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Mechanical, thermal and morphological characterization of recycled LDPE/corn starch blends

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Abstract

Recycled LDPE/corn starch blends containing 30, 40 and 50 wt% starch were prepared by extrusion and characterized by the melt flow index (MFI), tensile test, dynamic mechanical thermal analysis (DMTA) and scanning electron microscopy (SEM). For comparison, virgin LDPE/corn starch blends were prepared and characterized under the same conditions. The addition of starch to LDPE reduced the MFI values, the tensile strength and the elongation at break, whereas the modulus increased. The decreases in the MFI and tensile properties were most evident when 40 and 50 wt% were added. SEM showed that the interfacial interaction was weak for blends containing virgin and recycled LDPE. Blends prepared with recycled LDPE showed the same behavior as those blends prepared with virgin LDPE, indicating that starch was the main factor that influenced the blend.

Keywords: Biodegradable polymers; Low density polyethylene; Starch; Polymer blends; Recycling

1. Introduction

Polyolefins, such as high density polyethylene (HDPE), low density polyethylene (LDPE) and polypropylene (PP), constitute the majority of thermoplastics currently used as packaging materials. Since the use of these plastics is continuously increasing, the problem of post-consumer recycling of these materials has become an important issue for economic and environmental reasons (Pracella, Pazzagli, & Galeski, 2002; Ishiaku et al., 2002). However, in contrast to glass and metals, which can be recycled into products with properties essentially similar to those of the primary materials, each time polymers are reprocessed, they lose some of their physical and mechanical properties because of peroxidation (Scott, 2000). In addition, recycling would be neither practical nor economical for certain applications such as bags, agricultural mulch films, and food packaging, since these artifacts contain many organic residues and have a low lifetime. For these applications, it would be better if the plastics degraded into safe by-products under normal composting conditions (Aburto et al., 1997).

The blending of biodegradable polymers, such as starch, with inert polymers, such as polyethylene, has received considerable attention. The reasoning behind this approach is that if the biodegradable component is present in sufficient amounts and if it is removed by microorganisms in the waste disposal environment, the plastic or film containing the remaining inert components should disintegrate and disappear (Chandra & Rustgi, 1998). Thus, blends of recycled LDPE with corn starch would have two environmental advantages: (a) to substitute the virgin synthetic thermoplastic matrix by post-consumer materials, and (b) the end product would be biodegradable and cheap.

Among the recycling technologies currently available, mechanical recycling, i.e. direct reprocessing in the melt phase, is the most effective, economical and reliable method for dealing with plastics. However, mechanically recycled plastics have less desirable properties when compared to the same articles manufactured from virgin materials (Ehrig, 1992; Kartalis, Papaspyrides, & Pfaendner, 2000;

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Pedroso, Mei, Agnelli, & Rosa, 1999). The irreversible chemical changes that occur in the polymer structure during its processing and lifetime are responsible for the deterioration of the physical properties and mechanical performance of recycled polymers (Bonelli, Martins, Mano, & Beatty, 2001; Kartalis et al., 2000; Scott, 2000). In addition, reprocessing increases the degradation of post-use material because of mechanochemical and oxidative reactions.

Starch is a natural polymer found in granular form in a variety of plants, such as corn and cassava (Carioca, Arora, Selvam, Tavares, & Kennedy, 1996). Starch is a blend of amylose and amylopectin, both of which are composed of α-D-glucopyranosyl units $(C_6H_{10}O_5)_x$. Native starch granules swell after absorbing water through hydrogen bonding with their free hydroxyl groups, but they still retain their order and crystallinity. However, when these swollen granules are heated, the hydrogen bonding between adjacent glucose units is disrupted and the crystallinity is progressively destroyed. This process is known as gelatinization (St-Pierre, Favis, Ramsay, Ramsay, & Verhoogt, 1997). The addition of granular starch to LDPE has various effects on polymer properties, i.e. the modulus increases because of the granules' stiffening effect, and the tensile strength and elongation decrease as the starch content increases (Jang, Huh, Jang, & Bae, 2001; St-Pierre et al., 1997). According to Jane, Schwabacher, Ramrattan, and Moore (1992), the carboxylic group and the ketone group of oxidized PE react with the hydroxyl groups of starch to form bonds among the components of the LDPE/starch blend.

In this study, we compared the thermal, mechanical and dynamic mechanical thermal properties, the melt flow index and the morphology of LDPE/starch blends prepared from virgin and recycled LDPE.

2. Experimental

2.1. Materials

Virgin low density polyethylene (LDPE), PB 526, melt flow index 0.25 g/10 min (190 °C, 2.16 kg) was supplied by Braskem (Triunfo, RS, Brazil). Post-consumer LDPE, in milled form, was supplied by EcoQuali (Várzea Paulista, SP, Brazil). Pre-gelatinized corn starch (RD 337) was supplied, in dried powder form, by Corn Products Brazil—Ingredientes Industriais Ltda. (Jundiaí, SP, Brazil), and contained 27 wt% amylose and 73 wt% amylopectin, with a number average molecular weight (M_n) of 486,000 g/mol. Fig. 1 illustrates morphology and particle size of the granular starch (Fig. 1a) and the pregelatinized starch (Fig. 1b).

2.2. Recycling of post-consumer LDPE

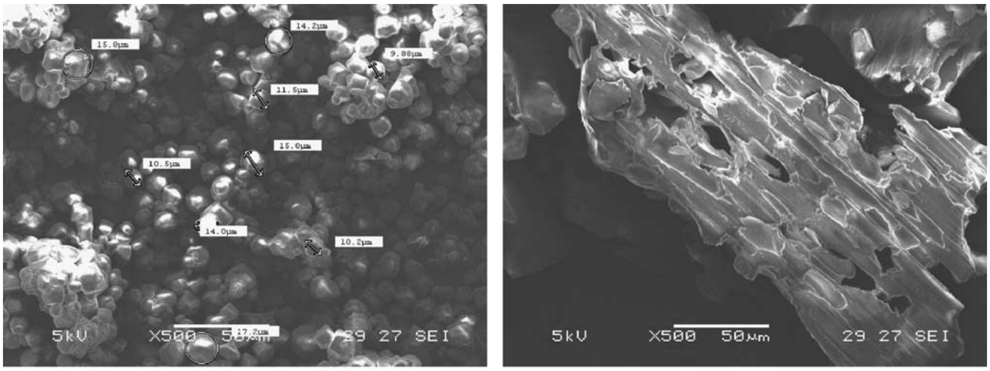
The post-consumer plastics from separate collections of LDPE packaging films were supplied by EcoQuali (Várzea Paulista, SP, Brazil). The material was initially washed with water at approximately 70 °C to remove non-polymeric residues and was then ground to obtain smaller particles and better homogeneity. The ground plastic was subsequently agglutinated using an equipment with a rotating cylinder that heated the ground plastic through friction transforming it into a paste and then, small quantity of water was added to cause a sudden cooling.

2.3. Blend preparation

Blends containing 30, 40 and 50 wt% starch were prepared with a co-rotating twin-screw extruder (Werner & Pfleiderer, model ZSK 30). LDPE and starch were dried in an air-circulating oven at 70 °C for 1 h before extrusion. LDPE was fed in the first zone and the starch was fed in the third zone. The thermal profile was 140–160–160–160–160–150 °C (zones 1–6, respectively) and the screw speed was 200 rpm. Recycled polyethylene, without starch, was extruded under the same conditions used to prepare blends of LDPE with starch. The materials were extruded and granulated.

2.4. Molding

Virgin and recycled LDPE and the blends were compression molded into sheets (180 mm × 180 mm × 2 mm)



(a) (b)

Fig. 1. Granular starch (a) and pregelatinized starch (b). 500×.

using a model MA 098 Marconi Press (Marconi—Equipamentos e Calibração para Laboratórios, Piracicaba, SP, Brazil). The mold containing the desired material was initially placed in the press and heated for 3 min without applying any pressure, in order to ensure uniform heat flow throughout the material. For all of the blends the temperature was kept at 170±5 °C for 3 min at 10 t. The resulting sheets were cooled to room temperature.

2.5. FTIR spectroscopy

The FTIR measures were carried out on a Perkin Elmer Spectrum One spectrometer coupled to an Auto Image light microscope. These analyses were performed on samples of molded sheets of the virgin and recycled polymers.

2.6. Melt flow index (MFI)

MFI measurements of LDPE and the blends were obtained using a model MI-1 plastometer (DSM Instrumentação Científica Ltda., São Paulo, SP, Brazil), according to ASTM D 1238 (procedure A, 190 °C/5 kg).

2.7. Tensile properties

The tensile properties were determined with an EMIC model DL 2000 universal testing machine (EMIC Equipamentos e Sistemas de Ensaio Ltda., São José dos Pinhais, PR, Brazil) using specimens (type IV) cut from compression molded sheets, according to the ASTM D-638 standard. The control program used was Mtest LBP version 3.00 and the load cell had a capacity of 200 kgf, at a rate of 20 mm/min. The average and standard deviations of the tensile strength, elongation at break and Young's modulus were determined for each formulation.

2.8. Differential scanning calorimetry (DSC)

Thermal analysis was done using a model 204 TASC 414/3A differential scanning calorimeter (DSC) (Netzsch-Gerätebau GmbH, Bavaria, Germany) under a nitrogen atmosphere, at a heating rate of 10 °C/min. Two heating cycles were used for each sample. The samples were first heated from room temperature to 160 °C to eliminate their thermal history, and then cooled to room temperature and immediately reheated to 160 °C. The second scan was done at the same heating rate. All DSC experiments were done in duplicate and the thermograms shown refer to the second heating.

The degree of crystallinity of LDPE was obtained from the ratio between the fusion heat of the samples (ΔH) and the fusion heat of 100% crystalline LDPE (277.1 J/g) (Brandrup & Immergut, 1975).

2.9. Dynamic mechanical thermal analysis (DMTA)

Dynamic mechanical thermal analysis (DMTA) was done using a dynamic mechanical analyzer, model MK III (Rheometric Scientific, Inc., Piscataway, NJ, USA) over the temperature range of -40 to 110 °C at a frequency of 1 Hz. The heating rate was 5 °C/min. The analyses were done in duplicate using specimens $30\times10\times1.5$ mm.

2.10. Scanning electron microscopy (SEM)

Specimens were fractured after freezing in liquid nitrogen and micrographs of the fractured surfaces were obtained using a JEOL model JSM-5900LV scanning electron microscope. The dispersed phase and the interfacial interaction of the LDPE/starch blends were analyzed.

3. Results and discussion

3.1. FTIR spectroscopy

Since the formation of carbonyl (C=O) is due to degradation process of polyethylene, the degradation of recycled polyethylene was investigated using FTIR spectroscopy and was compared with virgin polyethylene. Carbonyl index was calculated on the basis of the relative intensities of the carbonyl band at 1715 cm^{-1} to that of methylene scissoring band at 1464 cm^{-1}. The carbonyl index values obtained for virgin and recycled LDPE were 0 and 0.068, respectively. Fig. 2 shows the spectrum of recycled LDPE.

There was no degradation of virgin LDPE, whereas the carbonyl index for recycled LDPE showed a certain amount of polymer degradation. Recycled LDPE showed accentuated peaks in the range of 1740–1715 cm^{-1} (due to the carbonyl group) and another at 1640 cm^{-1} (due to double bonds) (Chandra & Rustgi, 1997), indicating an amount of degradation.

The level of degradation seen here was similar to that of recycled HDPE reported by Cruz and Zanin (2003). This behavior is desirable since the carbonyl groups present in recycled LDPE will favour a better interaction with the polar groups of starch (Jane et al., 1992).

3.2. Melt flow index (MFI)

Recycled LDPE had a higher MFI (0.29±0.01) than virgin LDPE (0.24±0.01) when the measurements were done using a load of 2.16 kg. Since the MFI is an indirect measurement of material viscosity, these results indicated that recycled LDPE had a lower viscosity, which would be explained based on the decrease in molecular weight during degradation. It was not possible to determine the MFI of the starch blends using a load of 2.16 kg since their viscosity were so high that an excessively long period would be

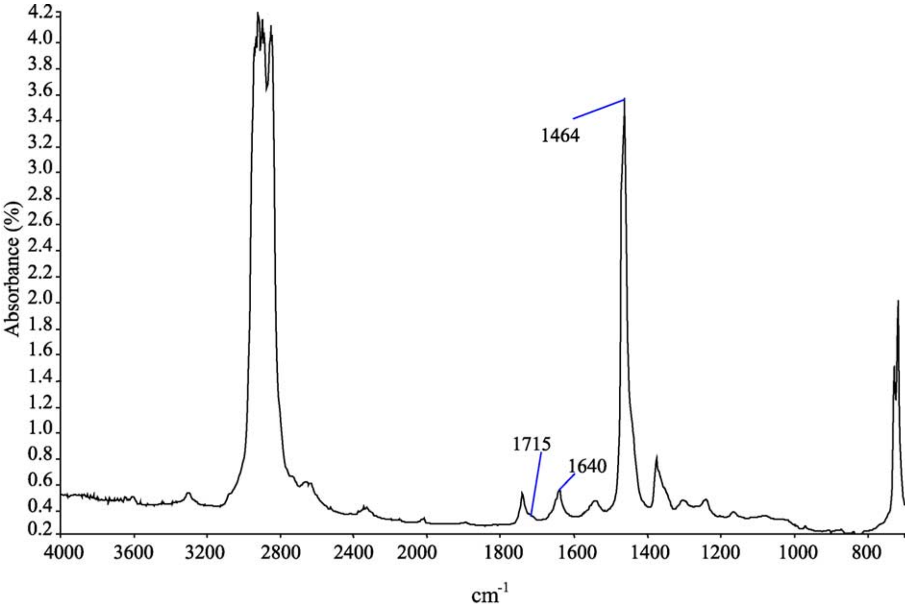


Fig. 2. FTIR spectrum for recycled LDPE.

needed before any measurements could be done. We therefore opted to use the same temperature (190 °C), but with a higher load (5 kg). Fig. 3 shows the MFI values for virgin and recycled LDPE and their blends with starch under these conditions.

The MFI values for virgin and recycled LDPE (190 °C/5 kg) were not significantly different. These values decreased with increasing starch content, independently of the LDPE used in the blends. Since the MFI is an indirect measurement of viscosity, it follows that the starch must act as rigid filler since the main effect of rigid fillers is to increase the elastic modulus of a composite or the viscosity of a fluid suspension (Nielsen, 1974). The recycled LDPE/starch blends had lower MFI values than virgin LDPE/starch blends. This difference could be attributed to the higher interfacial interaction between recycled LDPE and starch since the carboxylic and ketone groups of the degraded PE may react with the hydroxyl groups of starch to form bonds between these components (Jane et al., 1992). In addition, crosslinking of LDPE may have occurred during blend processing, as a result of degradation induced during recycling (Kartalis et al., 2000).

3.3. Tensile tests

Fig. 4 shows the tensile strength (σ) values for virgin and recycled LDPE and their blends with starch. The lower values for pure recycled LDPE and blends with recycled LDPE indicated the degradation of recycled LDPE. The tensile strength of all LDPE/starch formulations decreased with increasing starch content, indicating that corn starch behaved as a non-reinforcing filler. The tensile strength decreased 57, 60 and 74% in the blends of virgin LDPE containing 30, 40 and 50 wt% starch, respectively,

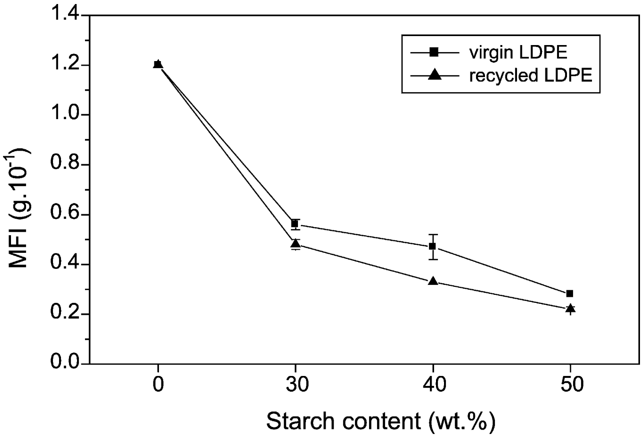


Fig. 3. MFI for virgin and recycled LDPE and their blends with starch. The points are the mean ± SD for five determinations.

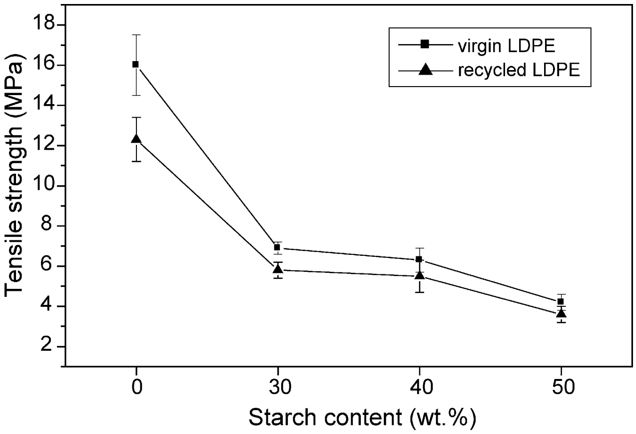


Fig. 4. Tensile strength for virgin and recycled LDPE and LDPE/starch blends. The points are the mean ± SD for five determinations.

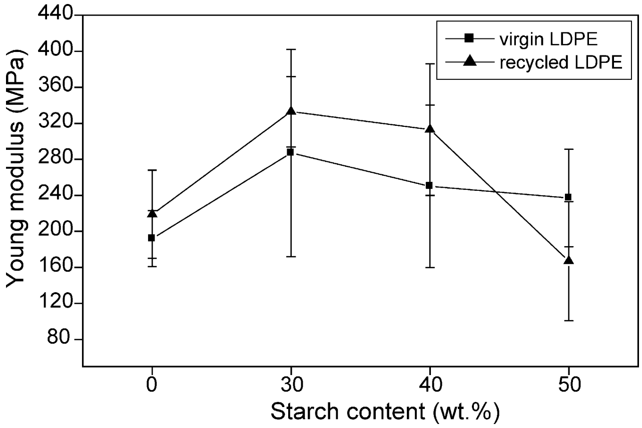


Fig. 5. Young modulus for virgin and recycled LDPE and LDPE/starch blends. The points are the mean ± SD for five determinations.

compared to pure LDPE. The corresponding decreases for blends with recycled LDPE were 53, 55 and 71%, compared to pure recycled LDPE. Thus, the use of recycled LDPE had no significant effect on the tensile strength of blends with 40 and 50 wt% starch. A possible explanation for the decrease in tensile strength after the addition of starch to PE, could be the low interfacial interaction between the components of the blend, which would lead to mechanical rupture at the blend interface.

There was no significant difference between the Young modulus values of pure virgin and pure recycled LDPE (Fig. 5). The higher average value of the Young modulus for recycled LDPE, which underwent degradation during processing and/or its normal service life, probably reflected the reactions induced by radicals exposed during reprocessing, that led to crosslinking and branching along the chains (Kartalis et al., 2000). As a result, the recycled polymer became more rigid and its Young modulus increased.

When starch was added to PE, the Young modulus showed high standard deviations expected of materials in which the heterogeneity of the specimens is higher than that of the monodisperse materials. The Young modulus values increased when 30 wt% starch was added, independently of the LDPE used. In general, the modulus is closely related to the rigid domains of the material (Jang et al., 2001). For starch contents greater than 30 wt%, the Young modulus tended to decrease, probably because of the lower rigidity associated with starch agglomerates. When 30% and 40 wt% starch was added to LDPE, the Young modulus values were higher for the blends containing recycled LDPE. However, when 50 wt% starch was added to LDPE, the Young modulus was higher for the blend containing virgin LDPE.

Comparison of the pure polymers (virgin and recycled) showed that elongation at break was lower for recycled LDPE (Fig. 6a), probably because the degradation induced by radical chains reactions resulted in crosslinking and branching along the polymer chains during reprocessing (Dintcheva, Jilov, & La Mantia, 1997; Kartalis et al., 2000).

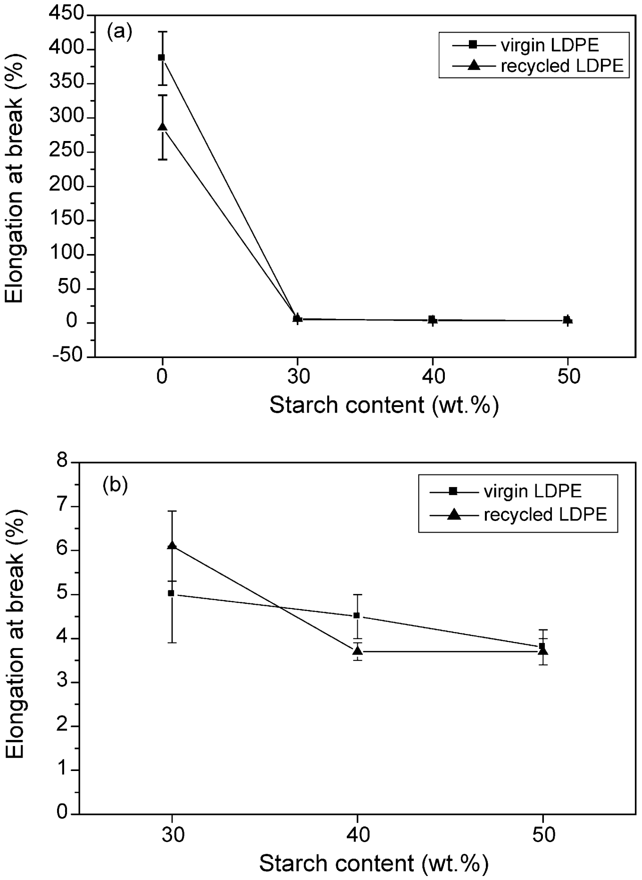


Fig. 6. Elongation at break for virgin and recycled LDPE and LDPE/starch blends. The points are the mean ± SD for five determinations.

Fig. 6b shows that the elongation at break decreased as the starch content increased. The addition of starch granules to LDPE produced the general trend for filler effects on polymer properties; i.e. the modulus increased through stiffening of the granules and elongation decreased as the starch content increased (St-Pierre et al., 1997). In synthetic polymer blends, the addition of a second immiscible phase to a ductile matrix material usually significantly decreases the elongation at break. In synthetic polymer blends with a ductile matrix, the elongation at break is considered to be highly sensitive to the interfacial interaction between the phases of the blend (St-Pierre et al., 1997).

Aburto et al. (1997) observed that LDPE/modified starch blends retained satisfactory elongation at break values up to 25 wt% octanoated starch, while in blends with plasticized starch the maximum tolerated value was 15 wt%; beyond this level, there was a drastic decrease in the elongation at break. St-Pierre et al. (1997) prepared blends of PE with gelatinized and plasticized starch. The Young modulus and the tensile strength at break showed a small decrease with increasing in starch content. The elongation at break decreased drastically when the starch content exceeded 10 wt%. The highest starch content used by these authors was 22 wt%.

In all starch blends, the values of elongation at break fell drastically compared to those of pure PE (Fig. 6a), and the slight variations among blends with different starch contents were not significant (the elongation at break for the blends varied by 3–5.5% while the average elongation at break for pure polyethylenes was 340%).

3.4. *Differential scanning calorimetry (DSC)*

Figs. 7 and 8 show the DSC curves obtained during heating and cooling, respectively, of the polyethylenes and their blends. Table 1 shows the average values for the melting temperature (T_m), crystallization temperature (T_c), fusion enthalpy (ΔH_f), crystallization enthalpy (ΔH_c) and degree of crystallinity (X_c) for virgin and recycled LDPE.

Recycled LDPE showed two main melting peaks (at about 112 and 125 °C), indicating that the material was a blend of polymers. The two peaks suggested the presence of LDPE (melting temperature at about 110 °C) and linear low density polyethylene (LLDPE) (melting temperature at about 120 °C) (Dintcheva et al., 1997). LLDPE is normally blended with LDPE to obtain films (Dintcheva et al., 2001).

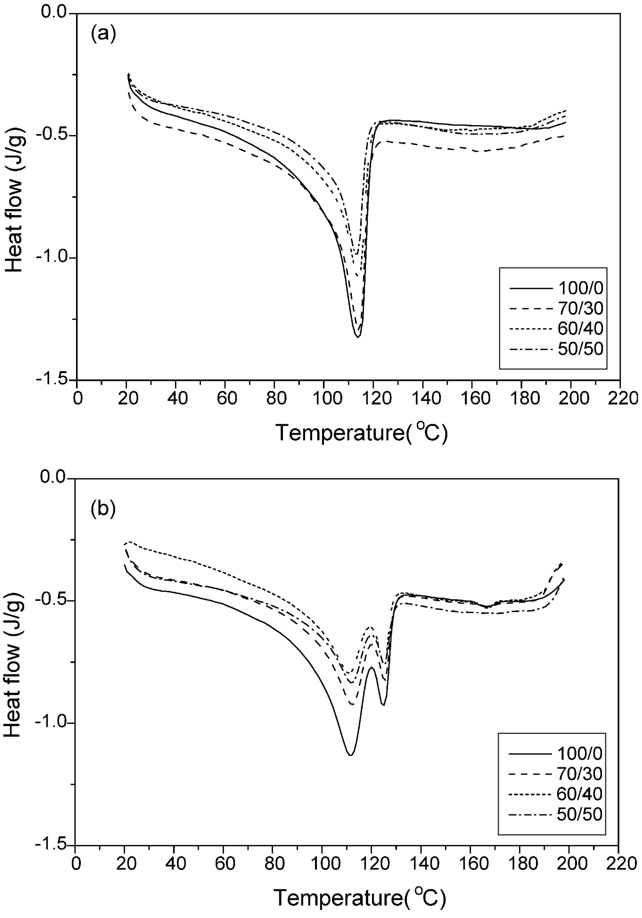


Fig. 7. DSC curves (heating) for virgin and recycled LDPE and LDPE/starch blends. (a) Virgin LDPE and their blends and (b) recycled LDPE and their blends.

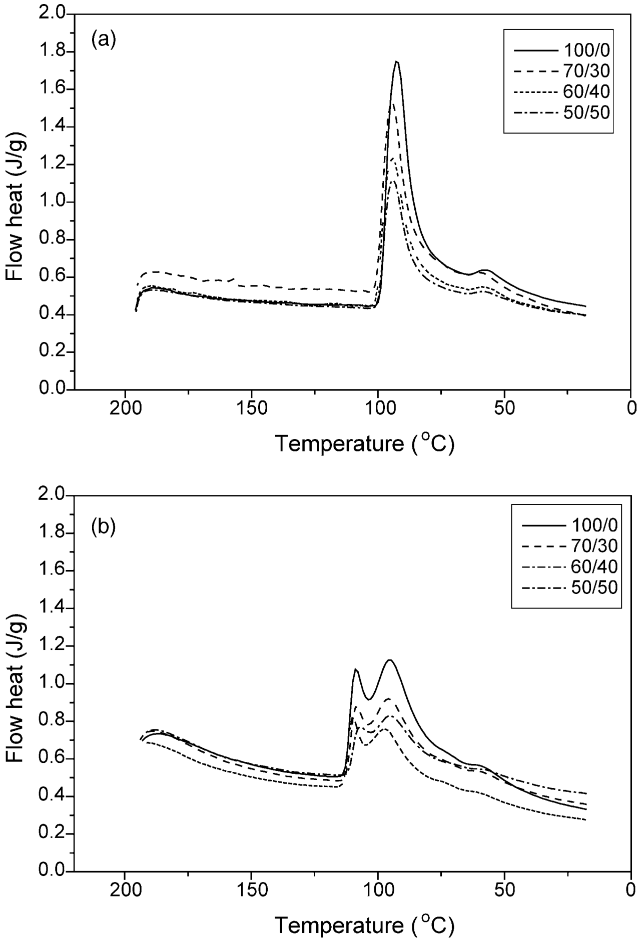


Fig. 8. DSC curves (cooling) for virgin and recycled LDPE and LDPE/starch blends. (a) Virgin LDPE and their blends and (b) recycled LDPE and their blends.

Virgin polyethylene had a melting temperature (T_m) of about 114 °C. During cooling, recycled LDPE showed two crystalization peaks due to the second component, as discussed above. Virgin material showed a T_c at about 92 °C.

The ΔH_m and ΔH_c values for recycled polyethylene (Table 1) refer to the total area of each curve, i.e. enthalpy relative to LDPE and another polymer, probably LLDPE. However, if one considers only the portion of the curve related to LDPE, the ΔH_m and ΔH_c values were lower compared to virgin polyethylene, indicating that recycled LDPE had a lower degree of crystallinity than virgin LDPE. As shown by the FTIR spectrum, degradation of the recycled material probably generated radical reactions that caused crosslinking of the LDPE and reduced its crystallinity.

The melting and crystallization temperatures of the blends were the same as for the pure polymers, which indicated incompatible blends. In addition, there was no significant decrease in the crystallinity of LDPE in the blends, indicating that LDPE was immiscible with the starch or that the LDPE/starch interactions were weak. Similar results were obtained by Aburto et al. (1997), for blends of PE with modified starch

Table 1
Melting temperature (T_m), crystallization temperature (T_c), fusion enthalpy (ΔH_f), crystallization enthalpy (ΔH_c) and crystallinity degree (X_c) for virgin and recycled LDPE

	T_m (°C)	ΔH_m (J/g)	T_c (°C)	ΔH_c (J/g)	X_c (%)
Virgin	114	115	92	116	42
Recycled	112; 125	107	108; 95	109	39

3.5. Dynamic mechanical thermal analysis (DMTA)

The glass transition values, T_g, can be defined as the temperature where: (1) the loss tangent (tan δ) is maximum; (2) the loss modulus (E'') is maximum, and (3) the inflexion point corresponds to a significant drop in the storage modulus (E') (Nielsen, 1974). The viscoelastic behavior of the polyethylenes and their blends was studied by DMTA. Fig. 8a–c shows E' vs T, E'' vs T and tan δ vs T, respectively, for virgin and recycled polyethylenes.

Recycled LDPE had higher E' values, i.e. higher rigidity, than virgin LDPE (Fig. 9a). These higher E' values for recycled LDPE could reflect the crosslinking or chain scission (increase in crystallinity) caused by the degradation seen in the FTIR results.

Virgin LDPE had a higher E''_{maximum} value than recycled LDPE (Fig. 9b). Loss modulus (E'') is related to the loss of energy as heat, i.e. to viscous behavior (irreversible deformation). The lower E''_{maximum} value implies higher elastic recovering, which can probably be attributed to higher polymer rigidity caused by the crosslinking resulting from degradation.

Assuming that the T_g is the temperature where E'' is maximum, the polyethylenes showed a transition in the range of −10 to 0 °C, that was attributed to β relaxation (relaxation of branches) (Bikiaris et al., 1999). Although the polyethylenes had similar ranges of relaxation, the β relaxation of recycled LDPE tended to occur at a higher temperature and to have a broader transition range. Again, this can be attributed to crosslinking and branching caused by degradation (Kartalis et al., 2000) and also to second polymer phase, seen in the DSC results.

Fig. 9c shows that the temperature in which tan δ was maximal for virgin LDPE was at about 90 °C, and was related to α relaxation, first described as vibrational and reorientational motion within the crystals. Later, this relaxation was interpreted as relaxation of the constrained molecules with reduced mobility close to crystallites (Bikiaris et al., 1999).

Fig. 10a shows that addition of starch to virgin LDPE generally caused a shift in the E' curves to higher temperatures, indicating that starch decrease the polymer chain mobility. The E' values increased with a starch content up to 40 wt% but then decreased with 50 wt% starch. These results corroborate with Young modulus results; this behavior was not observed for recycled polyethylene (Fig. 10b).

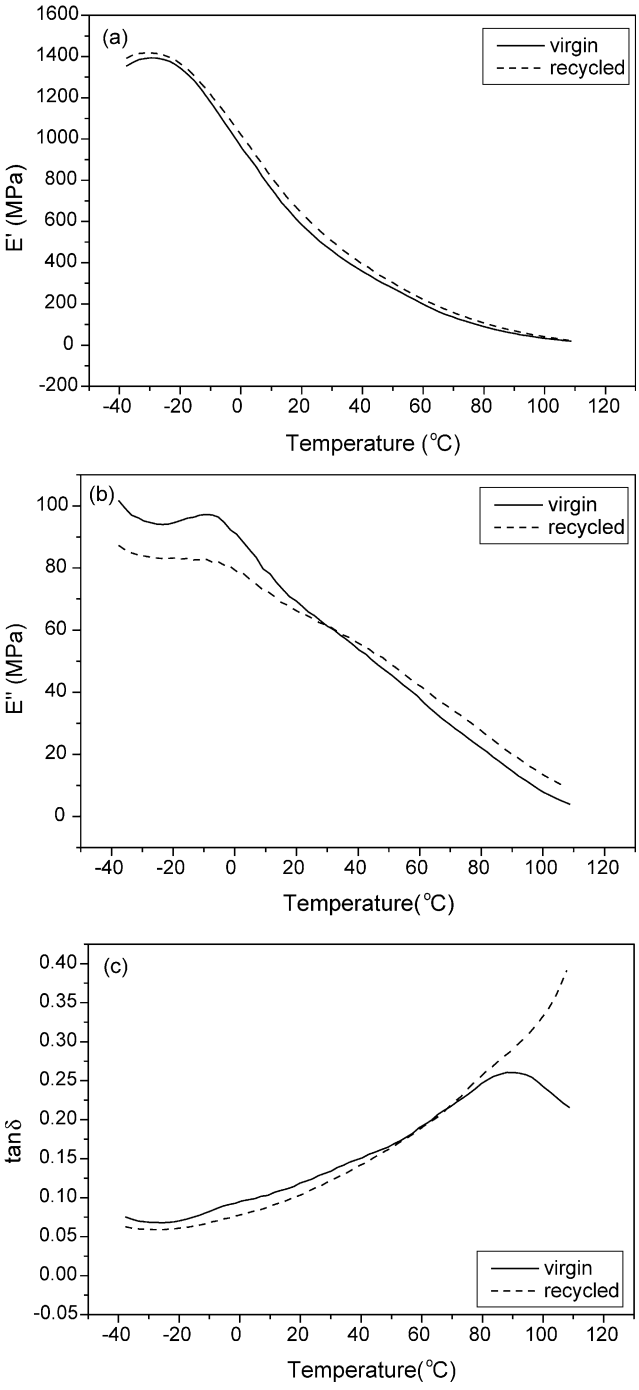


Fig. 9. DMTA curves for virgin and recycled LDPE.

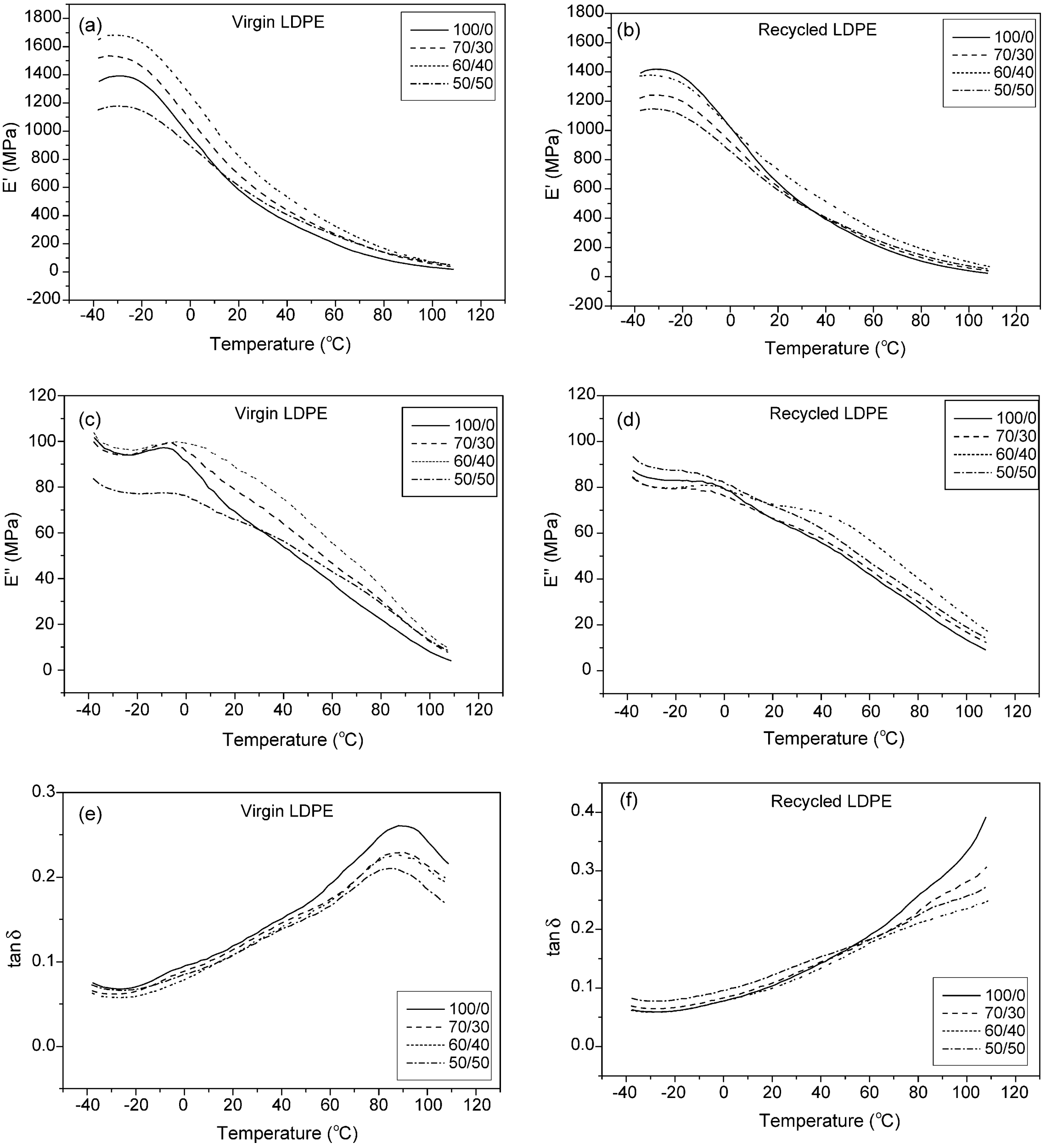
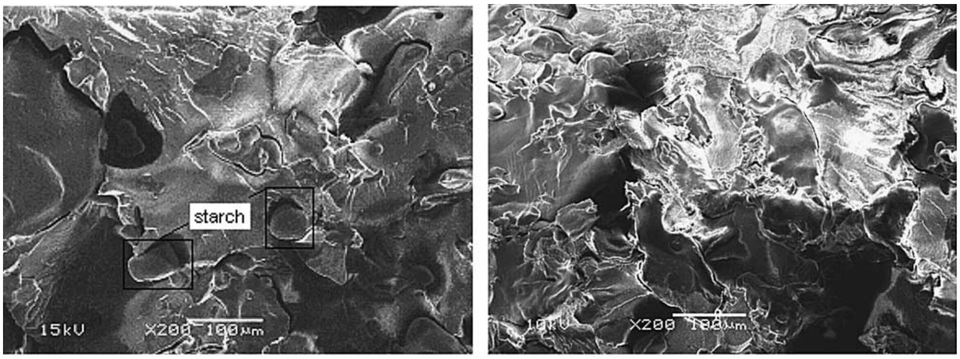


Fig. 10. DMTA curves for virgin and recycled LDPE and LDPE/starch blends.



(a) (b)

Fig. 11. SEM images of LDPE/starch blends. (a) Virgin LDPE/30%, (b) recycled/30%. (200×).

Fig. 10c and d shows that for all blends, E'' decreased slowly up to −25 °C, followed by the appearance of a peak. This peak was attributed to β relaxation, which has properties of the glass–rubber transition (Bikiaris et al., 1999). In general, the addition of starch caused a shift of the E''_{maximum} to a higher temperature, indicating that there was an increase in the relaxation temperature.

Fig. 10e shows that the peaks where tan δ was maximum broadened and shifted to the right when starch was added to LDPE. For blends with recycled LDPE, it was not possible to determine the temperature where tan δ was maximum since only a shoulder in the range 80–85 °C was observed (Fig. 10f).

3.6. Scanning electron microscopy (SEM)

Fig. 11a and b shows SEM images of LDPE/starch blends, with virgin and recycled LDPE containing 30 wt% starch. The lack of adhesion between LDPE and starch seen on the fracture surfaces indicated a poor interfacial interaction and could explain the decrease in the mechanical properties following the addition of starch. As the content starch increased, filler–filler interactions also increased. This could also account for the deterioration in physical properties since the transfer of stress from the matrix to the filler was less effective.

Although both blends, with virgin and recycled polyethylene, showed a lack of interfacial adhesion, the blend containing recycled LDPE showed a improved dispersion of starch.

4. Conclusion

Blends of LDPE with starch had a higher Young modulus and lower tensile strength and elongation at break than pure LDPE. The addition of starch to LDPE reduced the MFI, the tensile strength and the elongation at break, whereas the modulus increased. The decrease in the MFI and tensile properties were most evident for 40 and 50 wt% starch. SEM showed that the blends were immiscible and that the interfacial interaction was weak, regardless of whether virgin or recycled LDPE was used. Blends prepared with recycled LDPE had the same tensile strength as blends prepared with virgin LDPE since starch was the main factor that influenced blend properties. Thus, the use of recycled LDPE had no significant effect on the tensile strength of blends with 40 and 50 wt% starch, making this polymer a suitable substitute for the virgin LDPE.

Acknowledgements

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