Chemically Plasticized Chitosan Films by Grafting Succinic Acid: Surface Roughness and Mechanical Properties

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Abstract: A useful alternative to conventional plastic packaging manufactured from fossil fuels is packaging constructed of a biodegradable polymer. The final qualities of the chitosan cast films heavily depend on the type of plasticizer used and how it is incorporated into the chitosan-based material. This work investigates the synthesis of chitosan chemically grafted with succinic acid to improve the physical and mechanical properties of the film. The cortex of local shrimp was used to make a chitosan extract with a molecular weight of 2.702×10^5 g/mole and a deacetylation level of 80%. The chemically plasticized chitosan film was characterized by FTIR. It was found that chitosan and grafted chitosan film spectra were different from each other, implying the true grafting of succinic acid had occurred and the main backbone of the chitosan structure was unaffected. An examination of the mechanical properties of the films revealed that the results of the tensile strength measurement showed an improvement in the strength and %elongation at break for the chitosan grafted with succinic acid cast film for packaging, which is one of the potential applications for biodegradable chitosan.

Keywords: Chitosan, Chemical plasticization, Films, Tensile strength, Surface roughness.

1. INTRODUCTION

A naturally occurring cationic copolymer with many potential applications is chitosan. It is a unique cationic polysaccharide with an exceptional affinity for many different surfaces, even when left unchanged. The energy crisis and environmental worries have recently raised interest in natural polymers. Due to chitosan's outstanding attributes, including nontoxicity, biodegradability, biocompatibility, antibacterial properties, and bio functional characteristics, chitosan-based materials have received a lot of attention in both the biomedical and food contact industries. Drug transporters, wound-healing agents, hemodialysis membranes, surgical dressing materials, and food packaging film are examples of such applications. For the active transport of chloride ions in an aqueous solution, chitosan membranes have been used [1].

In the plastics industry, plasticizers have long been a popular element [2]. To name a few of its numerous applications, are automobiles, packaging, membranes, consumer goods, medicines, structures, and construction [3]. However, the industry is moving away from phthalate-based plasticizers and toward bio-based plasticizers due to environmental and health concerns [4, 5]. Low cost per volume, low volatility and diffusivity, low specific gravity, and good miscibility, strong intermolecular interactions between the plasticizer and the polymer resin are all desired properties of plasticizers [6, 7]. A well-plasticized product should be flexible and have a low elastic modulus and good tensile elongation but low tensile strength [8]. Physical blending is a conventional way of modifying chitosan to suit different purposes.

Chitosan is a natural polymer that is a deacetylated product of chitin, and it is a repeated unit mainly composed of α-(1,4)-linked 2-deoxy-2-amino-D-glucopyranose units. Chitosan can experience numerous responses, for example, etherification, esterification, and cross-linking. It is chemically defined as a copolymer of α-(1, 4) glucosamine ($C_6H_{11}O_4N$) having a different number of N-acetyl groups [9]. The amino groups of Chitosan have a pKa of 6.5 (the typical range is 6.2–6.8), giving the molecule an overall positive charge and solubility in dilute solutions of most of the organic acids, including acetic, malic, citric, tartaric, glycolic, and ascorbic acid solutions, with a charge density depending on the value of pH and the percentage of the degree of deacetylation [10].

The chitosan films are rigid and require plasticizers to reduce the frictional forces between the adjacent polymer chains. The physical and structural properties of chitosan-based films for food packing, biomedical, and other applications have been studied frequently [11, 12]. Plasticizers occupy the intermolecular gaps between polymer chains when they move away from each other, reducing chain retraction and increasing free volume, allowing polymer chains to move more freely [13]. The hydrogen bonding interaction between chitosan and plasticizer [14, 15] controls the physical and mechanical properties of the films.

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The goal of this study was to show how easily and effectively plasticized chitosan biodegradable composite films may be prepared for application in the packaging industry. The main characteristics to be taken into account are mechanical strength, surface homogeneity, and flexibility [16-18]. Additionally, this research investigated the impact of introducing the plasticizer chemically grafted rather than physically blended, which may be considered a new approach for getting a cast chitosan film suitable for packaging. green plasticizer as opposed to plasticizers that are offered for sale. The characterization of these films revealed their unique properties and competitiveness with commercially available physical synthetic plastics [19]. These conditions were tried for chemically grafted succinic acid plasticized chitosan extracted from the local shrimp cortex in this study.

2. EXPERIMENTAL

2.1. Materials

Chitosan was obtained by the deacetylation process of chitin extracted from local shrimp cortex waste as described in the literature [20, 21]. It had a viscosity average molecular weight of 2.702×10^5 g/mole as determined by the viscosity technique and a deacetylation degree of 80%. Succinic acid and acetic acid were purchased from Sigma-Aldrich Company and utilized without further treatment.

2.2. Instruments

FTIR-8101M Shimadzu spectrometer in the 4000–500 cm^{-1} range was used to investigate the chemical structures of the unplasticized and plasticized chitosan films.

Thin films of chitosan were studied using an Atomic Force Microscope model AA3000 Scanning Probe Microscope/Angstrom Advanced Inc. to assess topography and surface roughness for unplasticized and chemically plasticized chitosan surfaces (USA).

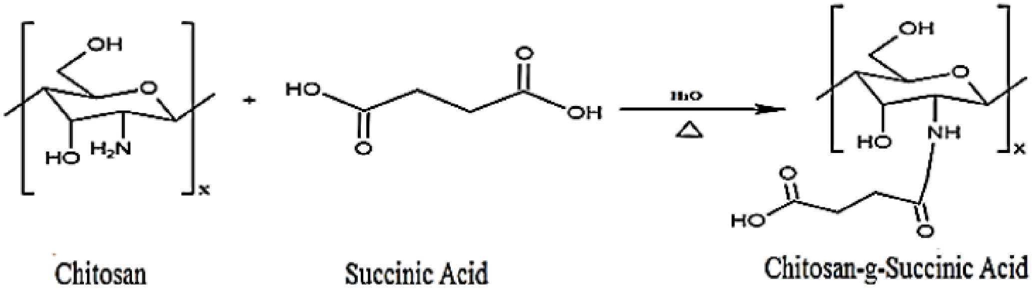
The mechanical properties (tensile strength, Young's modulus, and %elongation at break) of the unplasticized chitosan and their plasticizer blend films were measured in the tensile mode (speed 5 mm min^{-1}) with a BTI-FR2.5TN.D14 (ZwickRoell, Germany) mechanical testing machine. The ASTM D882-10 Standard Test Method for Tensile Properties of Thin Plastic Sheeting and Films was used to determine the mechanical properties of the chemically plasticized and unplasticized chitosan films in the form of stripes of 20 x 2 mm. This test method covers the determination of tensile properties of plastics in the form of thin sheeting and films (less than 1.0 mm (0.04 in.) in thickness). The plasticized and unplasticized chitosan films were examined in dry states.

2.3. Preparation of Chitosan-Grafted-Succinic Acid

Grafting succinic acid onto chitosan was carried out in a three-neck round bottom flask equipped with a condenser, magnetic stirrer, and thermometer to produce chitosan-g-succinic acid. The round flask was filled with 50 mL of water, 5 g of Chitosan, and 5 g of succinic acid. The mixture was heated to a refluxed temperature for two hours while being magnetically swirled. It was then given a good shake and allowed to cool to room temperature [22]. The Chitosan-g-succinic acid was filtered and repeatedly cleaned with acetone and deionized water, respectively, before drying in the vacuum desiccator. The equations for chemical grafting are shown in Scheme **1**.

2.4. Chitosan Films Preparation

The cast films of unplasticized chitosan and its plasticized blends were prepared using the solvent evaporation method by dissolving 1 g of chitosan in 100 ml of 2% (v/v) acetic acid solution under stirring at ambient temperature. Then, it was poured into a



Scheme 1: Grafting reaction of chitosan by succinic acid.

leveled Petri dish of 50 mm in diameter. The film was removed from the dish, and it was dried for 12 hours at 45°C, and then stored before the determination of its structural, physical, and mechanical properties [23]. The experimental flow sheet is illustrated in Figure **1**.

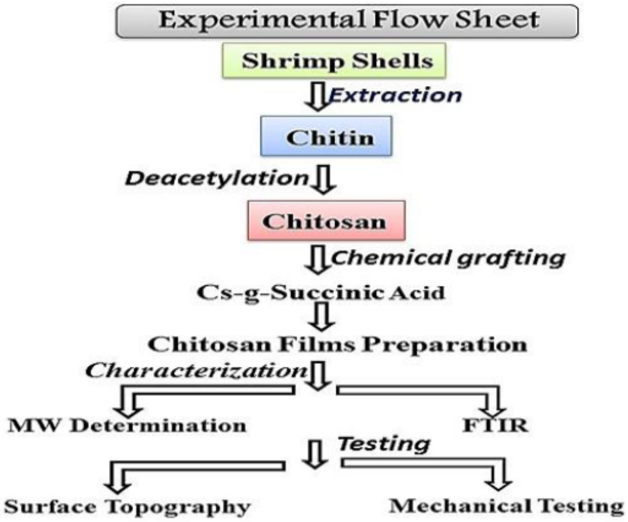


Figure 1: The experimental flow chart.

3. RESULTS AND DISCUSSION

3.1. Molecular Weight of Extracted Chitosan

Glucosamine and N-acetylglucosamine repeating units are the building blocks of the polymer chitosan. The molecular weight of chitosan is determined by the length of these chitosan chains. Since viscosity and molecular weight have a logarithmic relationship, viscosity can be used to define molecular weight [24]. When the viscosity of polymer solutions in organic solvents is evaluated, the result is proportional to the molecular mass of the polymer, which increases with increasing viscosity. The Mark-Houwink-Sakurada equations are known to represent the intrinsic viscosity [η] as a function of the viscosity average molecular weight Mυ [25].

$$[\eta] = k\,M\boldsymbol{\upsilon}^{\alpha}$$

The viscosity was measured by dissolving 0.5 g of chitosan in 100 ml of an equal volume of the buffer solution (0.15M ammonium acetate and 0.2M acetic acid) at 25 °C, where $k = 9.66\times10^{-5}$ dm^3g^{-1} and $\alpha = 0.742$ for chitosan, and depending on the solvent and temperature employed [26].

The linear plot of lowered viscosity against concentration for chitosan in the solubilizing aqueous mixture of ammonium acetate and acetic acid is shown in Figure **2**. The inherent viscosity is represented by the slope, which was found to be 1.036. The Mark-Houwink-Sakurada equation calculates chitosan's average molecular weight of 2.702×10^{5} g/mole. The results found are consistent with results reported in the literature [27, 28].

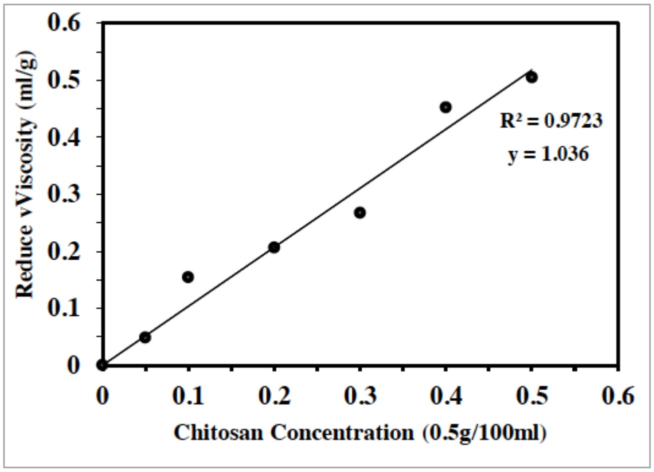


Figure 2: Linear relationship between reduced viscosity and various chitosan concentrations at 25°C.

3.2. Chitosan and Chemically Plasticized Chitosan Films FTIR Characterization

The films were easy to peel from the cast Petra dish and also simple to handle and treat further. FTIR spectra of the chitosan and chemically plasticized chitosan were examined and characterized.

3.2.1. FTIR Characterization of Unplasticized Chitosan Films

Figure **3a** displays the spectrum of the unplasticized chitosan film produced by casting a 2% acetic acid solution after it was peeled off the Petri dish and before storage. The distinctive features of the chitosan spectrum in this study are comparable to those in earlier reports [29, 30]. The usual polymer base-state peaks of pure Cs, such as those at 1654 cm^{-1} caused by C=O stretching (amide I) O=C-NHR, 1562 cm^{-1} due to NH bending (amide II) (NH_2), and 1037 cm^{-1} due to vibration of C-O groups [31-34]. All of the films under study had peaks between 3610 and 3000 cm^{-1}, which correspond to the stretching vibration of free hydroxyl and the asymmetrical and symmetrical stretching of the N-H bonds in the amino groups [35]. The bands at 2912 and 2843 cm^{-1} indicate the vibrations of the aliphatic C-H [36].

3.2.2. FTIR Characterization of Chemically Modified Chitosan Film

The powerful reactive basic amino groups that can be employed as a center graft to attach diverse chemicals to the chitosan backbone are what give the substance its distinctive chemical structure [37, 38].

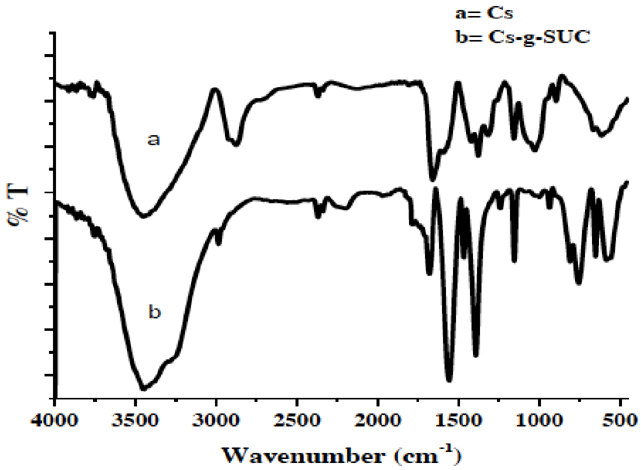


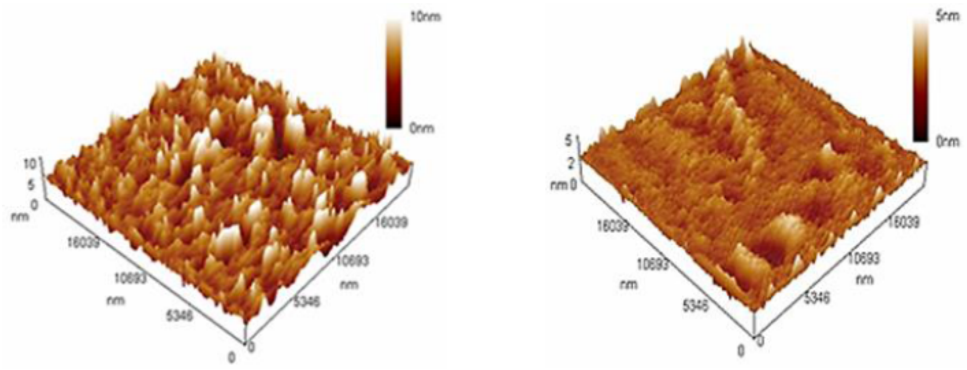
Figure 3: FTIR spectrum of Cs-succinic acid films.

FT-IR measurements were carried out to track the impact of chemical alterations to the chitosan structure upon interaction with grafted succinic acid. Figure **3b** shows the FTIR spectral features of the succinic acid solution cast film-modified chitosan surface. There were not many noticeable alterations in the spectrum [39]. The overlapping -OH and –NH_2 stretching bands caused a broad, significant absorption in the range of 3529–2820 cm^{-1}. The presence of an asymmetric N-H bend and an asymmetric COO-stretching corresponds to absorption at 1643 and 1577 cm^{-1}, respectively. Observations at 1500 and 1381 cm^{-1} were attributable to a symmetric N-H bend. The symmetric 1157 and 898 cm^{-1} seen in the chitosan-g-succinic acid spectrum was comparable to the native chitosan spectrum, indicating that the primary backbone of the chitosan structure was unaltered.

3.3. Topography and Surface Roughness

Atomic force microscopy was used to assess the surface concentration of unplasticized and plasticized chitosan particles in the first layer for a high-resolution topographical examination (AFM). Figure **4** displays the AFM topography images of UPCs that have been chemically plasticized with grafted succinic acid and have a molecular weight of 2.702×10^5 g/mole.

The root mean square roughness, which is derived by squaring each height value in the dataset and then taking the square root of the mean [40], is one of the dispersion factors for assessing surface roughness. One of the many characteristics that may be used to observe topography is the root mean square RMS roughness of a surface, which influences the surface properties of components and thin films from the very beginning [23, 41]. In general, surface roughness can help assess the film's quality and provide the appropriate film smoothness for a variety of applications. It is determined by first calculating the square root of the mean and then multiplying it by each height value in the dataset [40]. Figure **5** shows that their thin films computed RMS roughness was 1.6 nm and 0.378 nm, respectively. The magnitude of the decrease in RMS roughness of the UFCs thin film can be attributed to the Cs-g-SUC film appearing to be more homogeneous. The high contact forces between the cross-linked chitosan chains that result in high chain alignments and a smoothing mechanism involving surface diffusion [42] may be the reason for



Unplasticized chitosan

Chitosan-g-Succinic acid

Figure 4: AFM topography images of unplasticized chitosan and chitosan-g-succinic acid.

the homogeneity. This finding implies that chitosan has excellent chemical plasticization properties.

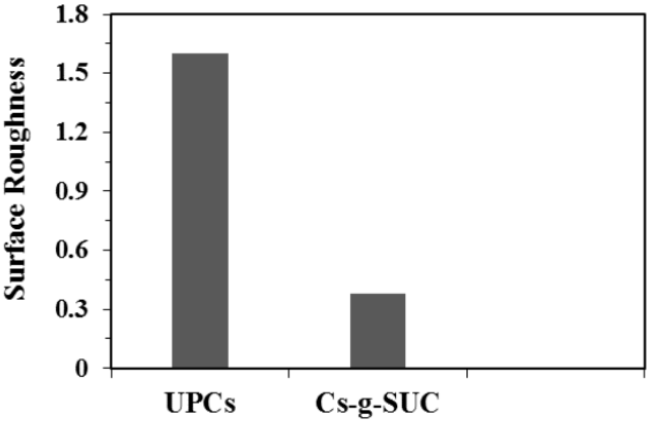


Figure 5: The effect of chemically grafted chitosan with succinic acid on the thin film surface roughness of based chitosan.

The AFM images visualization of the maximum height of nanoparticles of chitosan and its chemically plasticized films with succinic acid plasticizer were about 0–10 and 0–5nm based on the RMS roughness, respectively, in Figure **4**. This finding agrees with the FTIR discussed above, showing the existence of the grafted succinic acid with chitosan in the thin film.

3.4. Chitosan-g-Succinic Acid Mechanical Properties

Especially for single-use packaging when the material is stretched during use owing to continuous wear and tear, flexibility is an important property of packaging materials. The mechanical properties of synthetic bioplastics must be precisely studied to define their range of uses. Polymer film's strength and elasticity can be determined through mechanical testing [43]. These metrics are all closely correlated with the chemical composition and homogeneity of the material. The elastic modulus measures the film stiffness, the film strength is represented by the elastic modulus, and the percent elongation at break represents the material's deformation capacity [44, 45].

The mechanical parameters, *i.e.*, tensile strength, Young modulus, and %elongation at break are shown in Figure **6**. They are the most important mechanical properties studied for succinic acid chemically grafted onto the chitosan polymeric chain. As it can be seen from the values of Cs-g-SUC film, it had higher tensile strength and a higher %elongation at break than unplasticized chitosan film, and a lower Young modulus (Young modulus for unplasticized chitosan is about 228 MPa) [46]. This implies a less stiff film was prepared from chemically grafted chitosan. However, the improvement in the mechanical properties of chitosan films may be ascribed to the presence of covalent crosslinking between succinic acid and chitosan. In particular, the covalently cross-linked films had significantly higher tensile strength and strain than the ironically cross-linked films, which means that a more resistant and at the same time more flexible network results from the formation of amide bonds [47].

4. CONCLUSIONS

This work addresses the chemical production of chitosan grafted with succinic acid to enhance the

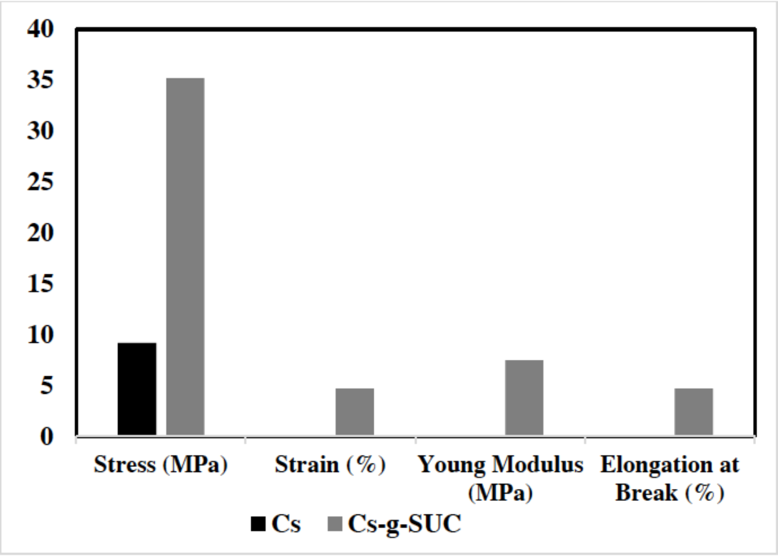


Figure 6: The effect of chemically grafted succinic acid on the tensile properties of the prepared films.

physical and mechanical properties of the film. It was found that the film FTIR spectra of chitosan and grafted chitosan were distinct from one another, indicating that real succinic acid grafting had taken place and the primary backbone of the chitosan structure remained unaltered. One of the potential uses for biodegradable chitosan was found when the mechanical properties of the films were examined. The findings of the tensile strength measurement showed an increase in the strength and %elongation at break and a tremendous decrease in the film stiffness for chitosan grafted with succinic acid cast film. The formation of cross-linking between chitosan molecules and the improvement of the physical properties of the films, both of which are significant in some applications, are potential benefits of employing chemically grafted succinic acid. This gives the impression that it can be used as a biodegradable and edible film to preserve foods.

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