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Preparation and properties of physically plasticized chitosan films

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ABSTRACT

Food packaging prevents conditions that can reduce food quality and shelf life. This leads to environmental pollution because it does not degrade naturally. The food packaging industry is increasingly adopting biodegradable polymer films as an alternative to plastic packaging. They are receiving great attention and are more suitable for food applications because they do not need to be eliminated as solid waste, which is why the industry has recently begun to pay more attention to food packaging films derived from natural chitosan polymers to replace traditional synthetic polymers. Shrimp cortex was used to extract the chitosan using the casting procedure; It was plasticized with different ratios of polyvinyl alcohol (PVC), namely 1:1, 1:2, 1:3, and 1:4 to create plasticized chitosan films from its solution in 2% acetic acid by casting technique. All films prepared were examined by infrared spectroscopy (FT-IR) and were found to be comparable to the original chitosan spectrum, indicating that the basic composition of the basic polymeric chitosan chains was not affected by the addition of various ratios of PVC plasticizer. Unlike unplasticized chitosan films. The results of the mechanical tensile strength measurements of plasticized chitosan films showed an improvement in tensile strength, % elongation at breakage, and a decrease in the Young modulus, which means that less rigid films were obtained, with an enhancement in their optical properties accompanying this by decreasing the opacity from 85 for unplasticized chitosan to about 3 for plasticized chitosan films. The addition of plasticizer to chitosan was also found to increase the solubility of prepared plasticized chitosan films in water and reached 100% for 1:3 chitosan:PVA in contrast to the unplasticized chitosan polymer, which is insoluble in water.

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1. Introduction

Plastic has been used in daily life since 1950, and the packaging sector is the main user of plastics, as it is an important barrier to conditions that can reduce food quality and shelf life [1]. An estimated 320 million tons of plastic are used worldwide for food packaging and a large part of this material is in landfills [2]. This is expanding due to the accumulation of plastic garbage from many different sources.

The most vulnerable natural resources from these nonbiodegradable plastics are water bodies and the creatures that live in them, and burning them causes the release of polluting gases into the air. Therefore, interest has recently increased in natural polymers that are considered an alternative to plastic packaging, which has recently received great attention mainly for environmental reasons such as chitin, chitosan, pectin, and polysaccharides due to their importance on the one hand and ecological concerns for unnatural or manufactured polymers on the other, in addition to their ability to form films and membranes with different shapes and sizes in combination with other additives [3]. Chitosan is of great importance among these biopolymers due to its widespread use in the food industry as it is odorless and nontoxic and for its biocompatibility and biodegradability [4,5]. In this context, chitosan and its derivatives have shown new light for various applications due to their chain structures and enormous potential for new important applications [6,7].

Due to the great importance of food packaging films, researchers have been looking for the best source of these envelopes, relying on the natural source available directly or indirectly after simple modifications using additives [8]. This source also has interesting antibacterial properties, as these films can protect foods against fungal degradation and change the environment of fresh fruits and other foods [9].

It should be noted here that the manufacture of chitosan films requires the addition of plasticizers because the chitosan film for a unit is fragile and easily cut or breakable and does not have durability and an appropriate coefficient of expansion when cutting, as plasticizers reduce pressures between molecules and enhance the movement of the polymer chain, improving the flexibility of the film and its ability to expand and prevent cracking and breakage during use or storage, as plasticizers occupy the gaps between the chitosan chains when they move away from each other, allowing the polymer chains to move more freely [9]. Therefore, we focused on the use of chitosan extracted from shrimp chitosan husks and plasticizing it with polyvinyl alcohol and biopolymer to facilitate the manufacturing process of forming them into environmentally friendly food packaging films.

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(a) (b)

Figure 1. (a) Unplasticized and (b) plasticized (CS:PVA, 1:3) chitosan caste films.

2. Experimental

2.1. Materials

Shrimp shell waste was obtained from the local market. It was used to extract chitosan by the chitin deacetylation process, as described in the literature [10]. It had a degree of deacetylation of 81% measured by the absorption method using infrared spectroscopy of a type (JASCO 4200) instrument [6,11], and a molecular weight average of 2.702×10^5 g/mol, as determined by the viscosity technique [12]. Polyvinyl alcohol (PVA) was used as a plasticizer, and acetic acid was used as a solvent. They were purchased from Sigma-Aldrich and used without further treatment.

2.2. Methods

2.2.1. Physical blending

Physical mixing is the most common method to prepare a chitosan plasticizer solution before making plasticized chitosan films [13]. Film formation preparation was carried out first by organizing a 1% chitosan solution (w/w) by solubilization in a 2% (v/v) acetic acid solution stirred for 30 minutes and drying for 24 hours, as described in the literature [14,15]. The PVA plasticizer was dissolved in water with a concentration of 1% (w/w) and mixed with a chitosan solution in four different ratios: 1:1, 1:2, 1:3, and 1:4 [16,17].

2.2.2. Formation and appearance of cast films

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 a 2% (*v*:*v*) acetic acid solution under stirring at room temperature. Then it was poured into a leveled Petri dish measuring 50 mm in diameter. The film was removed from the dish and dried for 12 hours at 45 °C [17]. The transparent films were easy to peel from and they were dried for 12 hours at 45 °C and then stored before FTIR examination and further studies. Figure 1 shows the obtained peeled chitosan films obtained.

2.2.3. Thickness of films

A SHAHE micrometer was used to measure the thickness of the films (SHAHE, China). The mean value was derived after testing the samples at 10 random points.

2.2.4. Optical characteristics

The optical properties of thin films were examined with a UV-vis spectrophotometer using Shimadzu UV 1800/Japan. Each film specimen was cut into a rectangular piece and placed directly in a UV-vis spectrophotometer test cell, and measurements were performed using air as the reference. A spectrum of each film was obtained at wavelengths between 200 and 800 nm. The results have been expressed as percentage transmittance. The measurements were done in triplicate, and the average of the three spectra was calculated. Transparency at 600 nm (T600) was obtained using Equation 1 [14],

$$T_{600} = \frac{-\log\%T}{b} \qquad (1)$$

where %T is the percentage transmittance and b is the thickness of the film (mm). The opacity of the films was calculated using Equation 2 [14],

$$\text{Opacity} = \text{Absorbance at 500 nm} \times \text{film thickness (mm)} \qquad (2)$$

2.2.5. Film solubility

The amount of dry matter in the film that dissolves in water is used to calculate the solubility of the film. The solubility of the films was evaluated using techniques previously described with some changes [16,18]. The films were divided into 2 cm by 2 cm squares and dried completely before storing. The films were repeatedly weighed until a stable weight was reached for the fully dried films; this weight was then used as the initial dry weight. The films were stirred at 25 °C for 24 hours while submerged in 50 mL of deionized water in a beaker. After being removed from the beakers, the films were dried at 105 ° C until they attained a constant weight. This quantity served as the final dry weight. The solubility percent was calculated using Equation 3 [19].

$$\text{Water solubility (\%)} = \frac{\text{Initial dry weight} - \text{Final dry weight}}{\text{Initial dry weight}} \times 100 \qquad (3)$$

2.2.6. Mechanical measurements

The ASTM D882-10 Standard test method for tensile properties of thin plastic sheeting and films was used to determine the mechanical properties of plasticized and unplasticized chitosan films in the form of stripes of 20 × 2 mm using a BTI-FR2. 5TN.D14 mechanical testing machine. This test method covers the determination of the tensile properties of plastics in the form of thin sheets and films (less than 1.0 mm (0.04 in) in thickness).

3. Results and discussion

3.1. FTIR characterization

3.1.1. Unplasticized chitosan film

Chitosan is a special cationic polysaccharide with a great affinity for various surfaces and exceptional complexion properties, even when left unaltered [11].

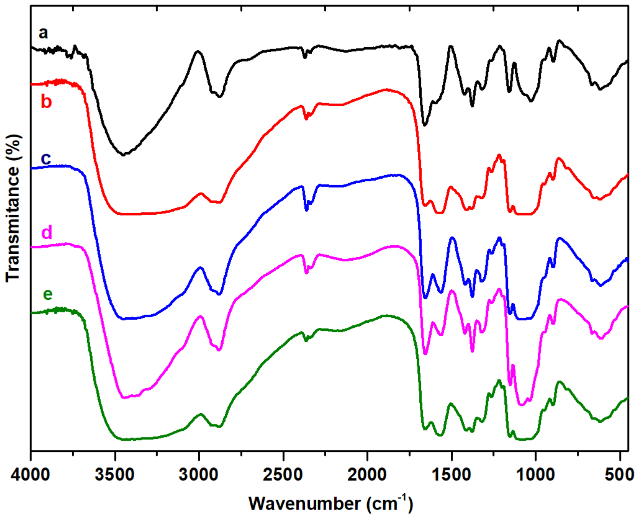

Figure 2. FTIR spectra of plasticized and unplasticized chitosan films.

Physical blending and chemical modification by grafting copolymerization are useful and significant techniques for modifying chitosan for different purposes [5]. The physical and structural properties of chitosan films for food packaging, biomedical applications, and other uses have been studied frequently [20,21].

The spectrum of the unplasticized chitosan film cast from a 2% acetic acid solution, after being peeled off the Petri dish and before storage, is shown in Figure 1a. It shows a strong and broad band in the range 3610 to 3000 cm^{-1}, which is attributed to hydrogen-bonded OH and hydrogen-bonded NH stretching vibrations, aliphatic C-H stretching vibrations appear at 2912 and 2843 cm^{-1}, these results agree well with the values reported in the literature [22,23]. The band that appeared at 1654 cm^{-1} is attributed to the asymmetric carbonyl (C=O) stretching of the secondary amide (Amide I band), while that at 1562 cm^{-1} is related to the NH bending vibration of the amine (N-H_2) that overlaps the amide vibration (Amide II band). The peaks at 1429 and 1367 cm^{-1} belong to the asymmetric C-H bending vibration of the CH_2OH group and the symmetric deformation of CH_3 in the acetamide group ($NHCOCH_3$), respectively. The band at 1320 cm^{-1} belongs to the CN stretching vibrations in the secondary amide group HNCO (Amide III band). The weak band at 1253 cm^{-1} is the result of the interaction between NH bending and CN stretching [24]. The bands at 1162, 1072, and 1037 cm^{-1} are assigned to antisymmetric C-O-C, N-H stretching vibrations, and C-O stretching vibrations in the secondary and primary OH groups, respectively [25].

3.1.2. Plasticized chitosan film

The FTIR spectra of the chitosan-PVA films were examined in light of the individual contributions made by various concentrations of PVA to the physical plasticizing of chitosan and the modifications brought about at the structural and molecular levels. Regardless of the ratio, samples with and without plasticizers were investigated by focusing on spectrum windows that show hydrogen bond interactions.

The FTIR spectra of chitosan and PVA-blend films with different percentages of PVA are shown in Figures 2b-2e. As noted, the band resulting from the NH bending (amide II) of chitosan at 1577 cm^{-1} had less intensity as the concentration of PVA in the blended films increased from 1:0 percent to 1:4. Furthermore, as the concentration of PVA increased, an increase in the intensity of the CH group was observed at about 2912-2939 cm^{-1} [26,27]. With the addition of more PVA to the mix, there was also a notable shift from a chitosan peak at 1033 cm^{-1} to a higher wavenumber of 1076 cm^{-1} in the different ratios of the Cs: PVA blend, Figures 2a and 2b.

In the spectra of pure chitosan, the C-C stretching band at 852 cm^{-1} was very weak. In contrast, the strength of this band grew as the PVA concentration increased in the Cs: PVA blended films. This may be because the reflection of physical blending and chemical interactions causes changes in typical spectra peaks when two or more polymers are combined [28,29]. These findings suggest that chitosan and PVA are miscible, which is more likely the result of intermolecular hydrogen bonds forming between the amino and hydroxyl groups in chitosan and the hydroxyl groups in PVA.

3.2. Optical properties

The shelf life of food is important and can be influenced by various environmental factors. It can be protected and increased by using the right packaging materials. The properties of the film, which depend on the type of material used and the processing parameters used, in turn, define the applications [30]. As one of the intended features of packing material, food should also be shielded from the effects of light, particularly UV radiation. Therefore, transparency is an important characteristic and is commonly measured in food packaging films. Mixed definitions of film transparency and opacity have been used in the literature and this work evaluates the optical properties of the prepared cast plasticized and unplasticized films.

The light transmission properties were investigated at wavelengths of 200 to 800 nm [9]. The results obtained are shown in Table 1, where the table shows the effect of the addition of plasticizer to chitosan on the thickness and transparency of the film compared to the unplasticized chitosan film, and it can be concluded that the plasticized chitosan films showed higher transparency values (lower opacity) than the non-plasticized chitosan film. However, it may be worth mentioning that our results are in good agreement with the results reported in the literature by Kanatt *et al.* [14]. These results are significant because they give an impression of the desired characteristics of food packaging material and protect them from the surrounding environment, without forgetting to show the refreshment of the packed food under protection at the same time.

Table 1. Transparency and opacity of plasticized and unplasticized chitosan films.

Type of the film	Ratio	Thickness (mm)	Transparency	Opacity ($\times 10^{-4}$)
Unplasticized chitosan	1:0	0.226	0.110	85.00
Chitosan:PVA	1:1	0.034	0.147	3.06
	1:2	0.039	0.153	2.80
	1:3	0.042	0.164	3.78
	1:4	0.035	0.285	4.90

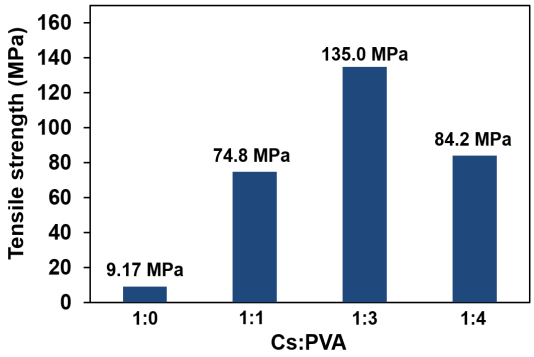

Figure 3. Effect of PVA ratios on the tensile strength of plasticized unfractionated chitosan.

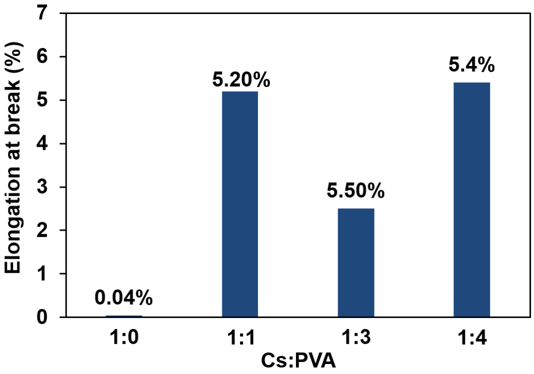

Figure 4. The effect of the PVA ratios on the % elongation at the break of plasticized unfractionated chitosan.

3.3. Mechanical properties

Flexibility is a crucial quality for plastics, particularly for single-use packaging, when the material is stretched during use due to continuous wear and tear. Mechanical testing indicates the strength and elasticity of polymer films [31]. The maximum tensile strength at break, the elongation at break (%), and the elastic or Young's modulus are the most common parameters that describe the mechanical properties of the chitosan-plasticizer blend films and are all strongly related to the homogeneity and chemical composition of the mixed material. The strength of the film is represented by the tensile strength, the deformation capacity of the material is represented by the elongation at break, and the film stiffness is measured by the elastic modulus [32,33]. The mechanical characteristics, structural integrity, and flexibility of chitosan-based plasticized films are projected to increase [34].

The two-component polymers should interact intermolecularly during blending, strengthening the blend's mechanical properties. Mechanical testing can measure the elasticity and strength of polymer films [35,36]. Figures 3 and 4 illustrate the tensile strength and percent elongation at the break of Cs: PVA blended films with various PVA ratios, respectively. Almost all Cs: PVA blended films had higher values than the UP and UFC films, which showed a tensile strength of 9.17 MPa (Figure 3). The plasticized chitosan film had a maximum tensile strength value of 135 MPa with ratios of 1: 3 of Cs: PVA, followed by a decrease in tensile strength as the PVA ratio increased, *i.e.* 1:4 of Cs: PVA. The compact film structure, which resulted from the good blending of the base polymers at approximately 75% PVA in the blended film, could explain the improvement in tensile strength. This demonstrates that blending improves strength. This improvement in tensile strength could be due to the interaction between the OH and NH_2 groups originating from chitosan and the OH groups of PVA [37,38]. These results are in good agreement with those published by El-Hefian *et al.* [39], who found that the combination of Cs with PVA improved strength by increasing the percentage of PVA by 40%, which is very close to the ratio of Cs: PVA 1: 3 used by Hyder and Chen [40], and Bahrami *et al.* [41], also reported similar behavior of the blended films of PVA / Cs concerning tensile strength.

The measured Young's modules for all blended films with lower moduli reflected this in the same way as shown in Figure 5. This means that the mixed films had better flexibility compared to pure unplasticized chitosan [42]. According to the literature, chitosan coupled with PVA behaved similarly; for example, Lusiana *et al*. discovered that the plasticized film of Cs/PVA made with acetic acid had a higher tensile strength and a much lower Young modulus than pure chitosan [43].

3.4. Solubility of chitosan films

Chitosan is a semicrystalline biopolymer that is insoluble in most solvents, including water, alkali, or alkaline solutions (pH ~ 7) and typical organic solvents, due to its hard crystalline structure [44].

Table 2. Solubility of plasticized and unplasticized chitosan.

Type of the film	Ratio	24 hour (%)	48 hour (%)	72 hour (%)	96 hour (%)
Unplasticized chitosan	1:0	2.1	2.8	3.3	3.9
Chitosan:PVA	1:3	100	0	0	0

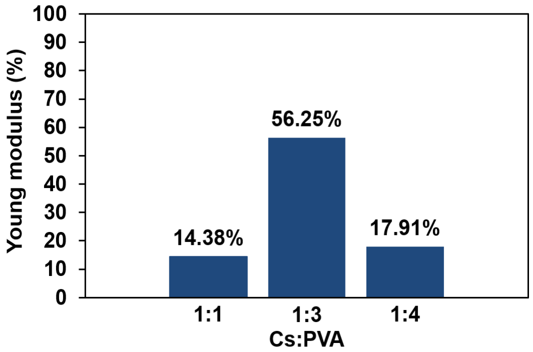

Figure 5. The effect of PVA ratios on the Young modulus of plasticized unfractionated chitosan.

Chitosan is soluble in a small number of acids, including hydrochloric, lactic, propionic, phosphoric, tartaric, citric, succinic, acetic, and formic acids, at specific pH levels when stirred continuously [45,46]. Lower solubility values of chitosan may indicate insufficient protein removal [47]. The effect of PVA plasticizer on the water barrier properties of chitosan films was investigated. The water solubility of the plasticized films increased depending on the chitosan: PVA ratios. The degree of solubility of the chitosan films is shown in Table 2. A similar behavior was observed by Rodríguez-Núñez *et al.* [47]. This implies that it is easy to dispose of poly plasticized chitosan (vinyl alcohol) with running water or stationary faucets in homes, factories, or anywhere else in the same way and does not leave or affect the environment [48].

What is amazing about the work is that the unplasticized chitosan film for four rounds, that is, for four days (96 hours) of immersion with stirring, its solubility rate was only 3.9%, and this is expected from it because the non-plasticized chitosan does not dissolve in water, while the most prominent result is the dissolution of polyvinyl alcohol plasticized chitosan films in the first round (one day -24 hours of immersion) by 100%, meaning that there is nothing left of it, and this means that it is easy to dispose of the packaging materials manufactured from this annealed chitosan with water running or stagnant faucet in homes, factories, or anywhere else in the same way and does not leave in the environment and does not affect it [49].

4. Conclusion

An environmentally friendly food packaging film made from plasticized chitosan cast films was successfully prepared. FT-IR analysis of the chitosan spectrum for the plasticized and unplasticized cast films showed that they were similar to the original. This suggests that the addition of the PVA plasticizer did not change the basic structure of the polymeric chitosan chains. In this study, we used different ratios of poly (vinyl alcohol) as a plasticizer, and the results obtained are much more promising for various applications, especially solubility results reaching 100%, which can find different friendly applications, in contrast to the unplasticized chitosan polymer, which is insoluble in water. Plasticized chitosan films were found to improve the optical characteristics of the films produced by making them more transparent. It was discovered that the addition of plasticizers to chitosan improved the solubility of the prepared films in water and reached 100%, Less rigid films were produced, as evidenced by the mechanical tensile strength measurements of plasticized chitosan films, which also revealed a decrease in Young's modulus and elongation at breakage.

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Disclosure statement

Conflict of interest: The authors declare that they have no conflict of interest.
Ethical approval: All ethical guidelines have been adhered to.
Sample availability: Samples of the compounds are available from the author.

CRediT authorship contribution statement

Conceptualization: Hadi Salman Al-Lami; Methodology: Hadi Salman Al-Lami, Sara Hikmet Mutasher; Software: Sara Hikmet Mutasher; Validation: Hadi Salman Al-Lami, Sara Hikmet Mutasher; Formal Analysis: Hadi Salman Al-Lami, Sara Hikmet Mutasher; Investigation: Hadi Salman Al-Lami, Sara Hikmet Mutasher; Resources: Hadi Salman Al-Lami, Sara Hikmet Mutasher; Data Curation: Sara Hikmet Mutasher; Writing - Original Draft: Hadi Salman Al-Lami, Sara Hikmet Mutasher; Writing - Review and Editing: Hadi Salman Al-Lami, Sara Hikmet Mutasher; Visualization: Hadi Salman Al-Lami; Supervision: Hadi Salman Al-Lami; Project Administration: Hadi Salman Al-Lami.

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