obtained via periodate oxidation, introducing reactive aldehyde groups into the polysaccharide structure. SF extracted from *Bombyx mori* cocoons was used as the protein component. The interaction between DAD and SF occurs through the formation of Schiff base (C=N) linkages between aldehyde and amino groups, resulting in covalent crosslinking. FTIR analysis confirmed these interactions by the decrease of aldehyde bands, shifts in amide regions, and the appearance of a new band at 1620–1640 cm<sup>-1</sup>. Water vapor permeability studies showed that the film properties depend on the SF: Dex ratio, with reduced permeability observed at higher crosslinking densities. The results demonstrate that chemical crosslinking significantly influences the structure and functional properties of the composite films.

**Keywords:** dialdehyde dextran; silk fibroin; composite films; schiff base; ftir spectroscopy; water vapor permeability

### Introduction

In recent years, the development of functional materials based on natural polymers has gained significant attention due to their low toxicity, biocompatibility, biodegradability, and wide application potential in fields such as medicine, biotechnology, and materials science (Mazurek, Ł., Szudzik-Kopec, M., & Czaja, K., 2022). In particular, composites derived from polysaccharides and protein-

based biopolymers offer advantages over synthetic materials, including environmental safety and tunable functional properties.

Polysaccharides are especially attractive due to the presence of reactive functional groups that enable chemical modification and control over physicochemical properties (Muhammad, M., Willems, C., Rodríguez-Fernández, J., Gallego-Ferrer, G., & Groth, T., 2020). Among them, dextran

stands out because of its high water solubility, biocompatibility, and ease of modification. Periodate oxidation of dextran introduces reactive aldehyde groups, forming dialdehyde dextran (DAD), which can interact with other biopolymers to form chemically crosslinked systems (Akhmedov, O., Khabibullaev, J., Abdurakhmanov, J., & Shomurotov, S., 2023; Loyeau, P. A., Spotti, M. L., Castel, V., Acosta-Müller, C., Acosta, C. A., Fioramonti, S., Vinderola, G., & Spotti, M. J., 2026).

Silk fibroin (SF), a protein-based biopolymer, is widely recognized for its fibrous structure, mechanical strength, and versatility in forming films, fibers, and hydrogels (Wang, P., He, H., Cai, R., Zhang, X., & Chen, J., 2019). The presence of amino groups in SF enables its interaction with aldehyde-containing polymers, leading to the formation of stable composite structures.

In this context, the aim of this study is to synthesize composite films based on DAD and SF and to investigate their physicochemical properties.

### Materials and Methods

Silk fibroin (CAS 92580–42–0), sodium periodate (TU 6–09–02–54–74), and dextran 40 (CAS 9004–54–0) were used as starting materials.

#### **Preparation of dialdehyde dextran.**

Dextran was oxidized with sodium periodate to obtain DAD. Briefly, dextran (5 g) was dissolved in sodium acetate buffer (pH 4.25) and reacted with  $\text{NaIO}_4$  at a molar ratio of 1:1.5 in the dark at room temperature for 3–7 h. The reaction was quenched with ethylene glycol, followed by dialysis for 24 h and freeze-drying to obtain DAD. The aldehyde group content was determined by alkaline titration, where a 0.1 g sample was treated with 0.2 N NaOH, heated at 70 °C, cooled, neutralized with 0.2 N  $\text{H}_2\text{SO}_4$ , and titrated with 0.2 N NaOH using phenolphthalein as an indicator until a stable pale pink endpoint was reached (Yan, X., Zheng, W., Yu, Y., Liu, R., Gong, Y., Huang, M., Fan, M., & Wang, L., 2025).

**Preparation of silk fibroin.** Silk fibroin (SF) was extracted from *Bombyx mori* cocoons by degumming in 0.02 M  $\text{Na}_2\text{CO}_3$  solution, followed by thorough washing to neutral pH. The purified fibroin was dissolved in Ajisawa's reagent ( $\text{CaCl}_2$ :  $\text{C}_2\text{H}_5\text{OH}$ :

$\text{H}_2\text{O}$ , molar ratio 1:2:8) at 60 °C, filtered, and dialyzed (MWCO 3 kDa) to obtain a homogeneous SF solution. For composite formation, SF solution was reacted with dialdehyde dextran (DAD) at aldehyde-to-amino molar ratios of 1:1–1:2 under stirring at room temperature. The resulting mixture was dialyzed and subsequently freeze-dried to obtain the final product (Riaz, S., Waheed, H., Ahmad, F., Khan, M. I., & Shanableh, A., 2025).

**FTIR Spectroscopy.** The FTIR spectra of the obtained samples were recorded using an IRSpirit-X FTIR spectrometer (Shimadzu, Japan). The samples were prepared as KBr pellets (3 mg sample mixed with 300 mg KBr), and spectra were collected in the wavenumber range of 400–4000  $\text{cm}^{-1}$ .

**Water Vapor Permeability Measurement.** The water vapor permeability (WVP) of the composite films was determined by a gravimetric method, where films sealed over water-containing vessels were monitored for mass loss over time (12–72 h), and WVP was calculated from the weight loss rate normalized to film area and thickness; all measurements were performed in triplicate (McHugh, T. H., Avena-Bustillos, R., & Krochta, J. M., 1993).

### Results and Discussion

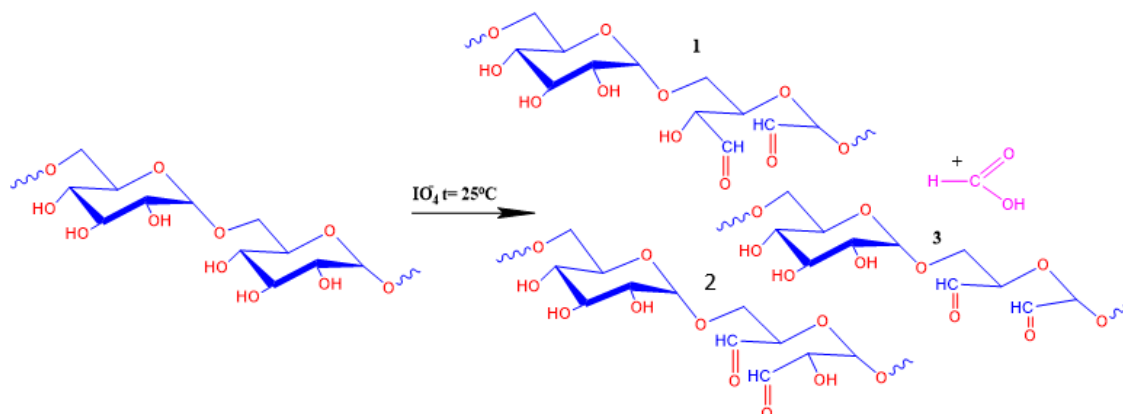
Dextran was oxidized with sodium periodate to obtain dialdehyde dextran (DAD), where cleavage of vicinal diol groups in the polysaccharide backbone generated reactive aldehyde functionalities, enhancing its chemical reactivity and enabling subsequent interactions with biopolymers. As shown in Scheme 1, periodate oxidation of dextran cleaves C–C bonds at C(2)–C(3) or C(3)–C(4), generating reactive aldehyde groups; under excess oxidant, further oxidation may produce formic acid and additional aldehydes.

In this study, DAD with an oxidation degree of ~30 mol% was obtained, which provides an optimal balance between chain integrity and reactivity. Silk fibroin (SF) was extracted from *Bombyx mori* cocoons by alkaline degumming in  $\text{Na}_2\text{CO}_3$  solution, followed by thorough washing and drying to obtain pure fibroin fibers suitable for further processing. Subsequently, SF was reacted with DAD via aldehyde amino interactions, where  $\epsilon$ -amino groups of SF form

azomethine (C=N) bonds with DAD through a Schiff base reaction. This process involves nucleophilic addition followed by dehydra-

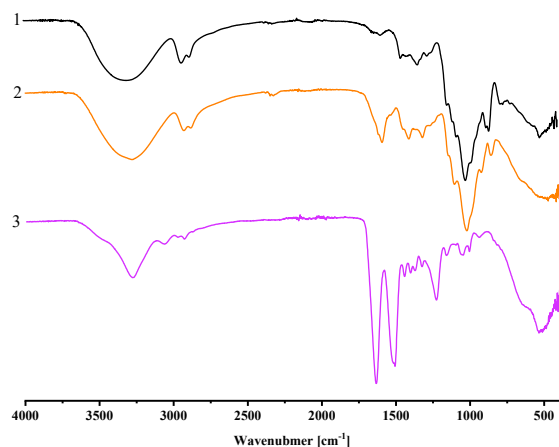
tion, resulting in covalent crosslinking that enhances the structural stability of the composite system.

**Scheme 1.** Periodate oxidation of dextran



The structure of the obtained compounds was characterized by various physicochemical methods. The FTIR spectra of SF, DAD, and their composite are presented in Figure 1 and reveal the characteristic structural changes occurring during composite formation.

**Figure 1.** FTIR spectra of DAD (1), silk fibroin–dextran composite (2) and SF (3)



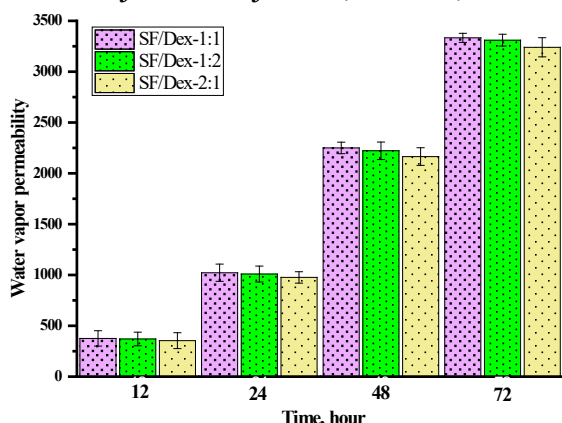
As shown in Figure 1, the FTIR spectrum of SF exhibits a broad absorption band around 3300  $cm^{-1}$ , corresponding to -NH and -OH groups, as well as characteristic bands at 1650  $cm^{-1}$  (amide I), 1530  $cm^{-1}$  (amide II), and 1230  $cm^{-1}$  (amide III), confirming the protein structure of fibroin. The FTIR spectrum of DAD shows a broad band near 3400  $cm^{-1}$  assigned to hydroxyl groups and intense absorption bands in the 1000–1100  $cm^{-1}$  region, which are characteristic

of C-O-C stretching vibrations in the polysaccharide backbone. In addition, a weak band around 1700  $cm^{-1}$  attributable to aldehyde groups confirms the successful oxidation of dextran. In the spectrum of the composite material, noticeable changes were observed compared to the spectra of the initial components. In particular, the intensity of the aldehyde-related band decreased, while the amide bands of fibroin showed shifts and changes in intensity. Moreover, the appearance of a new absorption band in the 1620–1640  $cm^{-1}$  region can be attributed to the formation of azomethine (C=N) bonds. These FTIR results confirm the chemical interaction between fibroin and DAD and indicate the formation of covalent crosslinks within the composite system.

In subsequent studies, the water vapor permeability of SF/Dex composite films was investigated as a function of time (Figure 2). The results showed that water vapor permeability (WVP) of all samples increased with time due to progressive diffusion of water vapor through the films. The SF: Dex ratio significantly affected the film structure, leading to noticeable differences in WVP values. In particular, increasing DAD content resulted in reduced WVP, indicating the formation of a denser and more ordered composite structure.

This behavior is consistent with FTIR results, where the formation of azomethine (C=N) bonds between SF and DAD suggests chemical crosslinking, enhancing structural compactness and influencing vapor permeability.

**Figure 2.** Water vapor permeability of SF/Dex composite films prepared at different ratios (SF: Dex = 1:1, 1:2 and 2:1) as a function of time (12–72 h)



## Conclusion

Composite films based on silk fibroin (SF) and dialdehyde dextran (DAD) were successfully synthesized, exhibiting homogeneous structure, smooth morphology, and good transparency. FTIR analysis confirmed the formation of Schiff base (C=N) linkages between SF and DAD, indicating covalent cross-linking. Water vapor permeability results showed that the SF: DAD ratio significantly affects the internal structure, with higher DAD content leading to reduced permeability due to a denser network. Overall, covalent crosslinking plays a key role in determining the structural and functional properties of the composite films.

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