



Preparation and characterization of polystyrene/starch blends for packaging applications

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Abstract

Polystyrene/thermoplastic starch blends from 90/10 to 50/50 (w/w) were prepared by melt blending. Blends were characterized by scanning electron microscopy (morphology); thermogravimetric analysis (thermal stability and weight content of each component); Fourier transform infrared spectroscopy (identification of functional groups); differential scanning calorimetry (thermal properties); tensile tests (strength, modulus, elongation at break and tenacity) and biodegradation in soil (biodegradability). The biodegradation process was also followed by thermogravimetric analysis calculating the loss of each component after removing the samples from soil at different time intervals. Scanning electron microscopy results showed good starch dispersion in the blend. The Fourier transform infrared spectroscopy analysis suggested that only physical interaction took place between the polystyrene and the thermoplastic starch. The tensile tests revealed a considerable decrease in the mechanical properties of the polystyrene-thermoplastic starch blends as a function of the thermoplastic starch content. The 50/50 blend showed decreases of 48% in the Young's modulus, 62% in the tensile strength and increases of 62% in the elongation at break, in comparison to neat polystyrene. The biodegradability tests showed that the greater the thermoplastic starch concentration in the blend, the faster the mass loss, which was also

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confirmed by the thermogravimetric analysis and Fourier transform infrared spectroscopy analysis.

Keywords

Biodegradable, characterization, packaging, starch, polystyrene, blends

Introduction

The development of conventional polymers derived from fossil sources has taken place since the first decade of the twentieth century, and because of their usefulness, the plastic industry has grown steadily, generating a massive consumption of plastics in many applications.¹⁻³ Thus, the global consumption of plastic materials has increased from 50 million tons in 1976 to 260 million tons in 2007.⁴ Because they have good resistance to environmental attack and are lightweight and inexpensive, plastics have displaced materials such as wood and glass in many applications including the building, food, pharmaceuticals and transport industries. About 30% of the total world plastic consumption is used in packaging materials. The materials used for this application often have short service time, so they end up mostly in landfills and stay there for over 100 years.¹⁻³ In many cases, products such as bags and bottles cannot be collected after being discarded and so, clogged sewers and drains, together with polluted streets, beaches and scenery, make a very costly impact on waste management. Therefore, the production of polymers from non-renewable sources has become a problem because of their long degradation times.⁵ About 5% of the oil produced worldwide is used for plastic production. This mineral fossil currently experiences continued growth in price, and projections predict that known reserves will be exhausted within 50 years, if the rate of consumption continues as today.¹⁻³

Mechanical recycling is a well-established technology to recover conventional plastics (e.g. polypropylene (PP), polyethylene (PE), polyethylene terephthalate (PET) and polystyrene (PS)). Its main advantage is that part of the resources consumed for the production of the plastic materials is not wasted but preserved for a use in the same, similar or different application. One disadvantage is that materials lose some of their physical and mechanical properties due to their increasing degradation as a function of the number of processing cycles. Degradation can be reduced through by using additives or virgin polymers, among other options.⁶⁻⁸ Even so, the use of recycled plastic materials is currently limited to 1-2%, due to the increasingly high product quality requirements. The recycled plastics obtained from plastic food containers and packaging cannot be used again to manufacture new food containers for sanitary reasons, and so, they must be used for other

applications. Moreover, much of this plastic household waste is film-type and thus very difficult to recover.^{9–11}

Another strategy to get rid of waste is biological treatment, which consists of using biodegradable residues in composting conditions.¹² The increasing environmental pollution has led to the development of biodegradable polymers that are mainly used as food packaging products.¹³ Composting is a biological process whereby the organic material present in the waste is used as an energy source for microorganisms. A degradable polymer is defined as a polymer that maintains its physical integrity during its service life but begins to change chemically after being discarded, influenced by environmental agents, which convert it into simple substances or minor components that are eventually assimilated to the environment. If these environmental agents are biological entities (such as bacteria or fungi), the material is called biodegradable polymer.¹⁴ Commonly known standards for composting plastics are ASTM D6400 (Standard Specification for Compostable Plastics) and EN 13432 (Proof of compostability of plastic products) and are related to the performance of plastics in the production of commercial composting plastics. The mentioned standards require a conversion of carbon to carbon dioxide at levels between 60% and 90%, in a period of 180 days for ASTM D6400 and EN 13432, respectively.

Natural biodegradable polymers include:

- starch (which has hydroxyl groups), whose links are attacked by amylases and glucosidases
- cellulose, which is attacked by the peroxidase secreted by certain fungi and by endo/exo-enzymes produced by bacteria
- chitin, which comes from a marine source and is degraded by chitinase.^{15,16}

Research on starch-based biodegradable plastics began in 1970 and continues today in several laboratories worldwide. Native starch can be transformed into thermoplastic resin-like products by deconstructurization. The plasticization of the native starch granule is obtained by structural disruption during the mixing process and the action of a plasticizer, forming a new material known as thermoplastic starch (TPS).^{17,18} The processing of TPS by conventional techniques such as extrusion, blow molding and injection molding is still being developed.

TPS by itself is unsuitable for packaging applications because of various disadvantages, including:

- its brittleness in the absence of suitable plasticizers
- its poor water resistance related to the hydrophilic nature of starch

- mechanical property deterioration upon exposure to environmental conditions.

Thus, in order to eliminate these disadvantages, TPS needs to be blended with other synthetic polymers.¹⁹ Polystyrene (PS) is a potential candidate that is widely used in packaging but not readily biodegradable.²⁰ Moreover, the cost of PS increases constantly due to the rising price of oil.

Blending PS with TPS is a solution that has been proposed to overcome the disposal problems of PS products. Direct mixing of TPS powder with PS pellets in organic solvents such as chloroform and melt mixing of compatibilized PS/TPS blends are some of the methods that have been studied.²¹ It has been postulated that microorganisms can consume TPS, increasing the porosity and causing a severe reduction in the mechanical properties of the PS matrix, making it lose its structural integrity, dividing it into small parts and thus reducing its molecular weight (chain length) to a level that can eventually be metabolized by microorganisms.^{22–26} Thus, increasing the starch concentration would improve the degradability and reduce the cost of products made of PS and would decrease the mechanical properties of the blends.

The aim of this work was to obtain PS/TPS blends with:

- acceptable mechanical properties for packaging applications
- high biodegradability for post-consumption treatment
- lower cost than packages currently manufactured from neat PS and prepared by techniques similar to those used in the conventional plastic processing industry.

Experimental

Materials

Cassava starch (humidity 10.3 wt%; ash 0.1 wt%; total nitrogen 0.03 wt%; fats 0.1 wt%; cellulose 0.2 wt%; pH 6.0 at 25°C) purchased from Almidonera DIESEL Argentina and polystyrene pellets supplied by Petrobras (trade name: INNOVA HF-555; MFI(200°C-5 kg) 20 g/10 min) were used to prepare the PS/TPS blends. Distilled water and glycerol (density = 1.26 g/ml, boiling point = 290°C) were used to plasticize the starch for the TPS preparation.

In order to prepare films of PS/TPS blends by casting; ethyl acetate (density = 0.90 g/ml, boiling point = 77°C) was used as solvent.

Methods

TPS preparation. TPS was obtained by mixing 7 g of cassava starch powder, 87 cc of water and 6 cc of glycerol. The constituents were mixed for 30 min to obtain a paste which was transformed to TPS by heating at 80°C in a water bath by continuous stirring for another 30 min.

PS/TPS blend preparation. PS/TPS blends (100/0; 90/10; 70/30; 50/50; 0/100; wt%/wt%) were prepared by melt mixing. An intensive Brabender type mixer with two counter-rotating roller rotors was used. The mixing temperature was 155°C, the speed of rotation 150 r/min and the mixing time 10 min. Then, films were prepared by compression molding in a hydraulic press as follows: 10 min at 155°C and no pressure, 10 min at 155°C and 50 kg/cm², water cooling of molds down to room temperature keeping the pressure, mold opening and sample extraction. The films obtained were 300–500 µm thick.

Characterization of the PS/TPS blends. Thermogravimetric analysis (TGA): TGA was carried out in a Shimadzu TGA-50 from 30°C to 600°C at 10°C/min under nitrogen atmosphere.

Fourier transform infrared spectroscopy (FTIR): The diffuse reflectance method (DRIFT) was used to obtain FTIR spectra. A total of 64 scans were carried out at wavelengths from 4000 to 600 cm⁻¹. The equipment used was a FTIR Genesis II.

Differential scanning calorimetry (DSC) tests: DSC tests were performed in a Shimadzu DSC-50 from -60°C to 300°C at a heating rate of 10°C/min under a nitrogen atmosphere.

Scanning electron microscopy (SEM): The SEM photographs of fracture surfaces were taken with a JEOL JSM-6460 LV SEM. Prior to the observation, the surfaces were sputter-coated with a gold layer about 100 Å thick to avoid charging under the electron beam.

Tensile tests: Tensile tests were performed in a universal testing machine Instron 4467 at a constant crosshead speed of 20 mm/min for TPS and 1 mm/min for the rest of the samples. Before tests, all specimens were preconditioned at 30% relative humidity (RH) and 25°C.

Indoor soil burial experiments: Indoor soil burial experiments were carried out as reported by Di Franco et al.²⁷ Basically, plastic boxes (30 cm × 15 cm × 10 cm) were used as soil containers. As-received Pinocha type soil was used as the degrading medium. Several specimens (rectangular shape, 10 mm × 20 mm × 0.3–0.5 mm) of TPS, PS and blends obtained from films were put into cups made of an aluminum mesh to allow the access of microorganisms and moisture and the easy retrieval of the degraded samples. The specimens in the holders were buried 8 cm from the surface to ensure

aerobic degradation. The average room temperature was 20°C and RH was kept about 40% by adding distilled water. Samples were removed from the soil at specific intervals (t), carefully cleansed with distilled water and superficially dried. Samples were then dried under vacuum at 35°C until constant weight. The specimens were weighed on an analytical balance to determine the average water absorption ($W_{\text{abs}}\%$) and sample weight loss ($W_{\text{loss}}\%$). See equations (1) and (2):

$$W_{\text{abs}}(\%) = \frac{(W_t - W_d)}{W_0} \times 100 \quad (1)$$

$$W_{\text{loss}}(\%) = \frac{(W_0 - W_d)}{W_0} \times 100 \quad (2)$$

where:

W_0	=	initial mass
W_t	=	remaining mass (after removing and cleaning) at time t
W_d	=	mass of the dried sample at the same time.

All the results are the average of at least two replicates.

Results and discussion

Figure 1 shows the TGA and the derivative TGA (DTGA) curves for the neat polymers and the PS/TPS blends at different concentrations.

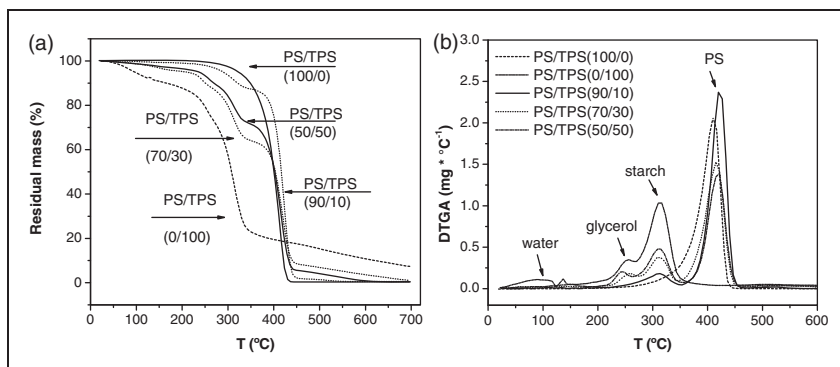


Figure 1. Thermogravimetric curves for polystyrene/thermoplastic starch (PS/TPS) blends at different compositions: (a) Residual mass versus temperature; (b) derivative thermogravimetric analysis (DTGA) versus temperature.

The peak temperatures corresponding to thermal degradation were determined from the previous figure and are summarized in Table 1.

Table 1. Characteristic derivative thermogravimetric analysis (DTGA) peaks of the polystyrene/thermoplastic starch (PS/TPS) blends versus TPS content.

PS/TPS	Peak temperature (°C)		
	Glycerol	Starch	PS
(100/0)	—	—	411
(90/10)	Not detectable	312	419
(70/30)	258	312	418
(50/50)	244	312	415
(0/100)	254	313	—

DTGA curves displayed several peaks; the first one is observed (except for pristine PS) at temperatures close to 100°C and corresponds to the evaporation of water. In the PS/TPS blends, the peaks corresponding to the PS decomposition temperature (around 411°C) and those corresponding to the main TPS components (evaporation of glycerin around 250°C and starch decomposition around 312°C) confirm the presence of TPS and PS in the samples. The results are in accordance with those by Schlemmer et al.,²⁴ who found that PS/TPS blends with greater TPS content show more thermal decomposition stages. These authors also observed a decrease in the peak temperature corresponding to PS decomposition. In the present study, we observed negligible shifts corresponding to PS ($\approx 415^\circ\text{C}$) and glycerin ($\approx 250^\circ\text{C}$) peaks. In addition, the mass losses appear directly linked to the starch concentration in the blends.²²

On the other hand, the real composition of prepared samples can be estimated from the de-convolution of DTGA peaks and from the mass change in each degradation step. Results are summarized in Table 2.

Table 2. Real composition of the polystyrene/thermoplastic starch (PS/TPS) blends (before biodegradation tests).

PS/TPS	Water (wt%)	Additives (wt%)	Starch (wt%)	PS (wt%)
90/10	1	5	8	86
70/30	2	13	15	70
50/50	3	10	27	61

This study is really relevant to compare the original values with those of the degraded samples, as it will be shown later. It is possible to observe that the water content is directly related to the starch content in the blend. On the other hand, although the compositions are increasing in TPS and decreasing in PS, the real values are not identical to the initial ones, which could be related to the sample preparation.

Figure 2 shows the FTIR spectra of TPS, PS and PS/TPS blends.

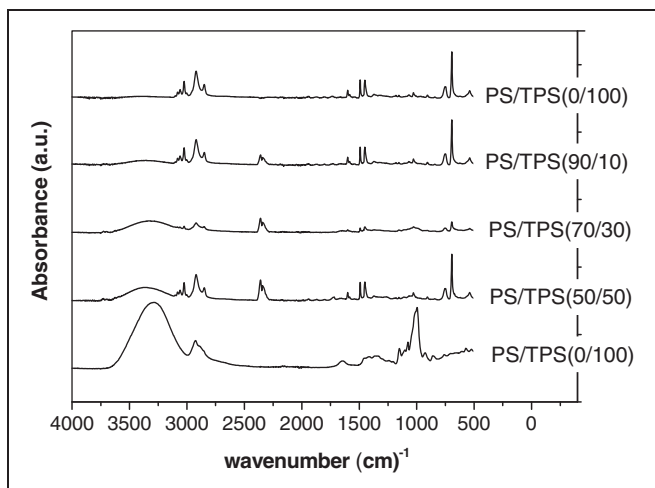


Figure 2. Fourier transform infrared spectroscopy (FTIR) spectra for polystyrene/thermoplastic starch (PS/TPS) blends at different compositions.

The characteristic peaks of pristine PS corresponding to 698, 752, 1495, 1599 and 3018 cm^{-1} are related to the C-H bond stretching of the aromatic rings. The peaks between 1660 and 2044 cm^{-1} are associated with the C=C bond stretching in the aromatic rings, and those at 2848 and 2922 cm^{-1} correspond to the C-H bond stretching of CH_2 groups.²⁶ For pristine TPS, an absorption band at 999 cm^{-1} is associated with three peaks characteristic of polysaccharides and is attributed to the extent of deformation and bending of C-O-C and OH groups of the starch structure. The peaks at 1342 cm^{-1} and 1647 cm^{-1} are associated with the C-H bond bending and the OH group deflection of water, respectively, indicating that TPS is hygroscopic. The absorbance at 2929 cm^{-1} corresponds to the extension of the C-H vibrations, and the strong broad absorption band at 3280 cm^{-1} corresponds to the OH group stretching. The amplitude of this last band indicates the presence of

intermolecular hydrogen bonds.²⁸ The PS/TPS blends obtained displayed absorption peaks at nearly all the wavelengths previously mentioned. Few of the previously mentioned peaks were not identified but this was due to the appearance of other peaks producing overlapping. These data confirm the presence of PS and TPS in the blends studied. Negligible peak displacements were observed suggesting that only physical interaction between the components in the blends took place.²⁸

Figure 3 shows the DSC curves for PS/TPS blends at different compositions.

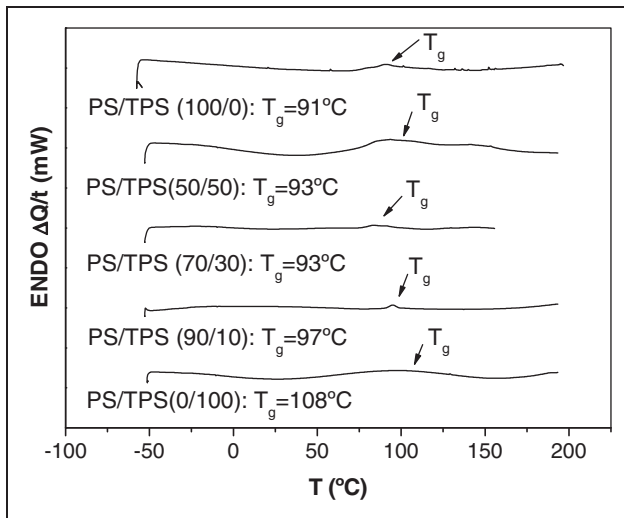


Figure 3. Differential scanning calorimetry (DSC) curves for polystyrene/thermoplastic starch (PS/TPS) blends at different compositions.

It may be noted that the glass transition (T_g) is visible from these curves and that the values are located close to pristine PS. PS seems to have more influence on the T_g value of the blends than TPS. Schlemmer et al.²⁴ obtained the same result for PS/TPS blends prepared with different plasticizers. These authors explained that the plasticizer used does not act on PS under the conditions studied.

Figure 4 shows SEM micrographs of PS/TPS blends at different compositions.

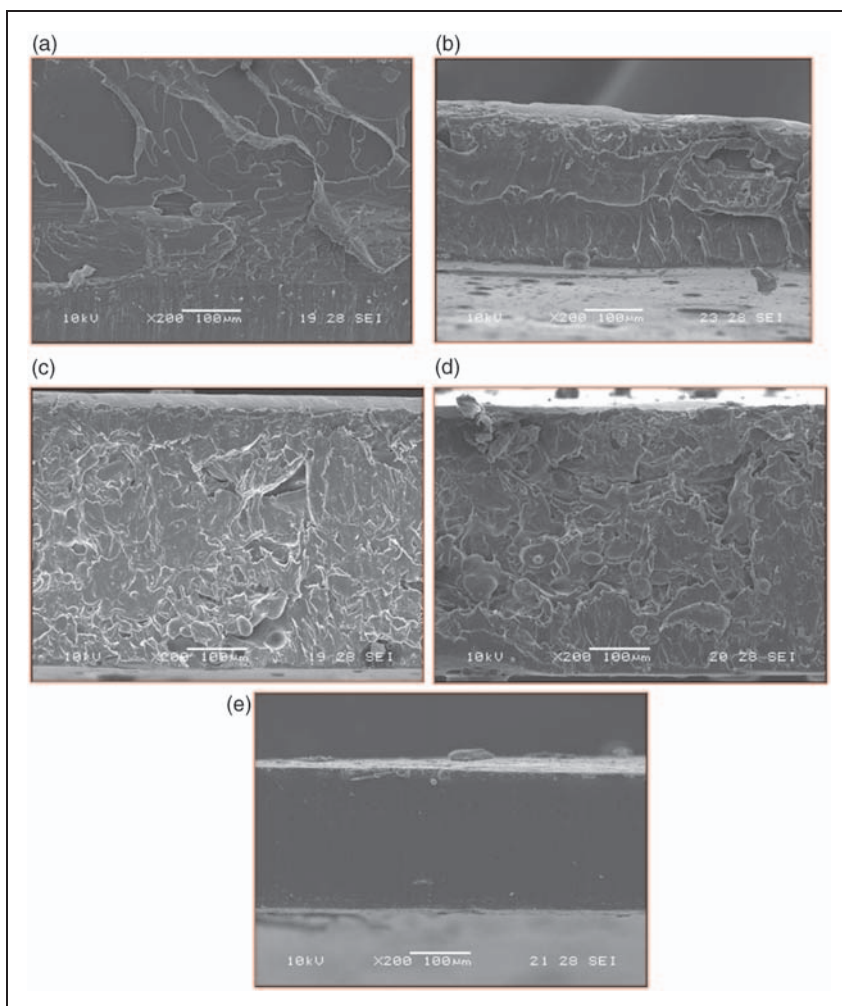


Figure 4. Scanning electron microscopy (SEM) micrographs of polystyrene/thermoplastic starch (PS/TPS) blends at different compositions: (a) PS/TPS (100/0); (b) PS/TPS (90/10); (c) PS/TPS (70/30); (d) PS/TPS (50/50); (e) PS/TPS (0/100).

In the 50/50 PS/TPS sample (Figure 4(d)), spherical particles or lenticular cavities typical of the starch granules, between 5 and 35 microns, can be observed. These characteristics are less visible in the 70/30 blend (Figure 4(c)) and no longer visible in the 90/10 blend (Figure 4(b)). The absence of agglomerates of starch granules indicates that there is a good dispersion of TPS in PS.

The stress–strain curves obtained in the pristine PS, pristine TPS and the 50/50 blend tensile tests, conditioned at 30% RH, are shown in Figure 5.

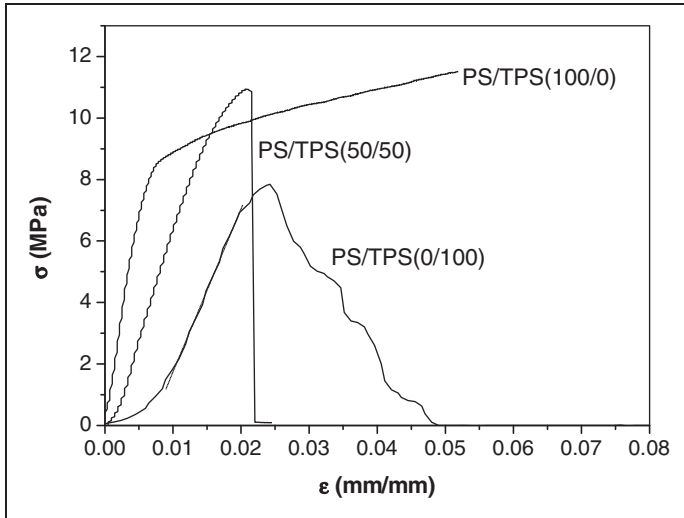


Figure 5. Pristine polystyrene (PS), pristine thermoplastic starch (TPS) and the 50/50 blend stress–strain curves, conditioned at 30% relative humidity (RH).

The pristine PS curve, i.e. the 100/0 blend, shows the typical behavior of a material with elastic response with subsequent plastic deformation hardening. It can be observed that, at the beginning of the curve, the relationship between stress and strain is linear ($\sigma = E \cdot \varepsilon$, where E is the constant of proportionality or Young's modulus); then there are short intervals in which a slightly nonlinear deformation takes place and then plastic deformation, reflected in the curve as a sudden change in the slope of the curve, begins. The positive slope of the curve in the range of plastic deformation in the material implies that hardening occurs, which, in amorphous polymers such as PS, is related to the polymeric chains aligning parallel to the load, until the specimen finally breaks. The 50/50 blend seems to have very little plastic deformation, behaving as a brittle polymer, with an elastic modulus intermediate between PS and TPS. For pristine TPS, i.e. the 0/100 blend, the curve also shows a linear elastic behavior interval and a subsequent plastic deformation but, in this case, without strain hardening (Figure 5). It is important to note that, in the case of TPS, the environment has a huge effect on the mechanical behavior, so, we also studied the effect of relative humidity on such behavior and the results are displayed in Figure 6.

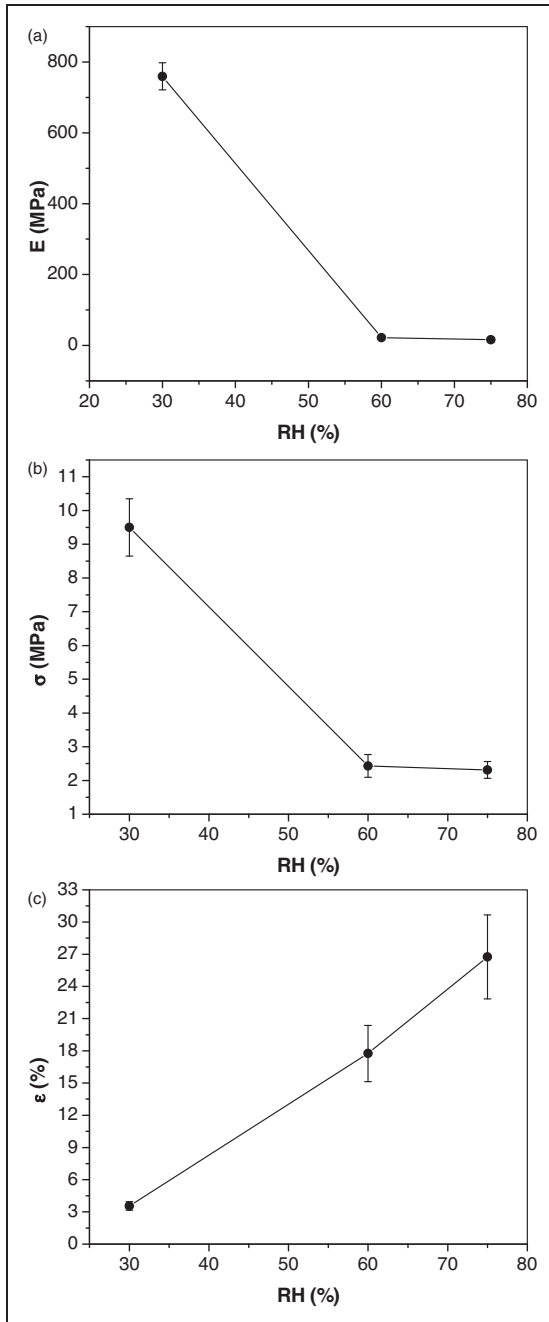


Figure 6. Thermoplastic starch (TPS) mechanical properties versus relative humidity (RH%): (a) Modulus; (b) Tensile strength; (c) Elongation at break.

The data in Figure 6 show the importance of pre-conditioning the samples for the tensile tests, because humidity has a significant effect on the mechanical properties of TPS. The absorbed humidity (water vapor) acts as plasticizer for TPS, lowering its Young's modulus and tensile strength and increasing its elongation at break.

The curves obtained from the tensile tests for the different blends pre-conditioned at 30% RH and 25°C were used to calculate the mechanical properties of the materials. Figure 7 shows the results versus TPS content.

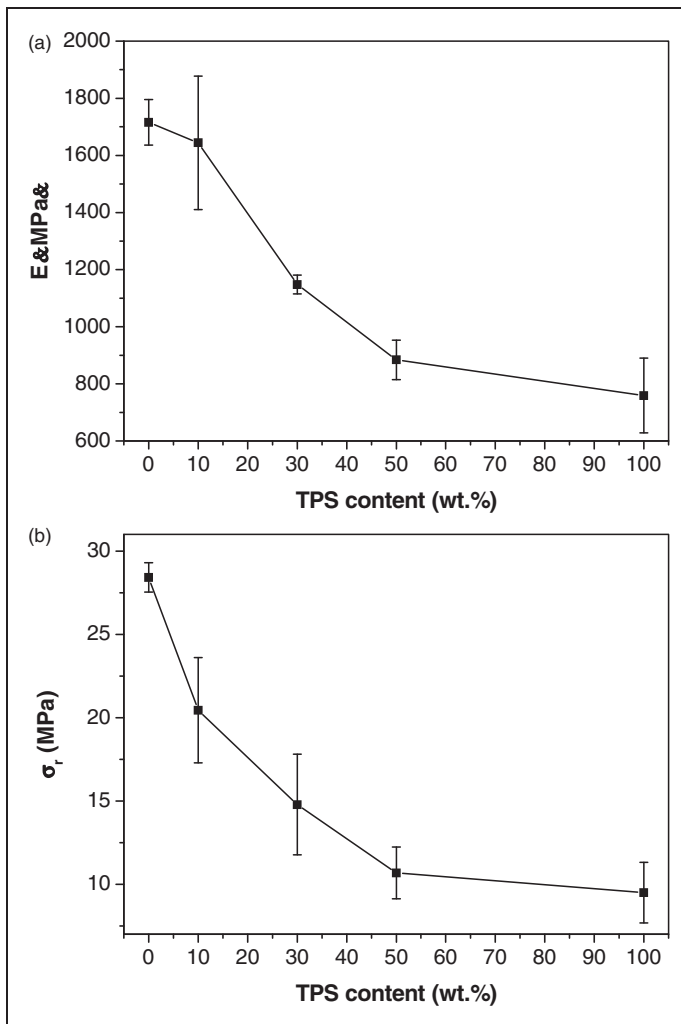


Figure 7. Polystyrene/thermoplastic starch (PS/TPS) blend mechanical properties versus TPS content: (a) Modulus; (b) Tensile strength; (c) Elongation at break.

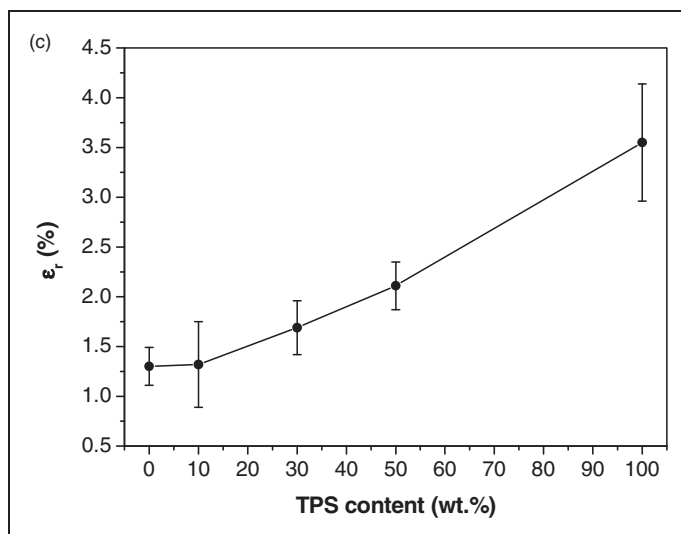


Figure 7. Continued.

Figure 7 shows that adding TPS to PS lowers the Young's modulus and tensile strength, with respect to pristine PS, and that this effect is generally greater as the TPS content increases. This result is a consequence of blending the PS with a weaker polymer (lower Young's modulus and tensile strength) such as TPS. A greater effect than that predicted by the rule of blends can be attributed to strong phase separation and poor compatibility and weak adhesion between the blend components, but the almost linear increase in the elongation at break versus TPS content suggests good adhesion between components. The same tendencies were obtained by Kiatkamjornwong et al.²⁵ for similar systems.

The water absorption and weight loss versus exposure time obtained during the biodegradation tests for the different blends are summarized in Figure 8.

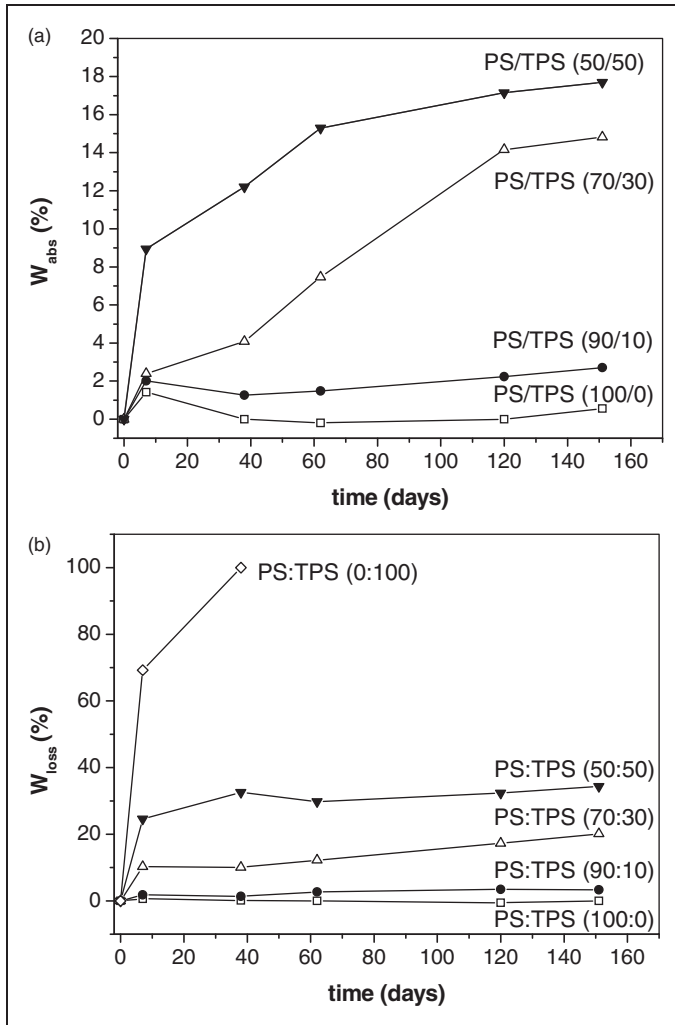


Figure 8. Water absorption (a) and weight loss (b) versus exposure time for the neat PS and the polystyrene/thermoplastic starch (PS/TPS) blends.

It is possible to observe that both water absorption (Figure 8(a)) and weight loss (Figure 8(b)) increased versus TPS content, confirming that TPS is the component being biodegraded in the PS/TPS blend.

Figure 9 shows the TGA/DTGA curves of biodegraded samples (151 days of exposure).

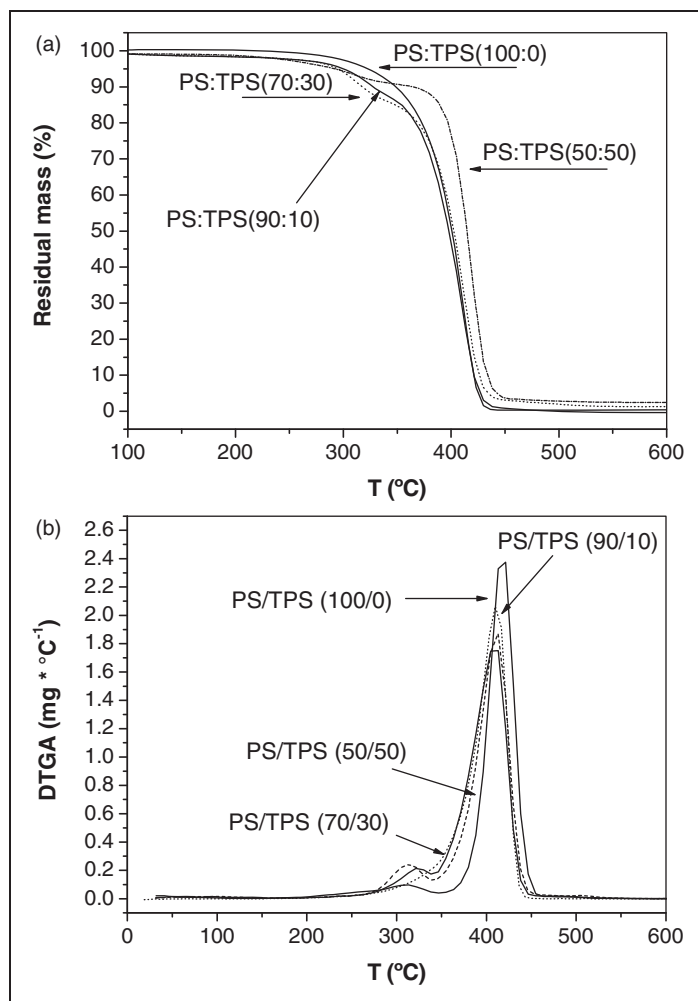


Figure 9. Thermogravimetric curves for polystyrene/thermoplastic starch (PS/TPS) blends at different compositions after biodegradation in soil (151 days of exposure): (a) Residual mass; (b) derivative thermogravimetric analysis (DTGA).

These tests confirmed that biodegradation consumes the starch, which is the bio-susceptible component in the blends.²⁹ The peak corresponding to PS is clearly distinguishable in all samples whereas those related to the additives (water and glycerol) disappeared completely, which means that they are completely degraded. On the other hand, the peak associated with starch thermal degradation has been reduced in comparison with DTGA curves of the original samples (before biodegradation tests). Table 3 summarizes the

characteristic peak temperatures observed in the DTGA curves and the real composition of starch in the biodegraded samples (151 days of exposure).

Table 3. Temperatures of the characteristic peaks from derivative thermogravimetric analysis (DTGA) and final starch composition in the samples (total and loss) after the biodegradation tests (151 days of exposure).

PS/TPS	W_{loss} (%)	T_{starch} ($^{\circ}\text{C}$)	Final starch (wt%)	Starch loss (%)	T_{PS} ($^{\circ}\text{C}$)
90/10	3	347	8	0	411
70/30	20	346	10	36	411
50/50	34	363	4	84	412

After being buried in soil for 151 days, the thermal degradation step near 346°C (T_{starch} , Table 3) is still present; however, the peak values are shifted to greater temperatures, reaching up to 56°C difference, for the samples with highest TPS content, in relation to the non-degraded samples. Schlemmer et al.²² obtained similar results for similar PS/TPS systems and explained that part of the non-biodegraded TPS during the soil burial tests has an increase in thermal stability probably due to some structural modification in TPS after biodegradation of its components. The peak values corresponding to the thermal decomposition of TPS (T_{PS} , Table 3) remained almost unchanged (around 412°C) after the soil burial test, indicating that the PS structure is the same after exposure. By combining the TGA results with weight loss curves, it is possible to estimate the total weight loss in the sample and, supposing that no PS was degraded, the starch loss can also be determined. It was not possible to include the TPS curves because that sample (the 0/100 blend) was completely degraded after 25 days (Figure 8(b)). The values obtained are also summarized in Table 3. All the results indicate that the biodegradability increased as the starch content increased.

Figure 10 shows the FTIR spectra of biodegraded samples (extracted from the soil after 151 days).

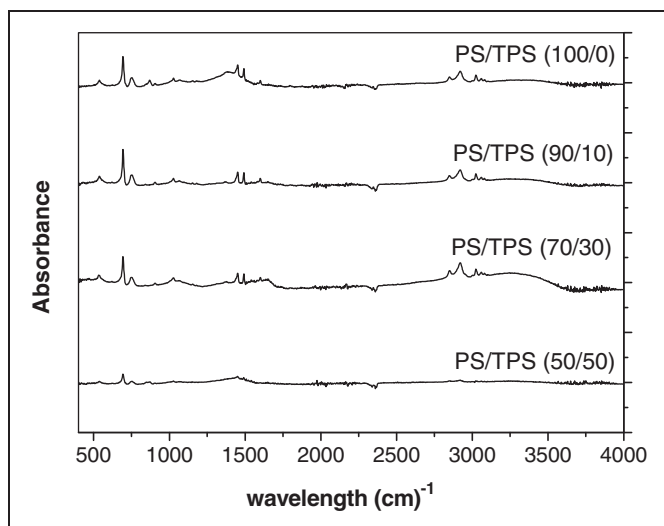


Figure 10. Fourier transform infrared spectroscopy (FTIR) spectra for polystyrene/thermoplastic starch (PS/TPS) blends at different compositions after 151 days of biodegradation in soil.

In the curve of the 100/0 blend extracted after 151 days of burial in soil, the principal peaks are distinguished at the same wavelength as that of the blend before burial, with similar relative height between peaks in each graph, which suggests almost no degradation of neat PS took place. In the case of the blends, TPS biodegradation was observed by the decrease in absorbance of the characteristic band, mainly at 3280 cm^{-1} , confirming the biodegradation of this component, which is in accordance with the soil burial and thermogravimetric tests.

The biodegradation tests allowed us to conclude that as microorganisms consume the surrounding starch, the PS/TPS sample will lose its structural integrity, enhancing other degradation mechanisms.²⁵ And as the starch concentration increases, the degradability characteristics increase.

Conclusions

- Polystyrene and thermoplastic starch blends were obtained by melt mixing and subsequently compression molded.
- The presence of each blend component was followed by thermogravimetric analysis and Fourier Transform Infrared Spectroscopy. The results suggest that only physical interaction between polystyrene and thermoplastic starch took place.

- Polystyrene glass transition temperature (calculated by differential scanning calorimetry) was not affected by the thermoplastic starch concentration in the blends.
- Blending polystyrene with thermoplastic starch decreased the mechanical properties and the deterioration was related to the TPS concentration in the blend.
- The biodegradation rate of the blends significantly increased as a function of the thermoplastic starch concentration in the PS/TPS blend.
- The objectives of the research have been met. Although the blend mechanical properties are inferior to neat polystyrene, they are suitable for use in manufacturing packaging products for food or disposable beverage containers (trays, cups, bottles, etc.) with the advantage of being biodegradable and thus promoting environmental care.

Acknowledgments

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