

Research Article

Water Absorption and Thermomechanical Characterization of Extruded Starch/Poly(lactic acid)/Agave Bagasse Fiber Bioplastic Composites

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Water absorption and thermomechanical behavior of composites based on thermoplastic starch (TPS) are presented in this work, wherein the concentration of agave bagasse fibers (ABF, 0–15 wt%) and poly(lactic acid) (PLA, 0–30 wt%) is varied. Glycerol (G) is used as starch (S) plasticizer to form TPS. Starch stands as the polymer matrix (70/30 wt/wt, S/G). The results show that TPS hygroscopicity decreases as PLA and fiber content increase. Storage, stress-strain, and flexural moduli increase with PLA and/or agave bagasse fibers (ABF) content while impact resistance decreases. The TPS glass transition temperature increases with ABF content and decreases with PLA content. Micrographs of the studied biocomposites show a stratified brittle surface with a rigid fiber fracture.

1. Introduction

Nowadays, the interest in bioplastics is growing for market niches such as packaging, agriculture, or automotive parts among others. They are classified in biodegradable and biobased/nonbiodegradable. In 2014, their total global production capacity reached 1.67 million tons, where 643,000 tons corresponded to biodegradable plastics [1]. By 2018, the production capacity is expected to reach more than 6 million tons, where 1.06 million will correspond to the biodegradable type [1]. The main sources of biomass to produce bioplastics are grains (usually corn), sugar cane bagasse, potatoes, and castor oil. It is reported that other natural sources such as cellulose and corn stover will also become an important raw material [1].

Since starch (S) is an economical biopolymer that is contained in many natural products, it is attractive as a source to make biodegradable plastics [2]. In dry form, depending on the source and characterization method, starch shows a melting temperature (T_m) that varies from 200°C [2] to 220 [3] or even 240°C [4]; however, with 10 % of humidity, its T_m decreases to 160°C [3]. It is mainly composed of amylose and

amylopectin which show very different physical and chemical properties [2, 4, 5].

Starch has to be plasticized to lower its high T_m [3] and processing temperature to avoid degradation before [6] it melts. Plasticification with water, glycerol, sorbitol, sugars, or amino acids lowers its T_m and its glass transition temperature (T_g) [6] forming a thermoplastic starch (TPS) and increasing its moldability. Glycerol (G) is the plasticizer most commonly used for starch in proportions ranging from 20 to 50% by weight. Unfortunately, materials based on starch have low mechanical properties because of its hydrophilic character [7–9]. To overcome that problem, TPS has been mixed with other polymers (forming polymer blends) [3, 4], or with reinforcing agents (e.g., natural or synthetic fibers) [2, 5, 7]. Natural fibers as reinforcing materials offer some advantages; among them the following may be mentioned: improvement of some mechanical properties of the polymer matrix [10, 11], minimization of environmental pollution [9], and lower production costs; however, natural fibers present the limitation that processing temperatures are restricted to less than 200°C.

TABLE I: Composite formulations.

S, g	G, g	TPS*, g	PLA, g	ABF, g	PLA wt% in polymer blend [•]	ABF wt% in composite [▼]
70	30	100	0	0	0	0
63	27	90	10	0	10	0
56	24	80	20	0	20	0
49	21	70	30	0	30	0
70	30	100	0	11.11	0	10
63	27	90	10	11.11	10	10
56	24	80	20	11.11	20	10
49	21	70	30	11.11	30	10
70	30	100	0	17.65	0	15
63	27	90	10	17.65	10	15
56	24	80	20	17.65	20	15
49	21	70	30	17.65	30	15

*TPS represents the mixture of S and G. [•]Polymer blend represents the mixture of TPS and PLA. [▼]Composite represents the mixture of TPS, PLA, and ABF.

Dufresne and Vignon in an early work on starch/fiber composites reported that thermomechanical properties of potato starch films were improved when they were mixed with cellulose nanofibers, showing also a decrease in moisture sensitivity, while maintaining biodegradability. Additionally, they found that increasing the glycerol content the equilibrium moisture increased and that such parameter decreased when the fiber content was augmented [5].

Huneault and Li studied mixtures of TPS with poly(lactic acid) (PLA) using maleic acid as a compatibilizer. They reported that Young modulus (E) and tensile strength increased when PLA content was increased [12]. An increase in E and tensile strength of TPS was reported using poly(lactic acid) fibrillation for reinforcement [13].

Although there are several works that follow the effect of PLA or natural fibers on moisture absorption and mechanical properties of TPS, there are very few reports on the effect of the simultaneous addition of both materials to the TPS. Furthermore, there is only scarce data on properties of TPS composites containing cellulosic fibers that were obtained as a byproduct of an industrial process. Teixeira et al. reported the use of cassava bagasse to obtain fiber reinforced TPS and PLA/TPS blends, but tensile strength did not increase significantly and the fiber essentially acted as a filler [14]. Castaño et al. found that reinforcing a TPS/PLA/PVA blend with pehuen cellulosic husk, the increase on mechanical properties was small [15]. Using pineapple fibers in TPS/PLA blends, an improvement in mechanostatic properties and water resistance was reported [16]. Cellulose derivatives have also been used to reinforce starch [17].

Using PLA [18, 19] or PCL [20] as polymer matrix with *agave sisalana* (sisal) as reinforcer, the results showed an increase in storage [18, 19], flexural [18], and tensile moduli [20].

In this work, the effects of the amount of PLA and/or agave bagasse fibers on moisture absorption and mechanical and thermal properties of TPS are reported. This is the first report on the application of agave bagasse fibers to reinforce TPS/PLA blends, which are discarded fibers from industrial processes.

2. Experimental

2.1. Materials. The materials used in this work were corn starch (IMSA) with 10% humidity, glycerol QP (Golden Bell Products), agave bagasse (tequilana Weber blue var.) fiber, PLA (Ingeo Biopolymer 3521D Industries Leben), and Magnesium Nitrate (Fermont). To prepare TPS, after the starch was dried for 24 h at 60°C, it was manually mixed with glycerol (30 wt%) until a homogeneous mixture was obtained. TPS was mixed with different amounts of PLA and/or fiber (Table 1), in a corotating twin-screw extruder (Leistritz Micro 27 equipment GL/GG 32D) to obtain a continuous cord of polymer blend or composites that were pelletized after extrusion. The pellets of the different materials were first dried at 60°C until a constant weight was obtained; then, they were molded by thermal compression in a Schwabenthan Polystat 200T compression equipment at 180°C and 200 bars during 2.5 minutes, maintaining pressure for 10 more minutes during the cooling stage.

2.2. Equilibrium Moisture. First, composites were dried at 60°C for 24 h; then the material was weighed and placed at 25°C in a closed chamber maintained at a relative humidity of 53% (saturated solution of magnesium nitrate). The composites weight was recorded periodically until a constant weight was obtained.

2.3. Mechanical Tests. Mechanodynamic tests were carried out following ASTM D5023-01, using a thermomechanical analyzer (TA Q800 DMA) and the following conditions: temperature range, -85°C to 150°C, heating rate, 2°C/min, three-point bending clamp, and frequency of 1 Hz. Mechanostatic tests were carried out at 25°C following ASTM D638-04 for stress-strain (Instron 4411, crosshead speed: 5 mm/min), ASTM D790-03 for flexure (Instron 4411), and ASTM D6110-04 (Instron, Ceast 9050) for Charpy impact testing.

2.4. Thermal Characterization. Thermal behavior of the samples was followed by DSC (Q Series DSC Q100, TA

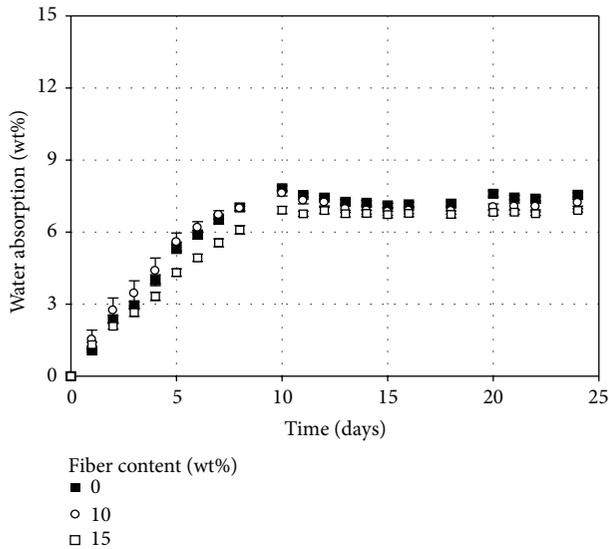


FIGURE 1: Moisture absorption of TPS/PLA/Fiber composites as a function of time for the polymer blend and composites containing 20 wt% PLA.

Instruments), using ASTM D3418-03. Heating rate was 10°C/min from 20 to 180°C.

2.5. Morphology. Samples were observed by Field Emission Scanning Electronic Microscopy (FE-SEM (Tescan, Mira3)). The samples were frozen in liquid nitrogen for 5 minutes before fracture. Subsequently, the samples were dried at 60°C before FE-SEM observation.

3. Results

3.1. Moisture Absorption. Figure 1 shows moisture absorption of the polymer blend and the composites containing 20 wt% PLA. Such figure illustrates that the rate of moisture absorption decreases as the ABF content increases and that equilibrium is reached in approximately two weeks. Similar moisture absorption behavior was obtained for the other materials.

For moisture absorption, it can also be noticed that an increase in ABF content reduces the equilibrium moisture value (Figure 2); that behavior can be explained in terms of the lower hydrophilic character of the fiber comparing with the TPS. Like in this case, it has been reported that composites of TPS with sugarcane bagasse fibers showed a decrease in moisture equilibrium when fiber content was increased [21]. Additionally, since PLA is a less hydrophilic polymer than TPS, independently of fiber content, increasing PLA concentration causes a decrease in the moisture absorption capacity of the composite (Figure 2).

3.2. Thermomechanical Analysis. In Figure 3 the storage modulus (E') is presented as a function of temperature for the composites containing 20 wt% of PLA. There, an overlapping plateau for the 3 samples can be observed from -30 to 40°C;

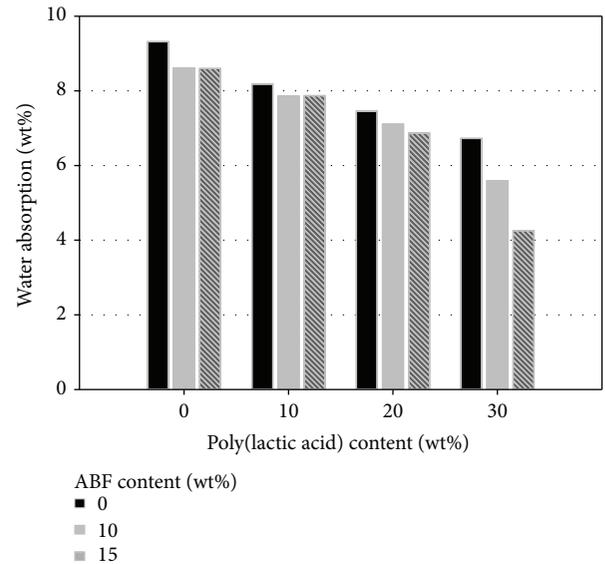


FIGURE 2: Equilibrium moisture of polymer blends and composites varying PLA content.

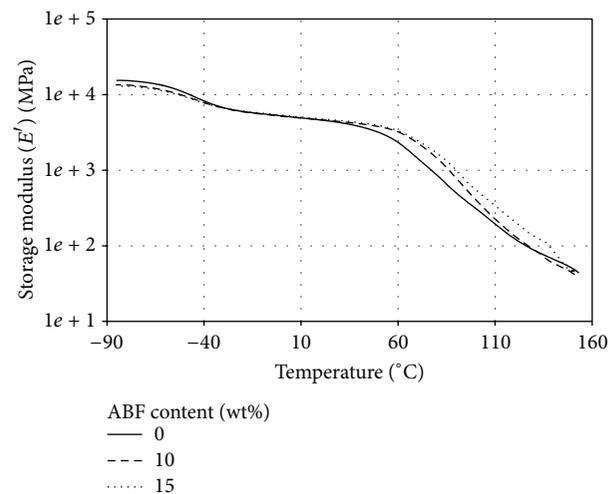


FIGURE 3: Storage modulus as a function of temperature for the polymer blend and composites containing 20 wt% of PLA.

however, the composites maintain the plateau E' value for about 10°C more than the polymer blend. Such additional temperature resistance before E' decay is important for outdoor thermoplastic applications and indicates that the fiber acts as a reinforcing material due to its rigidity and hydrogen bonding between the polymer blend and the ABF. Similar storage modulus behavior was obtained for the other composites. Such type of effect has been reported for TPS reinforced with cellulose fibers [22].

The storage modulus at 25°C of the studied composites is shown in Figure 4. Such figure indicates that higher storage moduli are obtained with the inclusion of ABF and/or PLA, and such increase is larger as more PLA and/or ABF are added. The reinforcement effect of PLA and ABF along with hydrogen bonding leads to a decrease in chains mobility. It

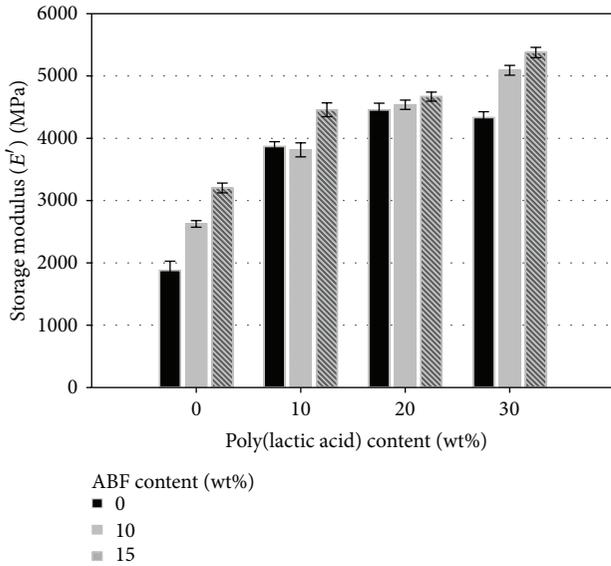


FIGURE 4: Storage modulus of polymer blends and composites at 25°C, varying PLA content.

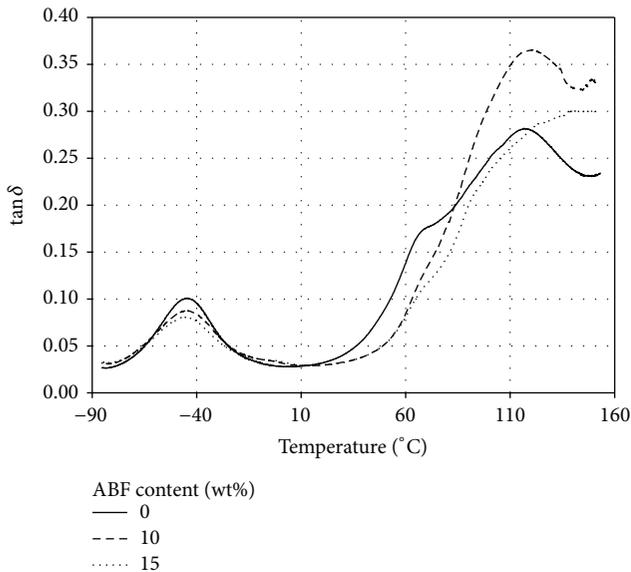


FIGURE 5: $\tan \delta$ versus temperature for polymer blend and composites containing 20 wt% PLA.

has been reported that modulus increases with fiber content [7, 22, 23] due to the high compatibility between TPS and cellulose fillers [5, 10]. Reports about it include bleached leaf wood fibers [24], fibers from bleached eucalyptus pulp [25], flax and ramie fibers [26], wood pulp [27], and tunicin whiskers [28–30].

Figure 5 shows $\tan \delta$ versus temperature for the composites containing 20 wt% of PLA. This figure shows that the peaks appear in three regions: (a) around -55°C (T_g of glycerol), (b) at $54\text{--}57^\circ\text{C}$, which is attributed to T_g of PLA, and (c) at $115\text{--}130^\circ\text{C}$ that corresponds to T_g of S.

TABLE 2: T_g of starch in polymer blends and composites.

PLA content in polymer blend, wt%	ABF content in composites, wt%		
	0	10	15
10	120°C	124°C	132°C
20	115°C	120°C	130°C
30	111°C	114°C	127°C

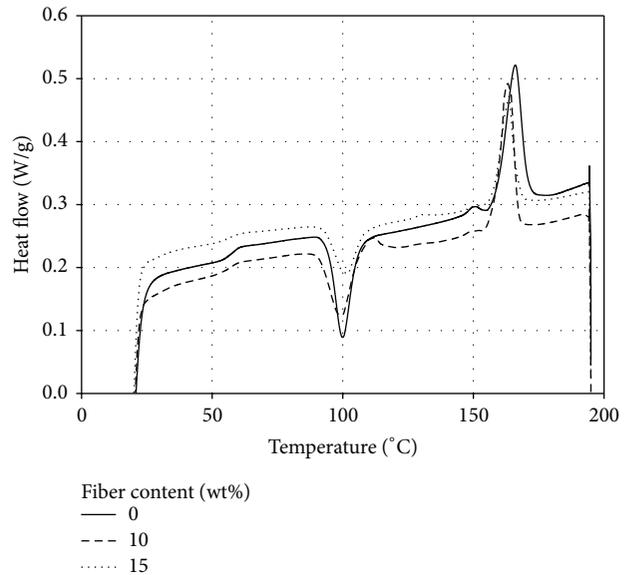


FIGURE 6: Thermograms of the polymer blend and composites containing 20 wt% of PLA.

Using $\tan \delta$ results of the polymer blend and composites containing different amounts of PLA and ABF, the T_g values of TPS are presented in Table 2. The values indicate that an increase in PLA content promotes a decrease in T_g ; such behavior can be explained by the lower T_g value of PLA compared to that of the starch ($T_g > 110^\circ\text{C}$) [3, 4]. In Table 2, it can also be noticed that for constant PLA content, there is an increase in T_g as fiber content increases; the T_g displacement can be attributed to the decrease of starch chains mobility and the H-bond interactions between ABF and TPS [7, 22, 24].

3.3. Thermal Analysis. The DSC thermogram (Figure 6) shows the thermal transitions of the polymer blend and composites containing 20 wt% of PLA. For the curves, at approximately 60°C , a change in slope can be observed due to the T_g of PLA. Those temperature values are presented in Table 3. The small decrement in T_g for the composites with respect to the pure PLA obtained here (62°C) can be attributed to glycerin migration, as Li and Huneault reported [31]. In addition, Martin and Avérous reported a small decrease in T_g of PLA by the presence of glycerol [32]. For the composites containing the larger amount of fiber (15 wt%), the T_g of PLA shows a slight decrease by enhanced PLA chain mobility. Jaafar et al. reported a decrease in T_g of PLA, which

TABLE 3: T_g of PLA in polymer blends and composites.

PLA content in polymer blend, wt%	ABF content in composites, wt%		
	0	10	15
10	57.0°C	57.1°C	54.5°C
20	57.0°C	57.3°C	55.5°C
30	57.0°C	57.0°C	56.0°C

TABLE 4: T_c of PLA in polymer blends and composites.

PLA content in polymer blend, wt%	ABF content in composites, wt%		
	0	10	15
10	100.0°C	100.7°C	99.1°C
20	99.9°C	99.8°C	101.0°C
30	98.9°C	100.4°C	100.2°C

TABLE 5: T_m of PLA in polymer blends and composites.

PLA content in polymer blend, wt%	ABF content in composites, wt%		
	0	10	15
10	163.0°C	164.5°C	161.0°C
20	165.9°C	163.6°C	163.4°C
30	166.1°C	165.9°C	163.2°C

they attributed to the inclusion of Kenaf fibers that increased PLA chains mobility because fibers separate PLA chains [33].

The exothermic peak that appears at approximately 100°C is related to PLA crystallization (T_c), as a result of the interaction of PLA and TPS, [32]. T_c values are shown in Table 4, where no noticeable effect can be seen for a fiber or PLA concentration increase (equivalent to TPS concentration decrease). Teixeira et al. reported a small decrease in the T_c of PLA using cassava bagasse [14].

For the polymeric materials of Figure 6, the small variation in the slope of the curves indicates that the T_g of S appears at 110–130°C. Those values correspond to the $\tan \delta$ peaks shown in Figure 5.

Figure 6 also shows peaks between 110 and 130°C due to T_g of starch, which coincide with the T_g obtained by mechanodynamic tests, which confirms that increasing fiber content increases T_g .

The endothermic peaks at the high temperature zone correspond to PLA melting temperature (T_m). The peak temperature values presented in Table 5 show that slight changes in PLA melting temperature correspond to an increase with PLA content and a decrease with ABF addition. Such decrease can be explained by the interference that fibers may cause to allow PLA crystallization. Before the PLA melting peaks, the correspondent small shoulder is attributed to rearrangement of lamellar fractions formed during PLA crystallization [34], or premelting of small crystals.

3.4. Mechanostatical Tests. Even though mechanodynamic tests allowed the thermomechanical characterization of

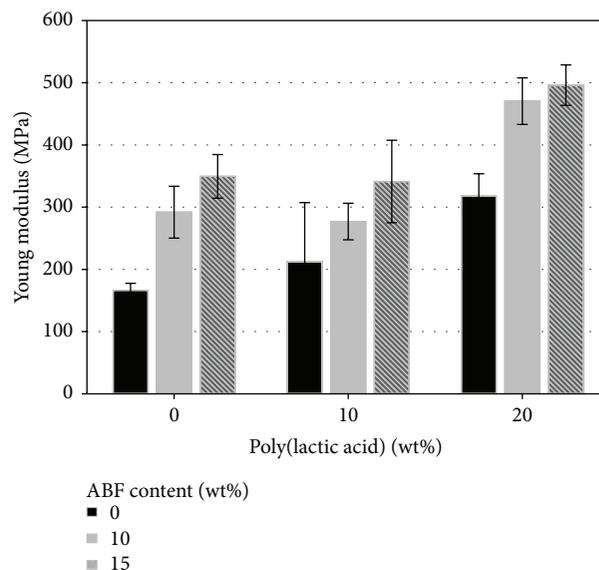


FIGURE 7: Young modulus of polymer blends and composites at 25°C, varying PLA content.

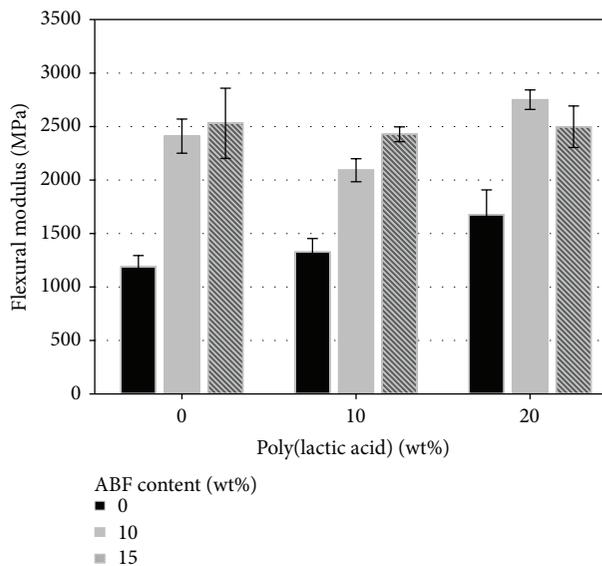


FIGURE 8: Flexural modulus of polymer blends and composites at 25°C, varying PLA content.

extruded materials, mechanostatical tests of some of the composites are included to confirm their moduli values pattern, as well as to determine impact resistance behavior. In Figure 7 Young modulus of polymer blends and composites as a function of PLA and fiber content is shown. The inclusion of PLA and/or ABF promotes an increase in modulus. Those patterns are in agreement with the storage modulus values obtained at 25°C (Figure 4).

Figure 8 shows flexural modulus of the polymeric materials as a function of PLA and fiber content. For this parameter, an increase in PLA and/or fiber content leads to composites

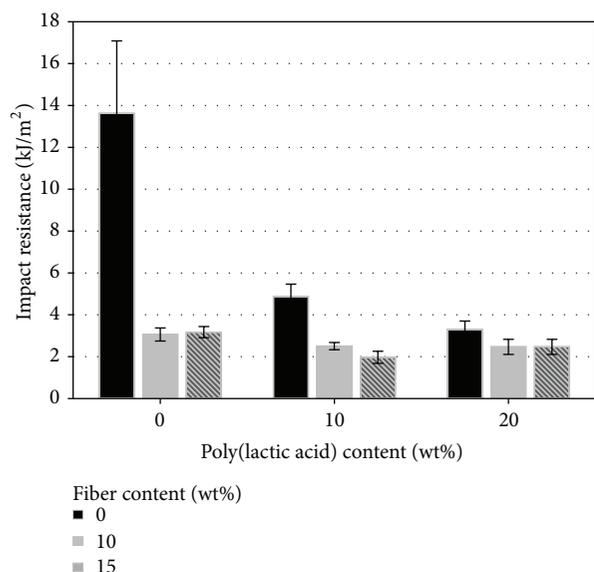


FIGURE 9: Impact resistance of polymer blends and composites at 25°C varying PLA content.

with higher flexural modulus, supporting the results obtained for the other moduli.

In Figure 9, it can be noticed that the increase in rigidity of the TPS caused by the addition of PLA and/or ABF leads to a large decrease in impact resistance.

3.5. Morphology. In Figure 10, micrographs of fractured samples of (a) TPS, (b) polymer blend containing 20 wt% of PLA, and (c) 80 TPS/20 PLA w/w, with 15 wt% ABF composite are shown. In Figure 10(a), uniform gelatinized starch granules can be observed. Figure 10(b) shows a stratified brittle surface. The fibers in the composite can be seen in Figure 10(c), and a rigid fracture is noticed.

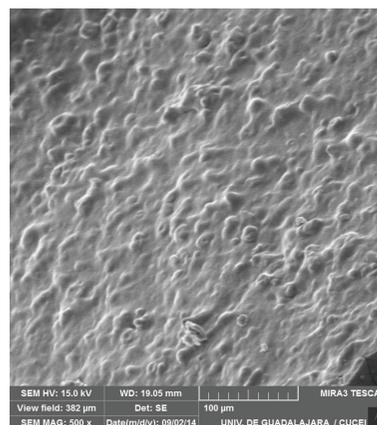
4. Conclusions

Blends of TPS and PLA and composites of TPS/PLA/ABF prepared by extrusion followed by compression molding were characterized. The reinforcing effect of PLA and/or ABF in TPS led to an increase in moduli and a decrease in moisture absorption and impact resistance. The TPS glass transition temperature increased with ABF content and decreased with PLA content.

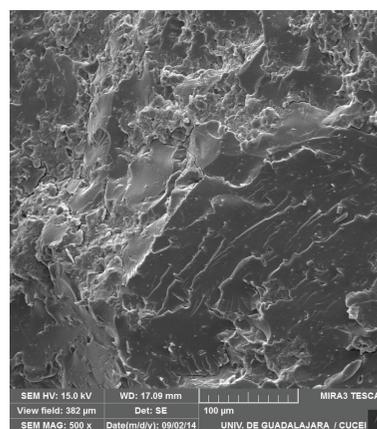
The reinforcing effect of PLA was enhanced by the incorporation of ABF, although the reduction in impact resistance is not convenient. That kind of behavior is expected generically for rigid materials; nevertheless, the wood appearance and biodegradability, along with the increase in moduli as well as thermal resistance, and the decrease in water absorption justify the production of this type of composites for many applications.

Conflict of Interests

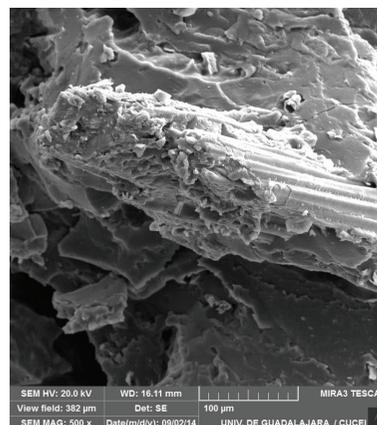
The authors declare that there is no conflict of interests regarding the publication of this paper.



(a)



(b)



(c)

FIGURE 10: SEM images of polymeric materials: (a) TPS, (b) polymer blend containing 20 wt% PLA, and (c) 80 TPS/20 PLA w/w, with 15 wt% ABF composite.

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