

Research Article

Improvement of Physicochemical Properties of Starch Films by Blending it with Poly(N-Vinyl-2-Pyrrolidone)

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Abstract

Polymer starch-based blends were developed between Starch (St) and Poly(N-Vinyl-2-Pyrrolidone) (PVP). The starch was obtained by extraction from Peruvian yuca (*Manihot esculenta*), known as sweet corn starch. The films were obtained through the casting method. The thermoplastic starch films were formed through the incorporation of glycerol at 20% wt. The concentrations of PVP incorporated in the polymer blend were equal to 10%, 20%, 30% and 40% (wt). Thus, this work aims to understand the interactions, structure and physical properties of the obtained polymer blend films, including the study of their barrier properties. The films obtained through

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the St-PVP blends show remarkable improvements upon mechanical and barrier properties, and they represent a highly resistant material with low oxygen permeability. The blends were compatibles between them in the whole range of concentration studied in this work.

Keywords: Improvement of mechanical properties; Peruvian yuca; Polymer blends; Poly(N-vinyl-2-pyrrolidone); Starch

Highlights

- Obtaining compatible polymer blends of thermoplastic Starch (St) and Poly(N-Vinyl-2-Pyrrolidone) (PVP) and developing their films in different ratios.
- The mechanical, thermal and barrier properties of the St films were improved through PVP incorporation inside the starch matrix.

Introduction

Over the past years, the world's production of plastics has grown steadily. Nowadays, the production of these materials is being estimated to exceed 300 million tons, whereas packaging, considered as the largest market for the plastics industry, represents about the 39.6% of the total demand for plastics. The polymers commonly used in commercial applications are derived from non-renewable sources and are associated with environmental pollution problems [1]. Taking into account the fact that today's consumers demand higher food quality and environmentally-friendly packaging materials with greater recyclability, there is a significantly growing interest in edible and biodegradable films. These materials are potentially used for food systems, playing the role of barriers to moisture, oxygen, carbon dioxide, lipids, taste and aroma between the food components and the surrounding atmosphere [2]. Thus, over the last few decades, there has been relevant progress in the development of biodegradable packaging materials derived from plant and animal resources, such as polysaccharides, proteins, lipids and their mixtures. These biopolymers have been used to obtain films and coatings for the packaging of high quality food stuffs in order to extend their shelf life and to reduce environmental impact [3]. Among these biopolymers, starch is one of the most abundant natural biodegradable polymers, and it is the main storage product for crops, such as wheat, rice, maize, cassava and potato. This compound is widely used in the food industry as a gelling agent, thickener or preparation of edible films and coatings [4]. In general, these polysaccharide films show adequate optical properties, but poor mechanical characteristics where in the water vapour and oxygen barriers are highly affected by moisture due to their hydrophilicity [5]. As a result of the low mechanical resistance and the high sensitivity to moisture exhibited by starch films, several alternatives have been investigated with the goal of improving their properties, such as blending with other polymers, like polyethylene, polyvinyl alcohol or polylactic acid, as well as reinforcing with nano fillers [6-9]. The effect of additives as well as the use of different starch sources and the control of process parameters on the physical properties have

already been reported [10,11]. In this framework, it has been determined that the physicochemical properties of the starch films vary widely depending on the botanical origin of the starch, the content and the type of the plasticizer, as well as the processing conditions [12]. Specifically, cassava starch presents ideal properties for the formation of films because of its ease of preparation, the clarity of paste, the low gelatinization temperature and the good gel stability [13]. Furthermore, cassava starch films have been described as odourless, tasteless, colourless, non-toxic and biodegradable [1]. Previous studies have already formulated cassava starch-based composite films and coatings by combining various other polymers. There are previous reports on blends of starch with poly(N-vinyl-2-pyrrolidone) but are not studies in films [14,15]. PVP has attracted considerable interest due to its hydrophilicity, lubricity, anti-adhesive property and excellent biocompatibility. Furthermore, previous research has already shown that blends of chitosan and PVP can form a homogeneous phase due to the strong hydrogen binding forces between these two kinds of molecules [16,17]. Thus, PVP is proposed in this study as an alternative for the formulation of starch-based material in order to prepare films with improved oxygen barrier properties and mechanical resistance [18]. Finally, the variation of the starch/PVP ratio and the film-forming ability of these polymers were studied for potential applications in food packaging.

Materials and Methods

Materials

Sweet native corn starch was obtained from Peruvian yuca (*Manihot Esculenta*) a tuber which was purchased at a local supplier in Santiago, Chile. Its moisture content was 10% w/w and the amylose percentage was 16%. This last value was determined by the method proposed by Takeda and collaborators [19]. Glycerol (Gly) and Poly(N-Vinyl-2-Pyrrolidone) (PVP) were provided by Sigma Aldrich and their molecular weights were 92,09g/mol and 40,000g/mol, respectively.

Film preparation

Films were produced by means of a casting method. Firstly, the sweet native corn Starch (St) was extracted as follows: The sample was washed, peeled and cut into small pieces (1cm³), then mixed with water in a ratio of 1:4 (St/water), and the mixture was ground for 2 minutes. The supernatant was discarded, and the suspension was then dried in a hot-air oven at 40°C for 24hrs. Finally, the starch was crushed and sieved (100µm mesh). For the obtaining of films, the following process was carried out: St was dispersed in aqueous solution under magnetic stirring and heated until reaching 80°C. Gly was added in ratio 1:0.2 with respect to starch. The sweet native corn starch dispersion and PVP solution were prepared at 8% wt. The starch dispersion was heated until reaching 80°C and Gly and PVP were added according to the blend that corresponded. The stirring and temperature of the blend were maintained at 80°C for 20 minutes. The blends were then placed in a centrifuge to remove the bubbles at 3000rpm for 3 minutes and placed onto polystyrene petri dishes and left in an oven at 40°C for 22hrs. The ratios prepared were St-PVP 90-10, 80-20, 70-30 and 60-40. In this work, the abbreviation St represents starch with glycerol (1:0.2). Starch-based films were conditioned at 25°C and 53% Relative Humidity (RH), using a Mg(NO₃)₂ oversaturated solution for at least 48hrs before every analysis. Starch films containing PVP were named St-PVP10, St-PVP20, St-PVP30, St-PVP40; the number corresponds to the percentage of PVP present in each film.

Characterization

Structural properties

Attenuated Total Reflectance Fourier Transforms Infrared Spectroscopy (ATR-FTIR): Fourier Transform Infrared spectroscopy (FTIR) was used to characterize the films. The films were analyzed in the range from 4000 to 400cm⁻¹, with a resolution of 2cm⁻¹ and 64 scans using a Bruker alpha spectrometer equipped with an attenuated total reflexion diamond crystal accessory (Bruker, Platinum). The spectra analyses were performed using Origin Pro 9.0.

Scanning Electron Microscopy (SEM): Ultrathin (50-60nm) sections were cut using a Sorvall MT-IIB ultra-microtome and placed on a copper grid (300 mesh). Samples were examined using a Scanning Electron Microscope (SEM) JSM-5410 Jeol (Tokyo, Japan) with accelerating voltage at 1.0kV. Images were recorded using a CCD camera Olympus Mega view G2.

Thermal properties

Differential Scanning Calorimetry (DSC): Thermal properties of the films were determined with a Differential Scanning Calorimeter Mettler Toledo Model DSC mark 822e (Switzerland). Commercial samples of indium (99.999% purity) with a melting point of T_m=156.68°C and a melting enthalpy of ΔH_m=38.4Jg⁻¹ were used as calibration standard. Thermo grams were obtained from 25°C to 200°C, cooling to 20°C, and a second heating process to 200°C with a heating rate equal to 10°Cmin⁻¹. Sample weight was about 8-10mg. All these experiments were carried out under purge of dry nitrogen. Glass Transition Temperature (T_g) was determined from the second heating process.

Thermogravimetric Analysis (TGA): Thermogravimetric Analysis (TGA) was carried out using a TGA/SDTA 851 Mettler Toledo thermal analyzer (Schwarzenbach, Switzerland). Samples (ca. 9mg) were heated from 20°C to 600°C at 10°Cmin⁻¹ under nitrogen atmosphere (flow rate 50mLmin⁻¹) in order to prevent any thermoxidative degradation.

Mechanical properties

Tensile strength, elongation at break, and modulus of elasticity for each material were measured at room temperature with a Zwick Roell model BDOFB 0.5 TH Tensile Tester, according to ASTM standard method D-882. Strips (10cm × 2.5cm) of films were cut using a die cutter and kept at 25°C and 53% RH for 48hrs before the test. These analyses were carried out with a 1kN load cell. The initial grip separation was 10cm and the crosshead speed used was 50mmmin⁻¹. Results are the average of 12 specimens for each nano composite film.

Oxygen permeability determination

The Oxygen Permeability (OP) of the films was determined at 53% RH and 23°C using an OX-TRAN (Model 2/21ML Mocon Lippke, Neuwied, Germany). The RH value is in agreement with the treatment for starch films [20,21]. The samples were conditioned at the relative humidity level of the test in a desiccator using an Mg(NO₃)₂ oversaturated solution. Samples were placed in the equipment for analysis, and the transmission values were determined every 20 minutes until reaching the equilibrium condition. The exposure area during the tests was equal to 50cm² for each sample. The thickness of the film was considered to estimate the oxygen permeability. Analyses were carried out in triplicate for each sample.

Swelling and solubility studies for films

The swelling and solubility studies of the films were determined according to the method described by Yoon [22], with slight modifications. Starch-PVP films were dried and weighed (W_{dry}). The films were immersed into 200mL beakers containing 100mL deionized water for 24hrs at room temperature. The samples were removed after every interval of time (30 minutes, 1, 2, 3, 6 and 24hrs), lightly blotted dry to remove the excess of water and then reweighed ($W_{swollen}$). Swelling Power (SP) was estimated at each time from the equation (1):

$$SP (\%) = (W_{swollen} / W_{dry}) \times 100 \quad (1)$$

After this procedure, swollen films were dried at 40°C in a vacuum oven until reaching constant weight (W_{gel}). The solubility and gel fraction were calculated according to the equations (2) and (3), respectively:

$$\text{Gel fraction } (\%) = (W_{gel} / W_{dry}) \times 100 \quad (2)$$

$$\text{Solubility } (\%) = [1 - (W_{gel} / W_{dry})] \times 100 \quad (3)$$

Statistical analysis

A randomized experimental design was considered for the experiments. Data analysis was carried out using StatGraphics Plus 5.1. This software was used to implement variance analysis and Fisher's LSD test. Differences were considered significant at $P < 0.05$.

Results and Discussion

Structural properties

Fourier Transform Infrared Spectroscopy (FT-IR): The FT-IR spectra of the pure components and blends provide additional information, which evidences interactions in different systems with more than one compound, such as polymer blends or nanoparticles contained in a polymer matrix. Specific interactions between components are readily evident through the band displacements, intensity changes, and broadening of the signals, among others [23-27].

Figure 1 shows the spectra of St, PVP and their blends (St-PVP). The spectrum of St shows the characteristic bands, which consider a broad band at 3276 cm^{-1} corresponding to the stretching vibration band of -OH groups. The band at 292 cm^{-1} belongs to the C-H stretching vibration and the bands at 1149, 1079 and 1005 cm^{-1} are bands of C-O stretching vibrations in the anhydroglucose units of starch [28]. The band at 1650 cm^{-1} is typical of water adsorbed in the amorphous regions of starch [29].

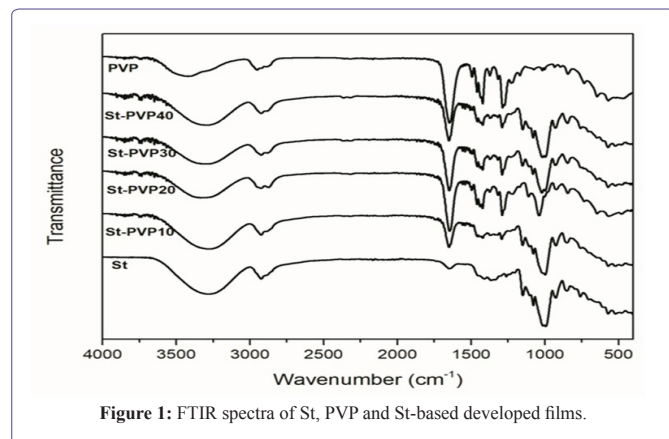


Figure 1: FTIR spectra of St, PVP and St-based developed films.

On the other hand, the spectrum of the PVP shows representative bands like carbonyl stretching at 1647 cm^{-1} and C-N stretching at 1287 cm^{-1} . In all these blends (St-PVP) the presence of the carbonyl band of PVP can be observed, and this is evidence that the blend was successfully prepared. Moreover, there are displacements in the -OH band of starch detected in the samples. The -CO band that belongs to the starch and the -CN band, which corresponds to the PVP, show displacements in blends with regard to pure compounds. This could be due to the strong hydrogen bonding interactions between the functional groups of the components.

Scanning Electron Microscopy (SEM): Figure 2 shows the scanning electron microscopy of the cross-section obtained from the starch film and their blends with PVP. From these images, it is possible to observe a remarkable difference between them. Figure 2A presents the cross-section image of the starch film where a smooth surface can be noticed; with few irregularities and that is free of large imperfections. These characteristics are explained by the presence of plasticizer used in the formation of the films. For the film with 10% (wt) of PVP (St-PVP10), the figure 2B shows a homogeneous and smooth surface, although different to the observed one in the St film. The incorporation of PVP in the St matrix changed its structure without a homogeneity modification of the film. The rugosity of the film increases when the incorporation of poly(vinylpyrrolidone) rises to 20% (wt) (St-PVP20). Figure 2C shows a film with larger imperfections and a rugose surface. It is even possible to observe two zones, as apparently there was not a good dispersion of PVP in the starch matrix at this concentration. Figures 2D and 2E show the blends with 30% and 40% (wt) of incorporated PVP, respectively. The SEM images show that the films were more rugose and less homogenous than the starch film. The increase of PVP content in the films leads to production of more heterogeneous films with defects present, and a less pliable aspect than the films with lower PVP content.

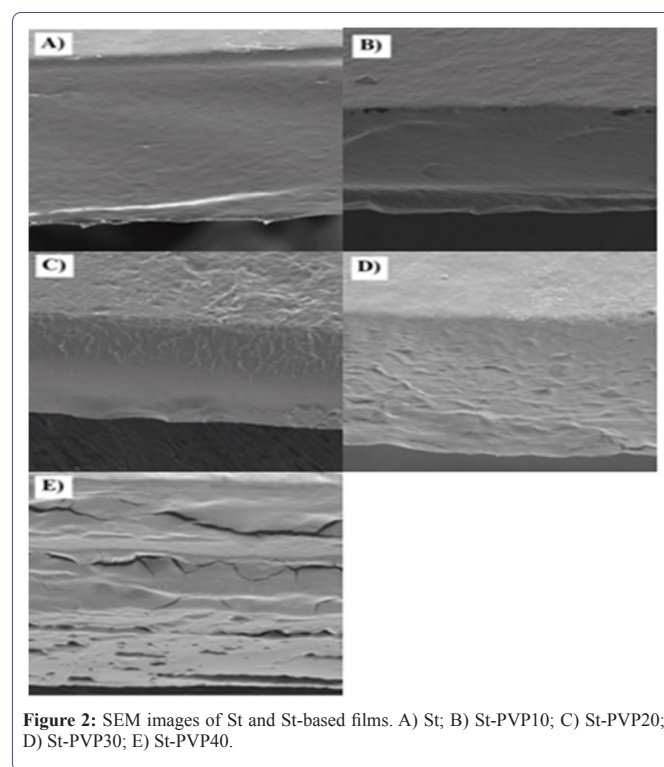


Figure 2: SEM images of St and St-based films. A) St; B) St-PVP10; C) St-PVP20; D) St-PVP30; E) St-PVP40.

Thermal properties

Thermogravimetric Analysis (TGA) was carried out in order to analyze the thermal stability of the films under study. Figure 3 shows the derivative thermo grams obtained. For the starch film, the decomposition occurred in three stages: The first stage corresponds to water loss in the range of 100-130°C; the second stage is related to the decomposition of the glycerol-rich phase, with the starch also present in this stage, that took place at temperatures close to the range 200-270°C; the third stage occurred at around 330°C, which corresponds to the decomposition temperature of the starch components. These results are in good agreement to those reported in the literature for films based on starch-glycerol [30-32]. The obtained PVP profile shows a single stage of decomposition. The onset temperature was at 380°C, while at 450°C the polymer reached its maximum decomposition. This temperature is coherent with the PVP chemical structure, where the amide groups are capable of forming intermolecular interactions such as hydrogen bonds that cause thermal stability of the polymer [33]. All films under study showed a similar trend, with four stages of decomposition, three of them corresponding to starch and the last one to PVP. The decomposition temperature of blends was generally maintained with regard to their pure components. However, for films with 10% and 20% of PVP (St-PVP10 and St-PVP20), the PVP decomposition took place at 434°C and at 444°C, respectively. This decrease could be explained by the interactions between starch and PVP chains, as in these levels of poly(vinylpyrrolidone) concentrations there is a higher contact between starch and PVP and therefore the auto-associations between PVP chains are getting reduced.

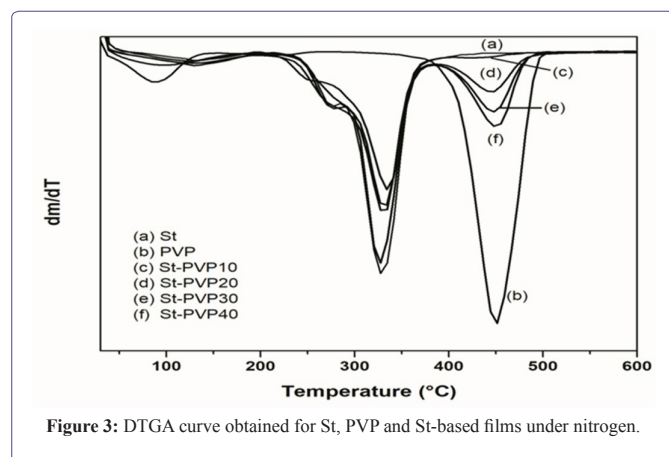


Figure 3: DTGA curve obtained for St, PVP and St-based films under nitrogen.

In order to investigate the miscibility of starch and PVP blends, the samples were assessed by means of DSC analysis. The presence of a single glass transition Temperature (Tg) in a material constituted by two or more polymers is the main criterion of miscibility [24,34-37]. Transparent films were obtained suggesting that phase separation should not be occurring. The reported data are coming from a second heating cycle. Table 1 shows the values of Tg from all the studied samples. The Tg for PVP was observed at 151°C. Especially in this analysis, the main focus is the variation of the Tg value from PVP in the analyzed blends. The Tg values obtained from starch-based blends showed that the Tg values are not the same as those of pure PVP. This fact indicates the blends compatibility for the whole range of the studied compositions. This can be seen in table 1, where it shows that for samples containing PVP, the Tg values were lower than the values

for those of pure PVP. This decrease could be justified by the specific interactions between starch and PVP chains. These intermolecular interactions break the auto-association in PVP chains improving the chain mobility, which generates Tg reduction in the studied blends.

Sample	Tg (°C)	Young's Modulus (N/mm ²)	Tensile Strength (N/mm ²)	Elongation at Break (%)
St	-	2.10±0.4 ^a	0.63±0.1 ^a	169.0±15.0 ^a
St-PVP10	130	14.0±2.0 ^a	2.4±0.2 ^b	29.0±5.3 ^b
St-PVP20	130	57.0±2.5 ^b	6.5±0.9 ^c	14.4±1.4 ^c
St-PVP30	137	122.0±10.0 ^c	9.0±1.5 ^c	5.0±0.4 ^c
St-PVP40	140	107.0±16.0 ^c	6.6±1.1 ^d	3.0±0.1 ^c
PVP	151	-	-	-

Table 1: Tg values and mechanical properties of developed St and St-based films.

Note: Superscript means in each column with different superscript letters are significantly different (p<0.05).

Mechanical properties

The results of mechanical tests of starch-based films are reported in table 1. From these results, it is possible to observe that the incorporation of PVP into the St matrix changed the mechanical properties of the starch. This fact can be detected not only by the Young's modulus increase and the tensile strength values, but also by the decrease of elongation at break values.

The increase in the Young's modulus and tensile strength values indicate that the mechanical resistance of the films increases along with the incorporation of PVP in the starch matrix. This behavior is associated to the intermolecular interaction between starch and PVP because of the hydrogen bonding of OH groups from starch and C=O groups from PVP. This strong association has resulted in a decrease in the local mobility of the chemical groups involved in the interaction with the hydrogen bonding, which generates rigidity in the final material. This conduct has been previously reported in literature [18], with the incorporation of PVP into pectin matrix where an increase of the mechanical resistance in the final film was observed. Furthermore, from the results reported in table 1, it can be seen that the St-PVP film flexibility, in all possible studied ratios, changed through the decrease of the elongation at break values with regard to the starch film value. The high value in this parameter for starch film is due to water content in the starch structure, where the water plays the role of a plasticizer agent, breaking the interaction between amylose and amylopectin chains and thus improving the mobility of polymer chains. In this way, the low flexibility values in the films containing PVP could be explained due to the molecules of water in PVP chains.

Oxygen permeability properties

Table 2 shows the oxygen permeability values obtained for the films prepared in this work. The films were measured at 23°C and 53% RH and the thickness of these samples was ranged from 240 to 280µm. The permeability was expressed in (cm²m⁻²day⁻¹) units. In the performance of the film regarding OP value, certain factors, such as chemical structure, free volume, crystallinity, cross linking, polarity or the preparation method of the film, are involved.

Sample Films	Oxygen Permeability (cm ² /m ² day)
St	38.7±3, 7 ^a
St-PVP10	4.4±1, 1 ^b
St-PVP20	1.7±0, 2 ^c
St-PVP30	5.5±0, 3 ^b
St-PVP40	1.4±0, 1 ^c

Table 2: Oxygen permeability values obtained for St and St-based films.

Note: Means in each row with different superscript letters are significantly different (p<0.05).

The OP value for St film is 38.0 (cm²m²day⁻¹), a value that is in agreement with previous works [20,38,39]. Table 2 shows the incorporation of PVP in a whole range of concentrations studied into the St matrix, where a drastic decrease in the oxygen permeability values can be observed. In this sense, the results show agreement with studies where the films based on biopolymers has generally a lower OP in comparison to synthetic films [40-43]. For the St-PVP30 film it is observed an increase in the OP value regarding the other films, this could be due to defects in the final material, since the general trend is that as increase PVP in the film the OP value decreased.

The decrease of OP values can be explained by the nature of the polymer incorporated into the St matrix. The PVP is a hydrophilic polymer, therefore the film formed has a marked polar trend and consequently, regarding these characteristics, the transfer of oxygen molecules into the film seems to be limited. Furthermore, the generated cross linking, due to the interactions between starch and PVP, mainly through hydrogen bonding, and previously verified through FT-IR analysis, had resulted in an increase of the cohesive energy density of the final material. This last one causes a decrease in oxygen permeability values, which means that the incorporation of PVP, in any concentration under study, improved the starch film oxygen permeability [44].

Swelling kinetics and solubility tests

The swelling kinetics and solubility studies of the materials are an important tool in the characterization of the level of incorporation of PVP in St matrix [22,45]. Figure 4 shows swelling kinetic curves extracted from starch and starch-based film samples. From these results, it can be observed that there is a growth in the amount of water absorbed by the material as a function of time, reaching equilibrium after 6hrs. During the initial stages, the solvent molecules come into contact with polymer chains, increasing its volume and mass. In this way, the presence of PVP increases the capacity of the films to absorb water significantly, expanding the hydrophilicity of the material. This is due to the presence of functional groups, capable of favouring the hydration process through the hydrogen bonds between the polymer network and water.

As the proportion of PVP gets higher in the films, the water absorption capacity increases as shown in table 3. This behaviour occurs together with the polymer chemical structure, where the interaction between starch and PVP generates a three-dimensional network cross linked through hydrogen bonds by hydroxyl groups and through weak intermolecular interactions between the hydrophobic sides, due to the presence of C-H chains. This behaviour transforms the films into superabsorbent materials with a high swelling capacity reaching a value equal to 48% for 60/40 ratio. This is mainly influenced by the

high solubility of PVP in water and the intermolecular interactions between the two polymers. The physical unions are those that favour the solubility of the material because of its affinity with the solvent, and therefore a smaller gel fraction is obtained (Table 3).

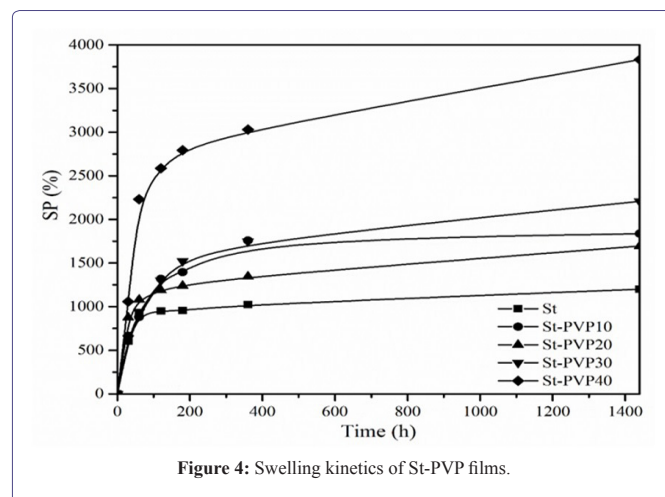


Figure 4: Swelling kinetics of St-PVP films.

Sample	SP (%)	Solubility (%)	Gel Fraction (%)
St	1200±50 ^a	20.00±0.15 ^a	80.00±0.15 ^a
St-PVP10	1800±300 ^b	22.00±1.00 ^b	78.00±2.60 ^b
St-PVP20	1700±200 ^b	28.00±1.70 ^c	72.00±1.60 ^b
St-PVP30	2200±80 ^c	36.00±1.40 ^d	64.00±1.40 ^c
St-PVP40	3800±100 ^d	48.00±0.10 ^e	52.00±0.12 ^d

Table 3: Swelling and solubility in water for St and St-based films.

Note: Means in each column with different superscript letters are significantly different (p<0.05).

Moreover, due to the loss of material in swelling tests, the solubility of the film samples was studied. Figure 4 shows solubility kinetic experiments for the films, which evidence behaviour similar to the observed one in the swelling experiments. The solubility increases considerably during the first hours of contact and stabilizes after 6hrs, almost reaching its equilibrium. An increase of solubility of the films was observed with the addition of PVP in the samples, showing values from 28% for St-PVP20 film reaching a 48% value for St-PVP40 film. This behaviour is mainly influenced by the high solubility of PVP in water, which is over the intermolecular interactions between St and PVP. Furthermore, a gel fraction was obtained and these results are reported in table 3.

From the solubility and swelling results for films, it can be possible to note that the gel fraction for St-PVP10 is closed to the value for the starch film, reaching a lower loss of material by solubility with higher starch content. Nevertheless, a high proportion of PVP in the mixture favours its swelling, also generating a greater solubility in water.

Conclusion

The incorporation of PVP in the sweet native corn starch matrix improved the physical properties of the starch in the developed films. The blends produced were compatible in a whole range of studied

concentrations, which could be observed through the shift from the T_g values for PVP of the films starch-based to the T_g value for pure PVP. In general, SEM images showed less homogeneous films as incorporation of PVP increases in the films.

The FT-IR analysis evidenced the PVP incorporation through the visualization of their characteristic bands and confirmed the interaction between the components by means of bands displacement capable to form strong interactions. These interactions resulted in improvements of the mechanical properties, where the resistance of the films was increased.

Furthermore, the measurement of oxygen permeability for the films evidenced an expected behaviour of the barrier properties, which show a decrease in the values of the starch-based films because of the nature of the incorporated polymer. In this sense, the swelling and solubility tests showed an increase in these films. These results can be explained by the nature of the obtained final material, which involves films with hydrophilic characteristics.

In this study, the incorporation of poly(N-vinyl-2-pyrrolidone) in different ratios in starch matrix resulted in an attractive final material, obtaining films with remarkable physical properties and an environmentally-friendly character. However, further research is necessary to verify their compatibility with real food and storage conditions.

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