



Sustainable use of cassava (*Manihot esculenta*) roots as raw material for biocomposites development



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ABSTRACT

This work is focused on the use of cassava roots peel and bagasse as natural fillers of TPS materials based on cassava starch. A deep insight into biocomposites microstructure was performed in order to support mechanical and barrier properties of the final materials. Cassava byproducts chemical composition and particle size distribution helped to explain TPS SEM morphology, mechanical and barrier properties modifications. Processing conditions favored starch-filler interactions leading to lower mixing energy requirements. The matrix-filler compatibility was demonstrated by FTIR and thermal analysis of TPS composites. Filler addition increased UV-barrier capacity and opacity of TPS materials, though water vapor permeability was maintained. Both byproducts reinforce TPS matrices even though low filler concentrations were used. Bagasse addition (1.5%) increased 260% elastic modulus and 128% maximum tensile stress of TPS composites, being the most efficient reinforcing agent due to its high residual starch content and lower proportion of smaller particles.

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1. Introduction

Cassava bagasse is a byproduct of cassava-processing industry, which is a fibrous material that contains about 30–50% starch on dry weight basis. By cassava bagasse fermentation non-food products can be obtained, such as pullulan (Sugumaran et al., 2014) or *n*-butanol (Lu et al., 2012) and food-related products such as nutritionally improved animal feed (Sriherwanto et al., 2009) or aroma compounds (Christen et al., 2000). The use of fiber obtained from cassava bagasse has been studied as natural filler intended to reinforce biomaterials, modifying their hydrophobicity and final properties (Pasquini et al., 2010; Teixeira et al., 2012, 2009). Fiber derived from cassava bagasse is also attractive as food additive; however, to the best of our knowledge, there are no reports about the use of cassava peel as filler of polymeric matrices.

Natural cellulosic fibers are playing an important role in a number of applications due to their inherent eco-friendly advantages since the last few decades. They are being explored as green reinforcement alternatives to traditional synthetic fibers for diverse applications. Composites including natural cellulosic fibers offer a

number of advantages over conventional materials such as considerable toughness, flexibility, easy processing, recyclability, and eco-friendliness.

In the light of its wide availability and renewable character, thermoplastic materials obtained by the plasticization of starch are among the most promising alternative to develop biomaterials (Averous and Boquillon, 2004; Shirai et al., 2013; Yu et al., 2006). Understanding of different processes involved on the transformation of starch into a thermoplastic polymer has considerably advanced in the last years (Da Róz et al., 2006; Liu et al., 2009; Mano et al., 2003; Xie et al., 2013). Nowadays, thermoplastic starch (TPS) is an interesting alternative for synthetic polymers in applications that do not require long-term durability nor elevated mechanical performance.

Due to the multiphase transitions of starch, the microstructure and mechanical properties of starch-based materials strongly depend on the processing techniques and conditions (Liu et al., 2009, 2013). For example, compression molding of starch led to sintered, relatively brittle materials while, by injection molding of native starch amorphous materials with enhanced mechanical properties are obtained (Cagiao et al., 2004). In order to fulfill their potential utilization as alternative to synthetic thermoplastics, mechanical properties of TPS must be enhanced. Reinforcement of thermoplastic matrix with natural lignocellulosic fibers is a good

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option to increase their mechanical performance and to preserve the environmental-friendly character of the final material. Thus, Azwa et al. (2013) and Shalwan and Yousif (2013) have extensively reviewed the use of fibers as reinforcement materials in biodegradable matrices. However, the use of industrial byproducts as reinforcement and/or support is still under study. Within this context, cellulose fibers isolated from wood pulp (Da Róz et al., 2006), sugarcane bagasse (Chiellini et al., 2001), jute (Khondker et al., 2006), cotton (Ludueña et al., 2012), and wheat bran (Famá et al., 2009) have been incorporated into hydrocolloid matrices. Moreover, Slavutsky and Bertuzzi (2014) reported that addition of cellulose nanocrystals obtained from sugarcane bagasse helped improve the water barrier properties of starch films.

Nevertheless, there are some disadvantages associated with the use of natural fibers as reinforcement of polymer composites, such as their incompatibility with hydrophobic polymer matrices, tendency to form aggregates during processing, poor moisture resistance, inferior fire resistance, limited processing temperatures, lower durability, variation in quality and price, and difficulty in using established manufacturing process (Azwa et al., 2013; Dittenber and GangaRao, 2012). Chemical treatment of fibers has been identified as an alternative to reduce their susceptibility to moisture and improve the filler-matrix interfacial interactions and thermal stability of the composites (Azwa et al., 2013). Thus, in order to evaluate the capacity of natural fibers as fillers of polymeric materials, it is necessary to gain a deep understanding of their inherent characteristics and composition.

This work is focused on the use of cassava roots peel and bagasse as natural fillers of TPS materials based on cassava starch. Composition of these byproducts, as well as, their compatibility with TPS matrix was investigated. A deep insight into biocomposites microstructure was performed in order to support mechanical and barrier properties of the final materials.

2. Materials and methods

2.1. Materials

Manihot esculenta (cassava) roots were provided by INTA Montecarlo Experimental Station (Misiones, Argentina; 26°33'40.15" latitude south and 54°40'20.06" longitude west). Starch was extracted from the roots following the procedure described by López et al. (2010) roots peel (P) and bagasse (B), obtained from the same extraction process, were dried at 50 °C to constant weight, ground and sieved (through a 500 µm mesh sieve) to be used as filler of thermoplastic cassava starch matrices.

2.1.1. Characterization of cassava byproducts: peel and bagasse

2.1.1.1. *Scanning electron microscopy (SEM)*. Microstructure and morphology of byproducts were studied by SEM, using a JEOL JSM 6360 microscope (Japan). Samples, mounted on bronze stubs using a double-sided tape and metalized with gold layer (40–50 nm), were analyzed under high vacuum mode.

2.1.1.2. *Chemical composition*. Dry matter, ash, lipid fraction, crude protein, total dietary fiber, and lignin content of cassava byproducts was determined following standard methods, as thoroughly described in a previous work (Versino and García, 2014).

2.1.1.3. *Solvent retention capacity (SRC)*. Cassava bagasse and peel solvent retention capacity was analyzed following the AACC method 56–11.02 (Rosell et al., 2009). Solvents used were deionized water, sucrose (50% w/w), sodium carbonate (5% w/w), and lactic acid (5% w/w) solutions. SRC was expressed as percentage (%).

2.1.1.4. *Particle size distribution*. Filler particle size distribution was studied as described in a previous work using laboratory test sieves (Versino and García, 2014).

2.2. Composites processing

Mixtures of cassava starch, glycerol (30% w/w), distilled water (45% w/w), and residues (0.5 and 1.5% w/w) were prepared. Components concentration was expressed in g per 100 g of starch (dry basis). Formulations were named TPS, TPS-P0.5, TPS-P1.5, TPS-B0.5, and TPS-B1.5, where P is referred to peel and B to bagasse, numbers indicate filler concentration. Byproducts were premixed with starch to achieve good dispersion within the matrix. Then, glycerol and distilled water were added, and samples were mixed and conditioned at 25 °C during 24 h. Mixtures were processed in a Brabender Plastograph (Brabender, Duisburg, Germany) at 140 °C and 50 rpm for 15 min. Curves corresponding to torque (Nm) versus processing were recorded.

Thermal degradation of developed biocomposites was carried out in a thermogravimetric balance TA Instrument Discovery Series (New Castle, USA). Samples, previously conditioned at 25 °C and 60% relative humidity (RH), were heated from 30 to 700 °C at 10 °C/min, employing an air flux (40 mL/min). Curves of loss weight as function of temperature were recorded and the maximum decomposition temperature of each component was obtained from first derivative curves.

2.3. Films preparation

Films were obtained by thermo-compression using an hydraulic press, following the processing conditions reported by Castillo et al. (2013). Mixtures were previously conditioned at 25 °C and 60% relative humidity (RH), and films were prepared at 140 °C and 150 kg cm⁻² during 6 min. Before characterization, films were conditioned at 25 °C and 60% RH. Film thickness was measured at least in ten different locations using a digital coating thickness gage Check Line DCN-900 (New York, USA).

2.4. Microstructural characterization

2.4.1. Scanning electronic microscopy (SEM)

Homogeneity and appearance of films were examined by SEM. This study was performed in a JEOL JSM-35CF electron microscope (Japan), with a secondary electron detector. Films were cryofractured by immersion in liquid nitrogen, mounted on bronze stubs and coated with a gold layer (~30 Å), using an argon plasma sputter coater (PELCO 91000). Samples were observed using a 10 kV accelerating voltage, under high vacuum mode.

2.4.2. Fourier transform infrared spectroscopy (FTIR)

Spectra were obtained using a Thermo Nicolet Nexus spectrophotometer (Milwaukee, USA). Samples were prepared by mixing thermoplastic starch mixtures as fine powder with KBr (Sigma-Aldrich, 99%) at 3% w/w. Mixture was pressed and a transparent sample was obtained. Spectra were achieved from 100 accumulated scans at 4 cm⁻¹ resolution in the range 4000–400 cm⁻¹.

2.5. Thermal properties

Modulated differential scanning calorimetry (MDSC) assays were performed in differential scanning calorimeter Q100 (TA Instruments, New Castle, USA). Approximately, 10 mg of film, previously conditioned at 25 °C and 60% RH, was weighted in hermetic pans in order to avoid water loss. An empty hermetic pan was used as reference. Samples were heated from 0 to 200 °C at 10 °C/min,

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