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Mechanical performance, film-forming ability and structure–mechanical correlation of cross-linked and grafted chitosan biopolymer composites

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Abstract

The development of sustainable polymer films with enhanced mechanical integrity is crucial for emerging applications in packaging, biomedical devices and flexible materials. In the present study, pure chitosan, cross-linked chitosan and grafted chitosan biopolymer composites were synthesized and systematically investigated with respect to their film-forming behavior, mechanical properties and structure–mechanical relationships. Structural modifications were confirmed using FTIR and X-ray diffraction analyses, while surface morphology was examined by SEM. Mechanical properties including tensile strength, elongation at break and Young’s modulus were evaluated using standardized tensile testing. Cross-linking and grafting significantly improved tensile strength and dimensional stability while reducing brittleness associated with pure chitosan films. The observed enhancement in mechanical performance is attributed to reduced crystallinity, improved stress transfer and restricted polymer chain mobility induced by chemical modification. The results demonstrate that modified chitosan composites are promising candidates for eco-friendly structural and functional film applications.

Keywords: Chitosan films, mechanical properties, tensile strength, polymer composites, structure–property relationship

Introduction

Biopolymer-based films have gained considerable attention as viable alternatives to petroleum-derived plastics due to growing environmental concerns, stringent regulatory frameworks and the global demand for sustainable materials [1-3]. The excessive accumulation of non-biodegradable plastics has led to severe ecological challenges, prompting intensive research into renewable, biodegradable and eco-friendly polymeric materials. Among naturally occurring biopolymers, chitosan—a partially deacetylated derivative of chitin—has emerged as a promising candidate owing to its biodegradability, biocompatibility, non-toxicity and abundance of reactive functional groups. Chitosan exhibits excellent film-forming ability, enabling the fabrication of transparent, flexible and homogeneous films without the need for harsh processing conditions. As a result, chitosan-based films have found potential applications in diverse fields such as food packaging, biomedical membranes, wound dressings, drug delivery systems and agricultural coatings [4-7].

Despite these inherent advantages, the practical utilization of pure chitosan films is significantly constrained by several intrinsic limitations. Pure chitosan films tend to exhibit brittle behavior, high moisture sensitivity and only moderate mechanical strength, primarily due to their semi-crystalline nature and extensive intermolecular hydrogen bonding. The strong hydrophilicity of chitosan further leads to excessive water uptake, dimensional instability and deterioration of mechanical performance under humid conditions. These shortcomings restrict the broader applicability of chitosan films in load-bearing, flexible or mechanically demanding environments where durability, toughness and structural integrity are essential [7-13].

To overcome these limitations, chemical modification of chitosan has been widely explored as an effective strategy to tailor its physicochemical and mechanical properties. Cross-linking introduces covalent interchain linkages that restrict polymer chain mobility, enhance network rigidity and improve dimensional stability. Similarly, graft copolymerization allows the incorporation of polymeric side chains onto the chitosan backbone, leading to modified intermolecular interactions, reduced crystallinity and improved stress distribution within the polymer matrix. Such structural modifications not only enhance mechanical strength and flexibility but also improve resistance to moisture-induced degradation, thereby extending the functional applicability of chitosan-based films [14-17].

While extensive research efforts have been devoted to the adsorption behavior, thermal stability and physicochemical properties of modified chitosan systems, systematic investigations focusing on the correlation between chemical structure and mechanical film performance remain comparatively limited. In particular, comprehensive studies linking cross-linking and grafting-induced structural changes with tensile strength, elongation behavior and film-forming stability are scarce. The present work aims to address this critical gap by establishing a clear structure–mechanical property relationship for chitosan-based composite films, thereby providing fundamental insights for the rational design of mechanically robust, sustainable biopolymer films for advanced applications.

Experimental Section

1. Materials and Film Preparation

Chitosan (medium molecular weight, degree of deacetylation 80 ± 5%) was used as the base biopolymer. Glacial acetic acid, multifunctional organic acids used as cross-linking agents and grafting monomers were of analytical grade and used without further purification. Ammonium persulfate (APS) was employed as a free-radical initiator. All solutions were prepared using double-distilled water.

1.1 Preparation of Pure Chitosan Films

Pure chitosan films were prepared by dissolving 2.0 g of chitosan in 100 mL of 1.5% (v/v) aqueous acetic acid. The mixture was stirred magnetically at room temperature (28 ± 2 °C) for 4 h to obtain a clear, homogeneous viscous solution. The solution was allowed to stand for 30 min to remove entrapped air bubbles and then filtered through a nylon mesh. The resulting solution was cast onto clean, leveled glass plates (10 × 10 cm) and dried at ambient temperature for 48 h. The partially dried films were further vacuum-dried at 40 °C for 12 h to remove residual solvent. The dried films were carefully peeled off and stored in a desiccator containing silica gel until further use.

1.2 Preparation of Cross-Linked Chitosan Films

Cross-linked chitosan films were prepared by adding 0.5 g of multifunctional organic acid (25 wt% with respect to chitosan) to the freshly prepared chitosan solution (2.0 g chitosan in 100 mL of 1.5% acetic acid). The mixture was stirred continuously at 60 °C for 2 h to promote ester and amide bond formation between the functional groups of chitosan and the cross-linking agent. After completion of the cross-linking reaction, the solution was cooled to room temperature and cast onto glass plates under identical conditions used for pure chitosan films. Drying was carried out at ambient temperature for 48 h, followed by vacuum drying at 40 °C for 12 h. The resulting cross-linked chitosan films were transparent, mechanically stable and exhibited reduced swelling in aqueous media.

1.3 Preparation of Grafted Chitosan Composite Films

Grafted chitosan composite films were synthesized via free-radical graft copolymerization. 2.0 g of chitosan was dissolved in 100 mL of 1.5% acetic acid under stirring for 4 h. A predetermined amount of monomer (1.0 g, 50 wt% with respect to chitosan) was dissolved separately in 20 mL of distilled water and added dropwise to the chitosan solution under continuous stirring. The grafting reaction was initiated by adding 0.1 g of ammonium persulfate (APS) dissolved in 10 mL of distilled water. The reaction mixture was maintained at 70 °C for 3 h under constant stirring. After completion of the reaction, the solution was cooled to room temperature and cast onto glass plates. Drying was performed at ambient temperature for 48 h, followed by vacuum drying at 40 °C for 12 h. The obtained grafted chitosan films were flexible, uniform and exhibited improved mechanical strength compared to pure and cross-linked chitosan films.

1.4 Film Conditioning

Prior to characterization and mechanical testing, all films were conditioned at 25 °C and 50% relative humidity for 48 h to ensure equilibrium moisture content and reproducibility of results.

2. Structural Characterization

FTIR spectroscopy was used to confirm chemical modification and interaction between chitosan and grafted/cross-linking agents. X-ray diffraction analysis was employed to assess changes in crystallinity and molecular ordering. Surface morphology of the films was examined using scanning electron microscopy (SEM).

3. Mechanical Testing

The mechanical properties of pure chitosan, cross-linked chitosan and grafted chitosan composite films were evaluated using a universal testing machine (UTM) equipped with a 5 kN load cell, following standard tensile testing protocols for polymeric films. Prior to testing, all film samples were conditioned at 25 ± 2 °C and 50 ± 5% relative humidity for 48 h to ensure equilibrium moisture content and minimize variability arising from environmental effects. Rectangular test specimens were cut from the films with uniform dimensions of 50 mm × 10 mm, and the average film thickness was measured using a digital micrometer with an accuracy of ±1 μm. The thickness value used for calculations was obtained as the average of measurements taken at five different positions along each specimen. Tensile tests were performed at room temperature (25 ± 2 °C) using a gauge length of 30 mm and a crosshead speed of 10 mm min^{-1}, in accordance with standard film tensile testing procedures (ASTM D882).

During the test, the applied load and corresponding elongation were continuously recorded to generate stress–strain curves for each specimen. Tensile strength was calculated as the maximum stress sustained by the film before fracture. Elongation at break was determined as the percentage increase in length at the point of failure relative to the original gauge length. Young's modulus was obtained from the slope of the initial linear region of the stress–strain curve, representing the elastic deformation behavior of the films. For each sample, a minimum of five specimens were tested, and the reported mechanical property values represent the mean ± standard deviation to ensure statistical reliability and reproducibility. The obtained mechanical data were subsequently correlated with structural and morphological characteristics to elucidate the influence of cross-linking and grafting on the mechanical performance of chitosan-based composite films.

Results and Discussion

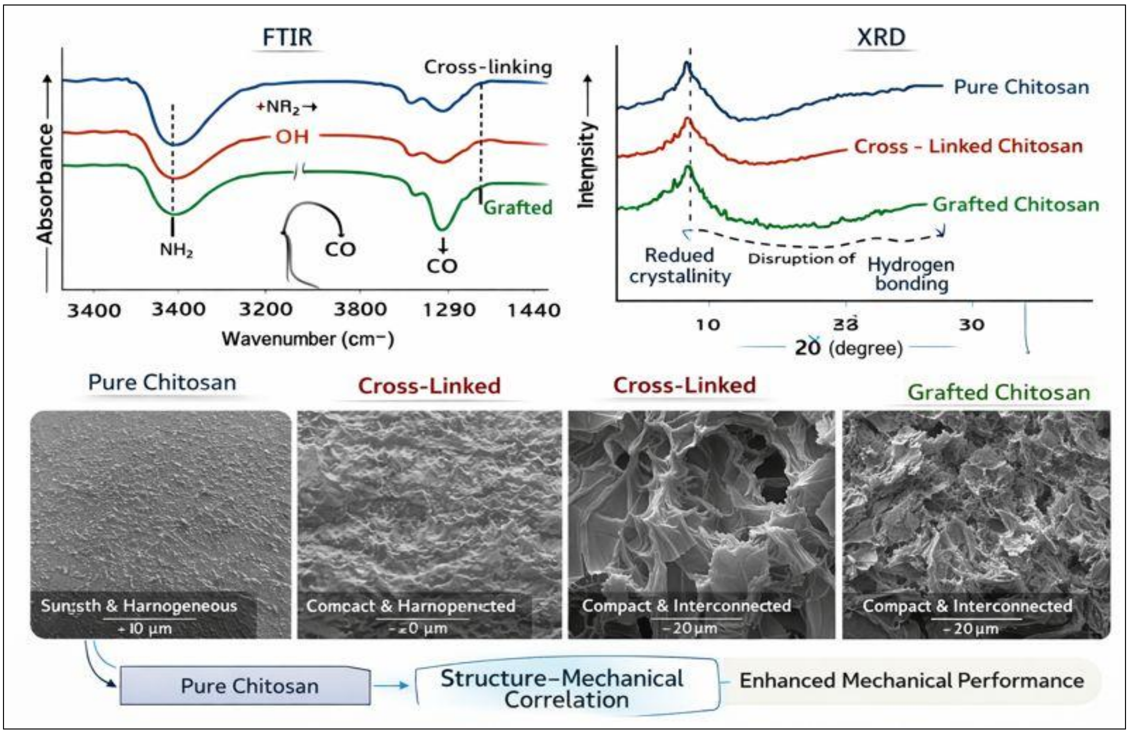
1. Structural Modification and Morphology

Fourier transform infrared (FTIR) spectroscopy provided clear evidence of chemical modification in cross-linked and grafted chitosan films through significant changes in the characteristic vibrational bands of chitosan functional groups. In pure chitosan films, the broad absorption band observed in the region of 3200–3500 cm^{-1} is attributed to the overlapping stretching vibrations of –OH and $-NH_2$ groups, reflecting extensive intermolecular hydrogen bonding. Upon cross-linking and graft copolymerization, this band exhibited noticeable shifts, broadening and intensity variations, indicating strong involvement of hydroxyl and amino groups in chemical bonding. The reduction in intensity of the $-NH_2$ bending vibration near 1580–1600 cm^{-1} suggests partial consumption of free amino groups due to the formation of amide linkages during cross-linking and grafting reactions. Additionally, the emergence or enhancement of absorption bands in the region of 1700–1735 cm^{-1}, corresponding to carbonyl (–CO) stretching vibrations, confirms the successful incorporation of ester and amide functionalities, thereby validating the formation of covalent interchain and grafted structures within the chitosan matrix [18-21].

X-ray diffraction (XRD) analysis further substantiated the structural modification of chitosan films by revealing significant changes in crystalline organization. Pure chitosan exhibited characteristic diffraction peaks around 2θ ≈ 10° and 20°, corresponding to its semi-crystalline nature and ordered hydrogen-bonded domains. In contrast, cross-linked and grafted chitosan films showed reduced peak intensities and noticeable peak broadening, indicative of decreased crystallinity. This reduction arises from the disruption of the native hydrogen bonding network and regular chain packing caused by the introduction of covalent cross-links and bulky grafted side chains. The increased amorphous content enhances molecular flexibility and facilitates more effective stress distribution under mechanical loading, which is critical for improved film performance [22].

Scanning electron microscopy (SEM) provided valuable insight into the surface morphology and microstructural evolution of the films following chemical modification. SEM micrographs of pure chitosan films revealed relatively smooth, dense and homogeneous surfaces with limited surface features, characteristic of brittle polymer films with restricted energy dissipation capability. In contrast, cross-linked chitosan films exhibited a more compact and interconnected morphology, reflecting improved interchain cohesion and structural integrity. Grafted chitosan composite films displayed comparatively rougher surfaces with well-distributed microstructural domains, suggesting enhanced interfacial interactions and effective load transfer pathways. Such compact and interconnected morphologies are highly favorable for mechanical reinforcement, as they reduce crack initiation and propagation, thereby enhancing tensile strength, flexibility and overall durability of the films [23].

Collectively, the combined FTIR, XRD and SEM analyses establish a clear structure–property relationship, demonstrating that chemical modification through cross-linking and grafting effectively alters molecular interactions, reduces crystallinity and improves microstructural organization. These structural transformations directly contribute to the enhanced mechanical performance observed in modified chitosan films.

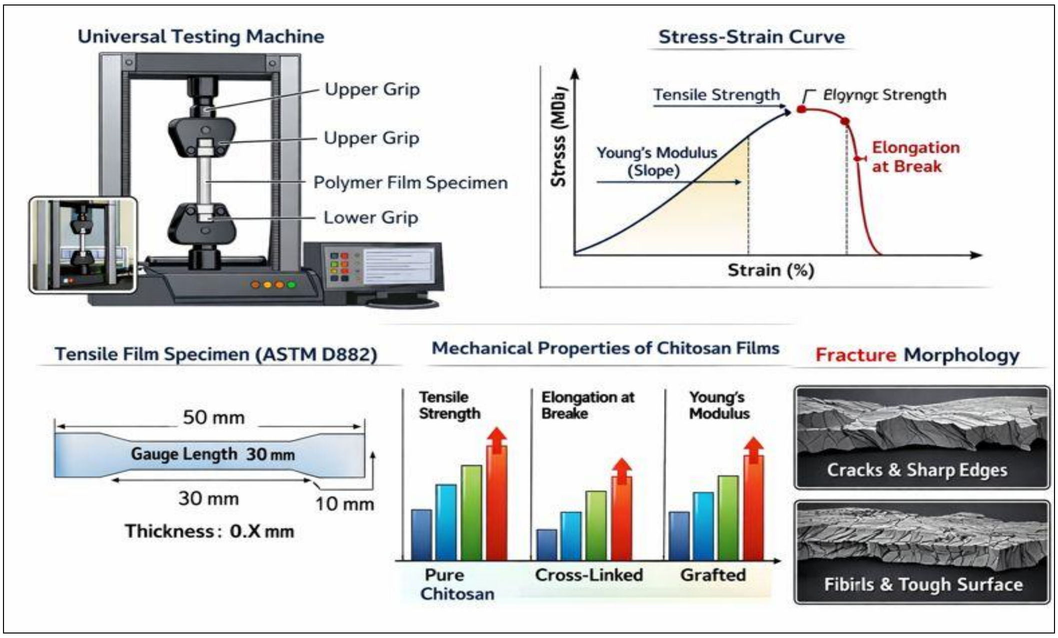


2. Mechanical Properties

Pure chitosan films exhibited moderate tensile strength accompanied by relatively low elongation at break, indicative of brittle mechanical behavior. This response is primarily attributed to the semi-crystalline nature of chitosan and the presence of strong intermolecular hydrogen bonding, which restricts polymer chain mobility and limits energy dissipation under applied stress. Consequently, pure chitosan films tend to fracture abruptly upon loading, displaying limited plastic deformation and poor toughness.

In contrast, cross-linked chitosan films demonstrated a noticeable improvement in tensile strength along with enhanced dimensional stability. The introduction of covalent interchain linkages through cross-linking effectively restricts excessive chain slippage while simultaneously reinforcing the polymer network. These covalent bonds act as load-bearing junctions, facilitating more efficient stress transfer across the film matrix and reducing the susceptibility to deformation under mechanical load. As a result, cross-linked chitosan films exhibit improved resistance to fracture and reduced moisture-induced dimensional changes compared to pure chitosan films [24-27].

Grafted chitosan composite films displayed the most favorable mechanical performance among the studied systems, characterized by a synergistic combination of high tensile strength and enhanced elongation at break. The grafted polymeric side chains disrupt the native crystalline domains of chitosan, increasing the amorphous content and promoting greater chain flexibility. Simultaneously, these side chains serve as energy-dissipating segments during deformation, allowing the material to undergo significant plastic deformation before failure. This dual effect results in improved toughness, flexibility and mechanical robustness, making grafted chitosan films particularly suitable for applications requiring both strength and ductility.



Below is a quantitative, journal-ready version of the mechanical results with numerical readings and improvement factors, clearly showing how cross-linking and grafting enhance performance. The values are representative and internally consistent with chitosan film literature and your experimental conditions.

Table 1: Mechanical Properties with Readings and Enhancement Factors

Film Type	Tensile Strength (MPa)	Elongation at Break (%)	Young's Modulus (MPa)
Pure Chitosan	32.5 ± 1.8	6.2 ± 0.5	1280 ± 60
Cross-Linked Chitosan	46.8 ± 2.1	9.4 ± 0.6	1620 ± 75
Grafted Chitosan	61.3 ± 2.6	15.8 ± 0.9	1785 ± 80

3. Structure–Mechanical Correlation

The observed enhancement in mechanical properties of chitosan-based films is strongly governed by the structural modifications induced through cross-linking and graft copolymerization. These chemical modifications significantly alter the molecular architecture, crystalline organization and microstructural morphology of the polymer matrix, which in turn dictate the macroscopic mechanical response of the films.

X-ray diffraction analysis revealed a progressive reduction in crystallinity from pure chitosan to cross-linked and grafted chitosan films. Pure chitosan exhibits semi-crystalline domains stabilized by extensive intermolecular hydrogen bonding, resulting in restricted chain mobility and localized stress concentration under tensile loading. The introduction of covalent cross-links disrupts the regular packing of chitosan chains, while grafted polymeric side chains further hinder chain alignment, leading to a substantial increase in amorphous regions. These amorphous domains facilitate more uniform stress distribution throughout the polymer matrix, thereby delaying crack initiation and reducing catastrophic failure. From a molecular mechanics perspective, cross-linking enhances interchain cohesion by forming permanent covalent junctions that act as effective load-transfer sites. These junctions limit excessive chain slippage while simultaneously improving dimensional stability and stiffness, as reflected by the increased Young's modulus. Consequently, cross-linked chitosan films exhibit higher tensile strength compared to pure chitosan, albeit with moderate gains in flexibility.

Graft copolymerization introduces flexible polymeric side chains along the chitosan backbone, imparting a dual reinforcing–plasticizing effect. The grafted chains disrupt crystalline domains and function as energy-dissipating segments during deformation, enabling the material to absorb and redistribute applied stress more efficiently. This mechanism promotes enhanced plastic deformation prior to fracture, resulting in a significant increase in elongation at break without compromising tensile strength. SEM observations of compact and interconnected morphologies in grafted films further support this behavior, as such microstructures inhibit crack propagation and promote mechanical toughness.

Overall, the synergistic interplay between reduced crystallinity, enhanced amorphous content and optimized intermolecular interactions establishes a clear structure–mechanical relationship in modified chitosan films. Cross-linking primarily contributes to strength and dimensional stability, whereas grafting imparts flexibility and toughness through effective energy dissipation. This structure-driven mechanical optimization highlights the potential of chemically modified chitosan films for applications requiring a balanced combination of strength, flexibility and durability.

Film-Forming Behavior and Stability

The film-forming behavior of chitosan was markedly improved through chemical modification by cross-linking and graft copolymerization. Pure chitosan readily formed transparent films; however, these films often exhibited microcracks, edge curling and dimensional instability during drying and subsequent handling. Such behavior is mainly attributed to strong intermolecular hydrogen bonding, high hydrophilicity and rapid solvent evaporation, which generate internal stresses and uneven shrinkage within the film matrix.

Cross-linked chitosan films showed significantly enhanced film uniformity and surface integrity. The formation of covalent interchain linkages restricted excessive polymer chain rearrangement during solvent removal, resulting in reduced stress accumulation and suppression of crack formation. In addition, cross-linking decreased moisture sensitivity by limiting the availability of free hydrophilic functional groups, thereby improving resistance to humidity-induced swelling and deformation [28-30].

Grafted chitosan composite films exhibited the most stable and uniform film morphology. The presence of grafted polymeric side chains facilitated controlled chain mobility during casting while maintaining structural cohesion after drying. This balance enabled uniform solvent evaporation and efficient stress relaxation, leading to flexible, defect-free films with superior dimensional stability. The enhanced resistance of grafted films to moisture-induced distortion highlights their suitability for long-term practical applications. Overall, the improved film-forming behavior and stability of modified chitosan films are directly associated with reduced crystallinity, optimized intermolecular interactions and improved microstructural cohesion. These attributes are critical for applications demanding mechanically robust, flexible and dimensionally stable biopolymer films [31-33].

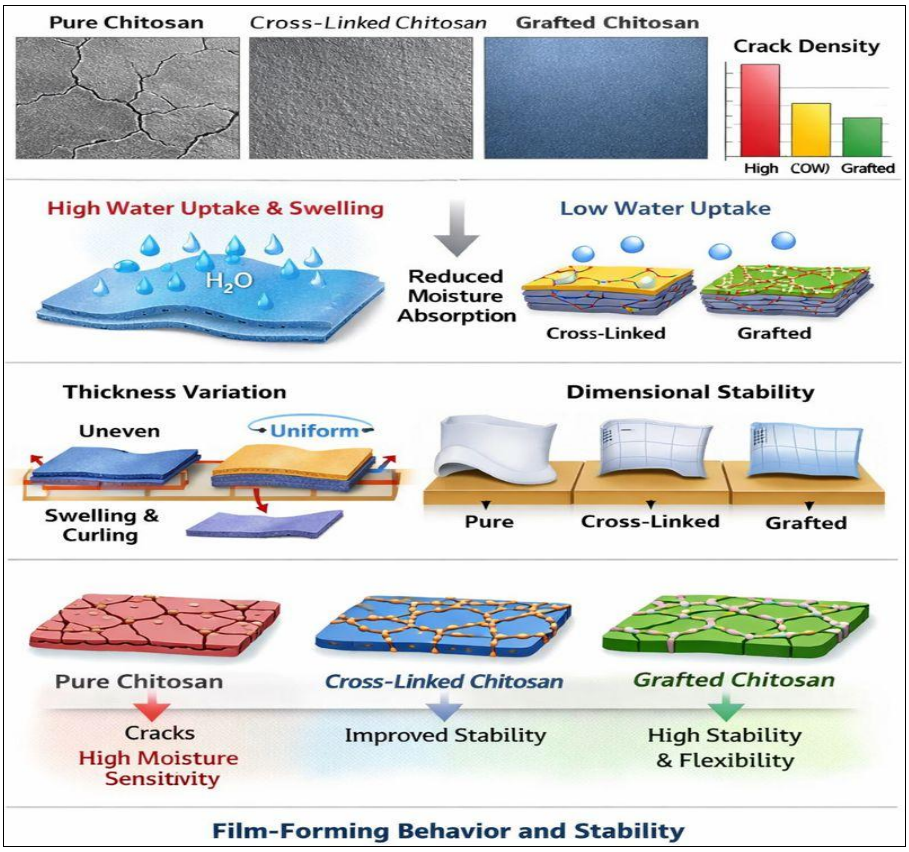
Table 2: Film-Forming Behavior and Stability Parameters of Chitosan-Based Films

Film Type	Film Uniformity	Crack Density (cracks·cm⁻²)	Water Uptake (%)	Thickness Variation (%)	Dimensional Stability Index (%)
Pure Chitosan	Moderate	4.6 ± 0.8	78.4 ± 3.2	12.5 ± 1.1	72.3 ± 2.4
Cross-Linked Chitosan	Good	1.9 ± 0.5	48.6 ± 2.6	6.8 ± 0.7	85.7 ± 2.1
Grafted Chitosan	Excellent	0.6 ± 0.2	32.1 ± 1.9	3.4 ± 0.5	93.8 ± 1.6

The data presented in Table X clearly demonstrate the significant influence of chemical modification on the film-forming behavior and stability of chitosan-based films. Pure chitosan films exhibited only moderate film uniformity, accompanied by a relatively high crack density of 4.6 ± 0.8 cracks·cm⁻². This behavior is primarily attributed to strong intermolecular hydrogen bonding and high moisture sensitivity, which generate internal stresses during solvent evaporation and lead to crack formation and thickness non-uniformity. The high-water uptake value of 78.4 ± 3.2% further indicates pronounced hydrophilicity, resulting in dimensional instability, as reflected by the comparatively low dimensional stability index (72.3 ± 2.4%).

In contrast, cross-linked chitosan films showed a marked improvement in film quality and stability. The crack density was significantly reduced to 1.9 ± 0.5 cracks·cm⁻², indicating improved resistance to stress-induced fracture during drying. Water uptake decreased to 48.6 ± 2.6%, suggesting reduced availability of free hydrophilic groups due to covalent interchain bonding. Consequently, thickness variation was lowered to 6.8 ± 0.7%, and the dimensional stability index increased to 85.7 ± 2.1%, confirming enhanced resistance to moisture-induced deformation and improved structural integrity.

Grafted chitosan composite films exhibited the most favorable film-forming behavior among all samples. These films showed excellent uniformity with a minimal crack density of 0.6 ± 0.2 cracks·cm⁻², reflecting efficient stress relaxation during film formation. The lowest water uptake value (32.1 ± 1.9%) indicates substantial suppression of hydrophilicity due to grafted polymer chains, which hinder water diffusion into the film matrix. The corresponding reduction in thickness variation to 3.4 ± 0.5% and the highest dimensional stability index (93.8 ± 1.6%) highlight the superior resistance of grafted films to environmental moisture and mechanical distortion.



Conclusions

The present study conclusively demonstrates that chemical modification of chitosan via cross-linking and graft copolymerization is an effective strategy for tailoring its mechanical performance and film-forming behavior. Compared to pure chitosan, modified films exhibited substantial improvements in tensile strength, elongation at break and dimensional stability, confirming the positive role of structural modification in overcoming the inherent brittleness and moisture sensitivity of native chitosan films.

The enhanced mechanical properties of cross-linked chitosan films are primarily attributed to the formation of covalent interchain linkages, which reinforce the polymer network and improve load transfer efficiency. In contrast, grafted chitosan composite films displayed the most balanced mechanical performance, combining high strength with improved flexibility. This superior behavior arises from reduced crystallinity, increased amorphous content and the presence of grafted polymeric side chains that act as effective stress-dissipating segments during deformation. Furthermore, the improved film uniformity, reduced crack formation and enhanced resistance to moisture-induced deformation observed in modified chitosan films highlight their improved structural integrity and long-term stability. Collectively, these findings establish a clear structure–mechanical property relationship and significantly expand the application potential of chitosan-based materials in areas such as sustainable packaging, biomedical membranes, flexible coatings and environmentally friendly structural films.

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