Starch–Mucilage Composite Films

Subjects: Materials Science, Biomaterials

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Generally, starch is an edible carbohydrate complex, composed of a linear polymer, amylose (a linear molecule with few branches), and amylopectin (branched-chain molecule). Therefore, the presence of amylose in large quantities provides excellent strength while a high level of amylopectin is responsible for the reduction of the tensile strength during the production of a film. However, starch-based films have limitations in their ability to bear various environmental factors such as temperature, pressure, and natural gases during the handling due to their low strength, flexibility, rigidity, and high hydrophilic nature. To overcome this issue, the combination of starch and mucilage can be used as a binary polymer alternative to improve the mechanical properties of the packaging film. Additionally, the addition of several biopolymers such as cellulose, gum, and gelatin into a starch blend can change the network formation in the film matrix, improving the physicochemical and biological properties of the film. Moreover, mucilage is a water-soluble edible polysaccharide, extensively used in the food industry due to its excellent functional properties (antimicrobial, antioxidant, water-holding, oil holding, and foaming capacity), and diverse industrial applications such as thickening agent, binding agent, emulsifying agent, and suspending agent. Mucilage has a great potential to produce a stable polymeric network that confines the starch granules, which delay the release of amylose in resulting the improvement of the mechanical property of films.

Keywords: starch ; mucilage ; biodegradable film ; food packaging

1. Synthesis of Starch–Mucilage Composite Films

Generally, biopolymer (starch–mucilage)-based edible film can be synthesized by two methods: the casting and extrusion techniques, also known as wet and dry methods [1]. Moreover, the solubility of starch–mucilage and other additives is an important factor for the casting method of film formation, while thermo-plasticity of starch–mucilage along with gelatinization characteristics, glass transition, and phase transitions can be recognized for the extrusion method [2]. Among these two methods, the casting method is widely used for the synthesis of starch–mucilage films due to its low production cost and is also known as the solvent-casting method. Furthermore, the solvent casting method comprises three successive steps to prepare a film from the binary polymers (starch and mucilage): (i) solubilization of starch–mucilage sample in an appropriate solvent, (ii) casting or forming of the prepared starch–mucilage solution in molds, and (iii) drying of starch–mucilage-casted solution [3][4]. The synthesis of the starch–mucilage film is explained in **Figure 1**.

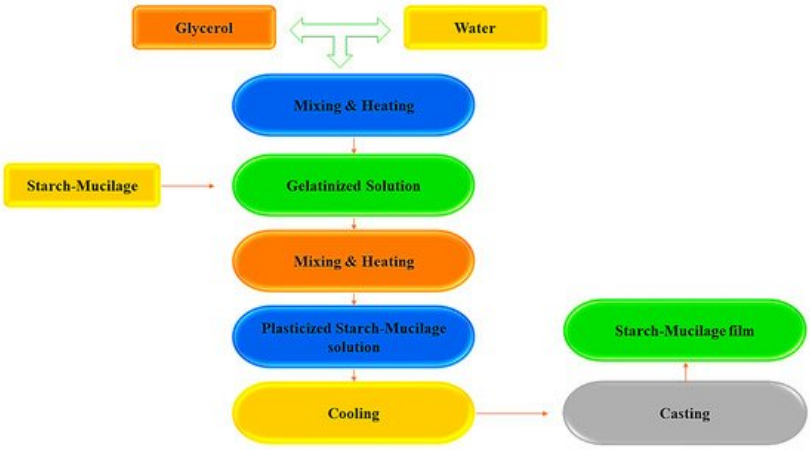

Figure 1. Synthesis of the starch–mucilage film by casting method.

Glycerol is most commonly used to dissolve or disperse the starch and mucilage polymers; this process is known as solubilization. The resulting solution is poured into a specified mold or glass plate during the casting process. The drying process allows the solvent to evaporate, resulting in a starch–mucilage film that binds to the mold [5]. For the casting of films, drying techniques such as vacuum driers, microwaves, tray dryers, and hot air ovens are used to evaporate the solvents and peel the film [6]. In this context, films were prepared from the *Plantago psyllium* starch and seed mucilage by

Krystyjan et al. [7]. In their study, they proved that films prepared with binary polymers have strong physicochemical properties. Additionally, the binary combination of starch and mucilage showed high thermal stability and reduced the decomposition process as compared to film prepared by single polymer starch only. Moreover, the amount of pseudo-plasticity was decreased with the increase in the starch concentration due to galactomannans structure being composed of a mannose backbone, which interchanges with galactose chains. The breakdown of the network induced by shear forces was faster than the recovery of the structure in systems where thixotropy characteristics were dominant. Weak physical bonds were broken as a result of shear forces, and the interior structure disintegrated into separate particles. Additionally, with the addition of mucilage, the rheological behavior of film-forming solution showed various changes such as increased shear stress, higher consistency coefficient, and influence on the value of the area of the hysteresis loop [8].

Consequently, plasticizers and cohesive matrix can be used for the development of easily peeled starch–mucilage edible films with an excellent uniform microstructure, thermal stability, barrier properties, and mechanical properties. The starch–mucilage pre-fabricated film can be used in several industrial applications, mainly in food applications. Here, the film can act as a good barrier, protector, and reduce the loss of water from food products [4]. Therefore, they can enhance the shelf-life of food products. The thickness of the film can be decreased by increasing the concentration of starch nanocrystal due to the excellent composite formation between starch nanocrystals and mucilage [9]. In addition, the thermal property, mechanical strength, and barrier property of starch–mucilage edible film can be improved with the increasing concentration of plasticizers. However, the casting method requires more drying time, which is the main drawback of this method. Likewise, edible films prepared from the potato husk starch and prickly pear peel Mucilage showed a positive impact on their properties such as excellent flexibility, transparency, and bright appearance. The water solubility of the film was influenced by the potato husk starch content and a high amount of glycerine and prickly pear peel leads to films with higher water retention capacity, moisture, and thickness [10]. Therefore, the good rheological property of starch–mucilage film is highly dependent on the synthesis method of films. However, this rheological property can be controlled by adding mucilage and starch in various concentrations. Meanwhile, extrusion or dry method of film formation is usually used at a large commercial scale. This method can improve the functional or physicochemical properties and chemical structure of starch–mucilage films [11]. Generally, this method is divided into three zones: the beginning part of the machine (feeding zone), mixing of the sample (kneading zone), and ending part from the machine (heating zone). In this regard, tubular films were prepared from *Opuntia ficus-indica* mucilage (10% *W*/*W*) with waxy maize and acetylated or normal rice starches (70% *W*/*W*) and glycerol (20% *W*/*W*). addition of *Opuntia ficus-indica* mucilage improved the functional and mechanical properties of tubular and extruded films [12]. Because of the interaction of mucilage and glycerol as a plasticizer and the partial breakdown of the starch structure during the extrusion process, it is less susceptible to acetylation. Moreover, polyvinyl alcohol (PVOH) is an important synthetic biodegradable polymer having excellent flexibility, tear, high strength, and gas barrier properties, although it has poor dimensional stability due to high moisture absorption [13]. Furthermore, as compared to other commercial polymers, it has a very high price. As a result, polysaccharides such as starch may be blended with renewable and abundant agro-resources to minimize production costs. Several studies have been reported to improve the compatibility of starch and polyvinyl alcohol such as fillers, cross-linking agents, compatibilizers, and plasticizers [14]. However, as most of these cross-linking agents are usually toxic, their potential use as biomaterials has been limited. To overcome these drawbacks, the mechanical properties and water resistance of starch and polyvinyl alcohol films must be improved using nontoxic functional additives and simple modification techniques. Comparative research was executed by Gomez-Aldapa et al. [15], and films were produced from potato starch (5% *W*/*V*) blended with polyvinyl alcohol (PVOH) (4% *W*/*V*). Therein, glycerol (25% *W*/*W*) was used as a plasticizer. The result of a study proved that films prepared blended with starch and PVOH are highly suitable and manageable for food packaging applications. This is because the incorporation of PVOH into potato starch enhanced the water absorption capacity, and improved mechanical and functional properties and gas permeability of the film. However, biodegradable films produced from potato starch and PVOH blends had a homogeneous appearance, with no obvious bubbles or phase separation, were transparent, and were easy to unmold. Satisfactory compatibility was observed during the processing in all the blended formulations. Moreover, bindings of hydrogen bonds between polysaccharide chains of starch–mucilage and glycerol were analyzed by Fourier-transform infrared spectroscopy (FTIR) analysis of films prepared from the potato husk starch and pear peel mucilage [10]. Hydrogen bonding interaction (**Figure 2**) is responsible for the interactions of biopolymers (starch–mucilage) with absorbed water molecules. However, mucilage consists of galacturonic acid, L-rhamnose, D-galactose, D-xylose, and L-arabinose. These carbohydrate molecules of mucilage have excellent potential to interact with other molecules such as starch (Amylose and Amylopectin) and glycerol due to their high foaming ability [16]. Furthermore, the alternative galactose or arabinose branches prevent intramolecular hydrogen bondings from forming, also maintaining the molecule in an extended state where it may interact with the amylose molecule in the system via non-covalent hydrogen bonds, resulting in a more extended conformation [17][18]. As a result, the degree of pseudo-plasticity can be increased. The creation of polymer complexes encouraged by the release of amylose and low molecular weight amylopectin during the processing may be responsible for the increase in the viscosity of the starch–

polysaccharide system on cooling. Moreover, the viscosity may be increased with increasing sucrose concentration due to crosslinking between starch chain and sugar units [8].

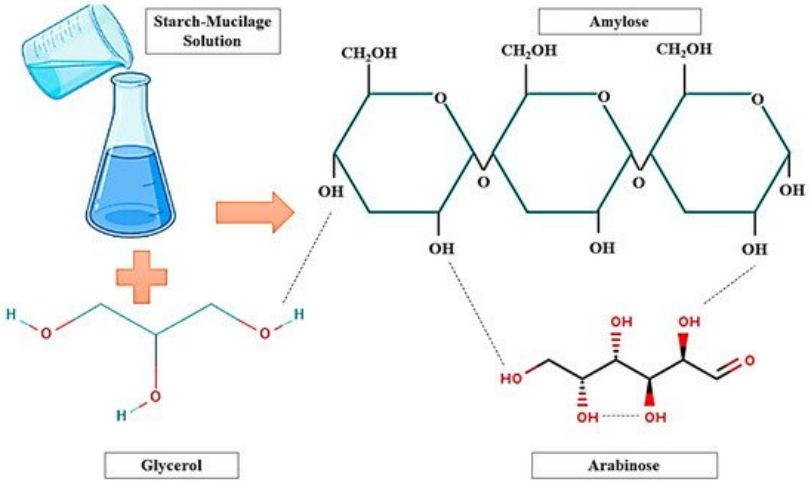

Figure 2. Interactions between starch–mucilage and glycerol through hydrogen bondings.

2. Physicochemical Properties and Characterization of the Starch–Mucilage Film

Nowadays, physical and chemical modifications have been suggested as ways to improve the physicochemical and mechanical properties of starch–mucilage-based films, which seem to be very effective [19]. The major properties of starch–mucilage films are illustrated in **Figure 3**. Starch–mucilage edible films have several advantageous properties. They can enhance the shelf-life of food products, provide excellent protection against UV rays, barrier properties against mechanical damage (cuts and dents), retain the bioactive compounds of foods (antioxidants), and are helpful in the transport of solutes (pigments, additives, and salts). Moreover, desirable physicochemical properties and characterizations of starch–mucilage can be a favorable choice as an alternative to synthetic polymers, especially for food applications. Therefore, all these properties of films mainly depend on the extraction methods of starch–mucilage and synthesis of the film [8]. Several techniques are used for the evaluation of the characterizations of films such as: scanning electron microscopy (SEM), used for determining the surface morphology; Fourier-transform infrared spectroscopy (FTIR), used to evaluate the specific functional groups present in the sample; nucleus magnetic resonance (NMR), used for the determination of organic molecules; X-ray diffraction analysis (XRD), used to check the intensity of materials; thermogravimetry analysis (TGA), used for measuring the mass variations of a sample by temperature; and differential scanning calorimetry (DSC), used for the determination of physical and chemical changes during the thermal processing of a sample [9][20][21][22][23]. Furthermore, it has been observed that two or more polymers are commonly blended to obtain a wide range of biological and physicochemical properties of films. However, several properties of chitosan can be improved by blending with natural polymers such as starch, mucilage, and cellulose, as well as with synthetic ones such as graphene oxide, poly(vinyl pyrrolidone), poly(ethylene oxide), poly(lactic acid), zein, konjac glucomannan, and sodium alginate. Physicochemical properties including thermal stability, surface morphology, hydrophilicity, and hydrophobicity of blended films are mainly dependent on the types of biopolymers [24].

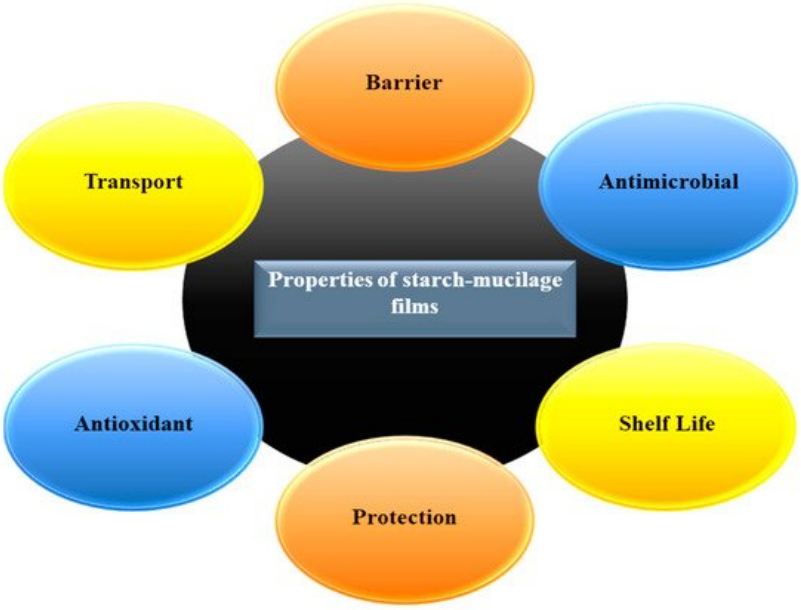

Figure 3. Major properties of starch–mucilage edible films.

2.1. Fourier-Transform Infrared Spectroscopy (FTIR)

Fourier-transform infrared spectroscopy (FTIR) is a technique used to determine the functional groups and chemical structure of the film. Generally, it is used in the wavelength range between 4000 and 400 cm^{-1} [25]. In this context, Krystyjan et al. [7] characterized the film prepared from the *Plantago psyllium* starch–mucilage by FTIR spectroscopy. In their study, a high number of changes was observed in the spectrum of the film prepared from the starch only, compared to film prepared from the starch–mucilage film. Broadband spectra were recognized at about 3200–3300 cm^{-1} in O-H stretching. Furthermore, asymmetric vibrations of the ring appeared at around 1100 cm^{-1}. Spectra at 1080–960 cm^{-1} corresponded to C-O groups with stretching vibrations. Spectra of all the prepared film samples presented bands at 3200 cm^{-1} and 2880–2900 cm^{-1} which confirmed the C-H group, as well as CH2 group, were confirmed bands at 1245, 1405–1465, 2855, and 2916–2936 cm^{-1}. Similarly, in chia-seed mucilage nanocomposites, films with starch nanocrystals were produced [9], and the FTIR spectra of the broad absorption peak were recorded around 3290 cm^{-1} linked to the OH (hemicellulosic) group. Characteristic peaks of protein structures were observed at 1417, 1545, and 1643 cm^{-1}. Spectra were deconvoluted at the range between 1200 and 900 cm^{-1}. Moreover, the observed peak at 1033 cm^{-1} was due to hemicellulosic compound glycosidic linkage. Peaks of aliphatic CH wagging were detected around 922 cm^{-1}. The OH groups in starch and glycerol, which are added to the mucilage film, are likely to create new functional groups with hydrogen bonds in the mucilage structure; therefore, the OH stretching band will become wider as the H bands increase due to the formation of new intermolecular hydrogen bonds. Likewise, Wang et al. [26] produced an edible film composed of *Dioscorea opposita Thunb*. mucilage (DOM) and starch. In their study, there were peaks at 1250 cm^{-1} when the effects of pure starch and DOM films are combined. Peaks at 1250 cm^{-1}, 923 cm^{-1}, and 525 cm^{-1} were seen in the spectra of *Dioscorea opposita Thunb*. mucilage films and starch only when the effects of pure mucilage, starch, and DOM films were combined. In another study, psyllium gum and modified starch composite films were prepared by Askari et al. [27]. In this study, films were examined for FTIR study. The characteristic peaks of starch were at 860–1250 cm^{-1} (C-H stretching and C-O stretching of the anhydroglucose ring), 1593 cm^{-1} (O-H blending of water absorbed), 2924 cm^{-1} (C-H stretching), and 3000–3600 cm^{-1} (O-H stretching). Moreover, increases in the percentage of *Psyllium* gum enhanced the intensity of the broad-band at 3300 cm^{-1} (O-H stretching of starch). This might be due to *Psyllium* gum having more hydroxyl groups, which could lead to more hydrogen bonding.

2.2. Scanning Electron Microscopy (SEM)

Scanning electron microscopy is a part of an electron microscope, used to evaluate the surface morphology of starch–mucilage films. Electrons are bombarded on the prepared sample through electron beams resulting in the reflection of electrons towards the object from the sample [28]. Generally, coating of film samples can be carried out with gold for evaluation of surface morphology. Moreover, swollen-dried and freeze-dried films were prepared from corn starch and okra mucilage [29]. The result of the study revealed that the structure of the dry film was free from cavities, and had a homogeneous appearance and flat structure, without major imperfections due to strong interaction between starch, mucilage, and glycerol. During the gelatinization (breakdown of starch bonds) heating of solution, these all were subjected. There are two zones of starch granules (crystalline zone and amorphous zone). In the case of the crystalline zone, hydrogen bonds can be broken down in the presence of heat [30]. Therefore, an amorphous zone causes the

gelatinization of starch, and swelling of the amorphous zone develops. Moreover, kinetic energy was decreased after cooling the solution, which creates new interactions between molecules. However, during the drying of films, strong bonds were created between the okra mucilage structure and corn starch. To obtain stable and flexible films, a plasticizing agent was used for reinforcement. The thickness and microstructure of a film matrix are directly influenced by the surface and interior heterogeneity of the film matrix, according to Gutiérrez et al. [31]. As a result, it is clear that the more complicated and irregular the arrangement of molecules, the more likely it is that micrographs may have errors. Likewise, the film was prepared from the *Dioscorea opposita Thunb.* mucilage and starch [26], and also, the effects of sodium carboxymethyl cellulose (CMC) and ultrasound were observed by scanning electron microscopy. They prepared four samples with different compositions of starch, mucilage, glycerol, and sodium carboxymethyl cellulose. SEM results showed that dry films were free from imperfections or cracks, free from cavities, had clear starch granules, smooth structure, and was homogeneous. This property may be due to interactions between starch, mucilage, glycerol, and CMC during the process of gelatinization with heating. The hydrocolloids assemble themselves in the film matrices, and during heating, the hydrogen bonds may be broken, causing the crystalline zone to develop. The starch expanded during film production, producing granule expansion and the gelatinization of the starch [32]. Likewise, films were prepared from the gelatin of chicken skin and tapioca starch by Loo and Sarbon [33]. Scanning electron microscopy (SEM) micrographs confirmed that the blended films had an excellent smoother surface and improved internal structure over pure gelatin films. Moreover, the surface of gelatin films with 5 and 25% tapioca starch was observed to be enhanced by removing surface unevenness, resulting in smoother film surfaces. The changes in the surface and cross-sectional area of the prepared films were most likely caused by changes in the components of the blended films, as well as interactions that occurred in the film matrix as a result of the addition of tapioca starch. Chen et al. [34] investigated the physicochemical and mechanical characteristics of maize starch films containing cotton linter nano-cellulose, bamboo nano-cellulose, and sisal nano-cellulose. The SEM pictures revealed that it has the strongest reinforcing mechanical strength because bamboo nano-cellulose has the highest aspect ratio.

2.3. Thermal Stability of Films

Differential scanning calorimetry (DSC) and thermogravimetric analysis (TGA) can be used to determine the behavior of physical and chemical changes during the thermal processing, and the difference between the distinct formulation of starch–mucilage films, respectively [35]. Araujo et al. [29] prepared a film from the okra mucilage and corn starch. The okra mucilage precipitate's differential scanning calorimetry curve showed loss of water from the material between 0 and 175 °C, and melting temperature of precipitated mucilage components with an endothermic peak was observed at 180 °C. Moreover, with the modification of pectin and comparing it with the DSC curve, an unusual endothermic peak was observed nearby 140 °C. Degradation of the films occurred at about 250 °C and endothermic peaks were reached at about 315 °C. However, DSC also confirmed that the high rate of mucilage degradation is due to the high degree of methoxylation of the carboxylic acids in pectin [36]. In the case of *Plantago psyllium* starch mucilage films, an endothermic effect was observed at low temperature with the relation of water polysaccharides [7]. Five films were evaluated for thermal analysis in different starch–mucilage concentrations. In their study, they observed that both film based on potato starch (H1) (3% *W*/*W*) and mucilage (0.1% *W*/W) and film based on potato starch (H2) (3% *W*/*W*) and mucilage (0.2% W/*W*) contained approximately 15% less water than film based on single potato starch (H0). The highest effect of exothermic with comparable magnitude was observed close to 330 °C. The thermal characteristics of S0 are different from those of S1 and S2 as determined by DSC. Its thermogram showed two exothermic peaks with maximum temperatures of 304 °C and 237 °C (which might be linked with decomposition), but S1 had just one peak at 291 °C and 317 °C which was connected with film composite degradation. This may be due to the addition of mucilage to the solution reducing decomposition, making the films more thermally stable. Furthermore, the addition of mucilage decreased the water content in the system based on the endothermic peak at around 70 °C, which corresponds to the dehydration of some film samples due to mucilage filling the gaps between starch molecules that were interacting with each other through hydrogen bonds. On the other hand, thermogravimetry analysis (TGA) is used for the measuring of mass variation of starch–mucilage films by temperature [37]. Edible films were prepared by *Dioscorea opposita Thunb.* mucilage and starch by Wang et al. [26]. Around 10% weight loss at 100C (indicating the water evaporation), and 48% weight loss at 300 to 350 °C were observed. Moreover, degradation of starch occurred at around 322 °C. Various studies revealed that the thermal property of mucilage is highly dependent upon the conformation of molecules, structures, and behavior of the materials. In this context, they observed continuous weight loss of *Dioscorea opposita Thunb.* mucilage films without apparent peaks in TG.

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