



Evaluation of the effect of reprocessing on the structure and properties of low density polyethylene/thermoplastic starch blends



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ABSTRACT

The great quantity of synthetic plastic discarded inappropriately in the environment is forcing the search for materials that can be reprocessible and biodegradable. Blends between synthetic polymers and natural and biodegradable polymers can be good candidates of such novel materials because they can combine processability with biodegradation and the use of renewable raw materials. However, traditional polymers usually present high levels of recyclability and use the well-established recycling infrastructure that can eventually be affected by the introduction of systems containing natural polymers. Thus, this work aims to evaluate the effect of reprocessing (simulated here by multiple extrusions) on the structure and properties of a low density polyethylene/thermoplastic starch (LDPE/TPS) blend compared to LDPE. The results indicated that multiple extrusion steps led to a reduction in the average size of the starch-rich phases of LDPE/TPS blends and minor changes in the mechanical and rheological properties of the materials. Such results suggest that the LDPE/TPS blend presents similar reprocessability to the LDPE for the experimental conditions used.

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1. Introduction

The use of polymers is growing due to its low cost, ease of processing, and versatility (Baird & Collias, 1998; Nassehi, 2002; Tadmor & Gogos, 2006; van Krevelen & Te Nigenhuis, 2009). However, the use of polymers in materials and products of rapid consumption (in other words, in products that have a short life cycle), when discarded by its consumers and not recycled, result in environmental problems due to the slow degradation of such materials and the possibility of transforming itself into byproducts with harmful emissions and great environmental impact (Baumann, 1998; Kumar, Karthick, & Arumugam, 2011; Raghavan, 1995).

To minimize the environmental problems and, at the same time, meet market demands, the need arises for a large investment in the production of special polymer systems that could be processed and have properties close to those of conventional polymers while having the ability to be transformed into safe byproducts through biodegradation (Chandra & Rustgi, 1998; Kolybaba et al., 2003). To achieve this aim, polymer blends that combine conventional (usually inexpensive, processable and tough) polymers with special biodegradable polymers (often expensive and displaying

poor processability and properties) are an important option. These blends can be obtained through physical mixtures of synthetic polymers and natural polymers that exhibit biodegradability through traditional processing operations in polymer engineering (Psomiadou, Arvanitoyannis, Biliaderis, Ogawa, & Kawasaki, 1997). Blends of natural polymers (such as starch and chitosan) and commodity polymers (polyethylene and polypropylene) are the subject of many studies (Chiu, Lai, & Ti, 2009; Salmah & Azieyanti, 2010) because they can reduce the use of stable polymers, increase the rate of biodegradation of materials and reduce the use of oil-based raw materials.

Alternatively, materials obtained by mixing synthetic polymers with natural polymers can potentially compromise environmental, economic and social aspects by reducing the recyclability when compared to traditional polymers (Soroudi & Jakubowicz, 2013). In past years, a well-structured network of infrastructure and services has been established in several countries to allow for the recycling of most of the commercial thermoplastics. This fact has enabled the recycling of polymers such as polyethylene and polypropylene to reach high levels, which has major impacts in reducing the levels of raw material and energy consumption and increasing the lifetime of these polymers (i.e., reducing their accumulation in the environment) (Achilias, Roupakias, Megalokonomos, Lappas, & Antonakou, 2007; Barnes, Galgani, Thompson, & Barlaz, 2009). Therefore, a primary concern associated with the strategy of introducing

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natural/biodegradable polymers into conventional polymers is related to the possibility that these blends have lower levels of recyclability compared to the original polymers, which would then have a negative impact on both environmental and industrial issues.

Starch is a semi-crystalline polymer composed of linear polysaccharide molecules (amylose) and branched molecules (amylopectin). It is a natural polymer stored in the form of granules in several plants which is an inexpensive, versatile and available material (Taguet, Bureau, Huneault, & Favis, 2014). Disrupting starch granules in presence with glycerol by conventional processing equipment can lead to thermoplastic starch (TPS) (Garcia, Ribba, Dufresne, Aranguren, & Goyanes, 2011; Rodriguez-Gonzalez, Ramsay, & Favis, 2004; Scmitt et al., 2015). Glycerol content, TPS concentration and processing conditions in blends of synthetic polymer and TPS can result in a wide variety of morphological structures and physical–chemical properties of the final product (Rodriguez-Gonzalez, Ramsay, & Favis, 2003).

Huang, Roan, Kuo, and Lu (2005) conducted a compatibility study of high-content starch/low-density polyethylene (LDPE) blend produced by different processing methods with short life cycle usage, acceptable biodegradation and mechanical properties. As a result, it was shown that the size of LDPE domains decreased for higher contents of the compatibilizing agent. The presence of starch increased the biodegradation rate of the blend to reach 65 wt% weight loss after two weeks of biodegradation.

A recycling study of the poly(lactic acid)/polystyrene blend was performed by reprocessing operations through multiple extrusions and injections. The rheological and mechanical properties of the blends were investigated, and a decrease in the viscosity was observed with the number of processing steps, including a decrease in the stress at break and in the strain at break after two processing cycles (Kotiba, Mosab, & Fawaz, 2010). For polycaprolactone (PCL; an important synthetic biodegradable polymer) submitted to several extrusion cycles, it was found that after five extrusions, a decrease in the mechanical and rheological properties of the samples occurred (La Mantia, Scaffaro, & Bastioli, 2002).

Polyethylene is well known for its high level of recyclability together with its excellent material properties (Meran, Ozturk, & Yuksel, 2009). The incorporation of starch into polyethylene has been tested and used as a means to improve the biodegradability of polyethylene systems while maintaining the properties of the blends at a high level (Chandra & Rustgi, 1997). However, a possible reduction in the degree of recyclability of these new blends (i.e., reduction of the properties and processability driven by recycling steps) compared to pure polyethylene could compromise the environmental appeal proposed for these materials. Therefore, this study aimed to determine the effect of reprocessing (simulated here by multiple cycles of extrusion) on the properties of low density polyethylene (LDPE)/thermoplastic starch (TPS) blends compared to pure LDPE.

2. Materials and methods

2.1. Materials

Low density polyethylene (LDPE) was supplied by Quattor (EI-1630, MFI=30). The native cassava starch (17–24% amylose, 76–83% amylopectin, Native T) and pure glycerol (99.5% purity with 0.5% water) were obtained from Starch Navirai and Synth, respectively.

2.2. Preparation of the blends

The blends were composed of 50% low density polyethylene and 50% thermoplastic starch (30% glycerol and 70% starch) by weight.

Samples of LDPE and of LDPE/TPS blends were first processed using a mixing chamber (Thermo-Haake at 130 °C and 30 rpm) and then extruded using a single screw extruder (AX Plásticos, $L/D=30$) with a temperature profile going from the feed zone to the matrix of 125/135/145/155 °C and a speed of 50 rpm. The samples were then pelletized and injection molded at 150 °C (Hsong JETMASTER – JN 35E, 35 ton) to yield dog-bone-shaped samples according to ASTM D 638. To simulate recycling, LDPE and the blends were submitted to reprocessing through 5 and 10 extrusion cycles before injection molding.

2.3. Characterization

2.3.1. Scanning electron microscopy – SEM

The morphological analysis of the LDPE and the blends that underwent 5 and 10 extrusion cycles were performed using the images obtained with a scanning electron microscope (model FEI Inspect S50). The samples were fractured in liquid nitrogen (cryogenic fracture) and then coated with a thin layer of gold using an SPI Supplies/D2 Diode Sputtering System.

2.3.2. Mechanical properties

A universal testing machine (EMIC-DL3000) was used to evaluate the tensile properties of the samples. The elasticity module (E), the mechanical strength (i.e., maximum stress) and elongation at break (ϵ) were measured and reported according to ASTM D 638.

2.3.3. Differential scanning calorimetry – DSC

The DSC analyses were performed using EXSTAR DSC/7020 equipment under nitrogen atmosphere with a flow of 30 ml/min and at a heating rate of 10 °C/min. Melting temperature, melting heat and crystallization temperature were obtained after erasing the thermal history of the materials. So a first heating run was performed from 25 °C to 150 °C, followed by cooling to –50 °C and again heating to 150 °C (second run).

2.3.4. Capillary rheometry

The rheological properties were measured using a capillary rheometer CEAST SR20. A capillary of $L/D=30$, a barrel temperature of 150 °C and shear rates in the range of 10–10,000 s^{-1} were used. The Rabinowitsch correction was used to adjust the deviation from Newtonian behavior. The samples were kept for 24 h in an oven at 50 °C to eliminate the adsorbed water prior to each analysis. The viscosity data were adjusted according to the Power Law Model.

2.3.5. Dynamic mechanical analysis – DMA

The analyses were performed using a DMS6100 Exstar apparatus. For each sample, the materials were compressed according to the ASTM D4703 standard and cut into dimensions of 50 mm × 10 mm with a thickness of approximately 0.75 mm. Next, the samples were subjected to a sinusoidal strain in tensile mode in the range of –90 to 90 °C, with an oscillation frequency of 1 Hz and a heating rate of 1 °C/min.

2.3.6. Contact angle

Measurements of the contact angles on the surfaces of the samples were obtained using a Digidrop contact angle meter DGD inst DI model. For such measurements, an image of a DI water droplet placed on the sample surface was captured via a CCD camera and then analyzed using the Digidrop software. The contact angles of the right and left sides of the drop and the average contact angle were determined. The measurements were performed at room temperature and five drops per surface were evaluated according to ASTM D 5946.

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