Reusing Surlyn® Ionomer Scraps in LDPE Blends: Mechanical and Thermal Properties

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This study aimed to evaluate the possibility of reusing industrial scraps of Surlyn® ionomer by obtaining blends with Low-Density Polyethylene (LDPE). Blends of LDPE and Surlyn® scraps were obtained by extrusion with compositions ranging from 25 to 75 wt% of the ionomer. Their melt flow index (MFI), morphology (SEM), mechanical (tensile, flexural, impact tests) and thermal properties (DSC, TGA, HDT tests) were analyzed. The morphology of the blends presented two phases, indicating the immiscibility of phases. Surlyn® incorporation promoted a decrease in the degree of crystallinity of LDPE and a slight increase in the thermal decomposition temperature. In addition, Surlyn® decreased the decomposition rate of LDPE. However, the decrease in the degree of crystallinity did not affect the mechanical properties of the blends. Incorporating ionomer in LDPE promoted an increase in tensile and flexural strength, tensile and flexural modulus and strain at break. Impact strength decreased with increasing ionomer concentration.

Keywords: *scraps reuse, ionomer, low density polyethylene, blends, mechanical properties.*

1. Introduction

Ionomers are polymers having ionic functional groups pendant to the main chain. These functional groups tend to form ionic aggregates in the polymeric matrix, which restrict the mobility of the adjacent regions, acting as cross-linking points. These cross-linking points can be overcome with increasing temperature. Once the temperature is lowered, its physical cross-linking returns. This phenomenon has made ionomers known for having "reversible physical cross-linking[1-5]."

Surlyn® is the trademark of a family of ionomers of (ethylene-methacrylic acid copolymer), EMAA, manufactured by DuPont. Surlyn® PC2000 is a copolymer of which the methacrylic acid groups, MAA, have been partially neutralized with sodium salt and is widely used in cosmetic packaging due to its high transparency, providing aesthetic resemblance to glass[6]. However, this ionomer has a limitation of primary mechanical recycling due to the loss of its transparency in reprocessing, which can generate black spots and yellowing of the injected components. This behavior makes it impossible to reuse in new packaging and, therefore, increases the amount of waste generated, causing detrimental environmental impact[7].

An alternative approach to replacing the disposal of the polymer is to reuse it by obtaining new polymeric blends with virgin polymers, combining economic interest and environmental benefits[7-9], since primary recycling of polymers is considered the best way for recycling polymers because it uses less energy and resources[10].

Among all the polymers, polyethylene (PE) is one of the most widely used polyolefins polymers due to its low cost, high flexibility, low-temperature toughness, ease of processing, transparency, lightweight, and versatility of applications in various industrial sectors such as packaging, agricultural, biomedical and energy[11-15].

Studies using ionomers as a compatibilizer in PE blends and nanocomposites reported the systems' enhancement in compatibility and mechanical performance[11,16-20]. Further studies have been performed on the surface properties, morphology and mechanical properties of PE/ionomer blends[13,14,21-23].

Horrion and Agarwal[21] evaluated the thermal and rheological properties of low-density polyethylene LDPE/ionomer blends with different ionomer concentrations and observed that the blends are immiscible and the time-temperature superposition principle is not entirely applicable. Suh et al.[23] reported that the incorporation of Surlyn® in PE resulted in a remarkable improvement in cable insulation's electrical properties (breakdown strength).

Cho et al.[22] studied the high-density polyethylene (HDPE)/ionomer blends with different ionomer concentrations ranging from 10 to 90 wt% and observed that the blend is immiscible in all compositions and the tear strength, tensile strength and elongation at break of the blends exhibit negative deviation from the linear additivity property. The authors attributed the lower mechanical behavior of the mixtures to the immiscibility and poor interfacial adhesion between components caused by the introduction of polar groups into the polyethylene chains of the ionomer.

Ali et al.[13] prepared PE/ionomer Surlyn® (90/10) films with tunable surface properties by manipulating the processing parameter during film blowing to provide an alternative method to prepare large-scale PE films with polar functional groups. The film surface exhibited an increase in roughness caused by the Surlyn® enrichment on the surface, which helped to increase the film hydrophilicity.

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In another study, Ali et al.[14] analyzed linear low-density polyethylene LLDPE/Surlyn® and LDPE/ Surlyn® blends during the film-blowing process using in-situ SAXS/WAXS measurements. They observed that blends exhibit the phase separation phenomenon whose resistance becomes stronger with increasing Surlyn® content.

So far, only a few studies have assessed the morphology and the thermal and mechanical behavior of LDPE/ ionomer blends and no studies have been conducted using ionomer scraps from an injection molding industry. Thus, this study aimed to evaluate the possibility of using scraps of Surlyn® ionomer, from a cosmetic packaging injection molding industry, in blends with LDPE. Blends of LDPE and ionomer scraps, with compositions ranging from 25 to 75 wt% ionomer were obtained by extrusion. The blends were characterized by mechanical and thermal properties. The morphology and melting behavior were also analyzed.

2. Experimental

2.1. Materials

The following materials were used in this study:
- Low-density polyethylene (LDPE) BC818 supplied by Braskem in the form of pellets with a density of 0.918 g/cm³ and a melt flow index of 8.3 g/10 min.
- Ethylene methacrylic acid (E/MAA) copolymer partially neutralized with sodium ions _ Surlyn® grade PC2000 (S) supplied by Silgan Dispensing Industry in the form of ground scraps from injection molding line. Surlyn® (PC2000) has a density of 0.97 g/cm³ and a melt flow index of 4.5 g/10 min.

2.2. Preparation of blends

The blends were obtained in different compositions, according to Table 1, by melt mixing using a Haake PolyLab 900/Rheomix PTW16 twin-screw extruder. The temperature ranged from 180 to 190 °C along the extruder cylinder and the screw rotation was 200rpm. Prior to extrusion, the Surlyn® was dried in a vacuum oven for 6 hours at 60 °C.

Samples for tensile, flexural, impact tests were obtained by injection molding (Battenfeld HM 60/350) using a temperature profile (180-208 °C), injection pressure 800bar and mold temperature (45 °C). Prior to injection molding, neat Surlyn® and the blends were dried in a vacuum oven for 6 hours at 60 °C.

2.3. Characterization

The effects of Surlyn® incorporation on the melt flow behavior of the LDPE were analyzed by melt flow index (MFI)

tests using a Ceast Modular Melt Index Tester 7023 (Italy), according to ASTM-D1238[24] (180 °C and 2.16 kg). The tests were carried out in duplicate. Prior to testing, the samples were dried at 60 °C for approximately 6 hours.

Blend morphologies were characterized by scanning electron microscopy (SEM) using a Quanta 650FEG_FEI microscope. Fracture surfaces from impact tests of blends were covered with gold before analysis.

Tensile and flexural tests were performed at room temperature using an Instron 5567 universal tester, according to standard conditions (ASTM D-638[25] and ASTM D-790[26], respectively). Charpy impact tests were performed at -70 °C using notched samples according to ASTM D256-D[27] (Instron Ceast 9050 Italy with pendulum weight of 5.4 J). For all mechanical tests, at least 10 specimens of each material were tested.

Differential scanning calorimetry (DSC) tests were carried out using a DSC-Q20 TA Instruments calorimeter under a nitrogen atmosphere. Samples with a mass between 10 and 12 mg were obtained from the central region of the impact samples. The samples were heated to 130 °C, with a heating rate of 10 °C/min, and then subjected to an isothermal of 3 min to remove the thermal history. Subsequently, the samples were cooled to 0 °C with a cooling rate of 10 °C/min. LDPE degree of crystallinity (Xc%) was determined from the first heating melting enthalpy according to Equation 1:

$$Xc\% = \frac{\Delta Hm}{\Delta Hmo \times \omega} \times 100\% \qquad (1)$$

where ΔHm is the measured enthalpy of melting, ω is the weight fraction of the blend components and ΔHmo is the enthalpy of melting of 100% crystalline polymer. The enthalpy of melting of 100% crystalline LDPE was considered 293 J/g[28].

Thermogravimetric (TG) analyses were performed using a SDT 600 TA Instruments under a nitrogen atmosphere. Samples taken from impact test specimens were heated at a scanning rate of 20 °C/min from 25 to 600 °C.

Prior to DSC and TG tests, neat polymers and the blends were dried in a vacuum oven for 6 hours at 60 °C. The DSC and TG tests were carried out in duplicate.

Heat Deflection Temperature (HDT) tests were performed in a CEAST HDT-VICAT equipment. Three specimens were tested according to ASTM D 648 method B[29].

3. Results and Discussion

3.1. Melt Flow Index

Figure 1 shows the melt flow index (MFI) of the neat LDPE and Surlyn® and LDPE/S blends. The dotted line represents the additivity mixing rule calculated from MFI of the neat polymers. MFI decreased with increasing Surlyn® concentration since the ionomer presents lower values of MFI. The MFI values showed a slightly negative deviation from the additive mixing rule, mainly for higher Surlyn® concentrations indicating a possible interaction between LDPE molecules and the polyethylene blocks of Surlyn®.

3.2. Thermal Properties

Figure 2 shows the heating (a) and cooling (b) thermograms of DSC of neat polymers and blends and Table 2 summarizes

Table 1. Blends composition.

Blends and neat polymers	Ionomer Surlyn® (wt%)	LDPE (wt%)
LDPE	-	100
LDPE/S 75/25	25	75
LDPE/S 50/50	50	50
LDPE/S 25/75	75	25
Ionomer Surlyn® (S)	100	-

Table 2. Thermal properties of neat polymers and blends obtained by DSC analysis.

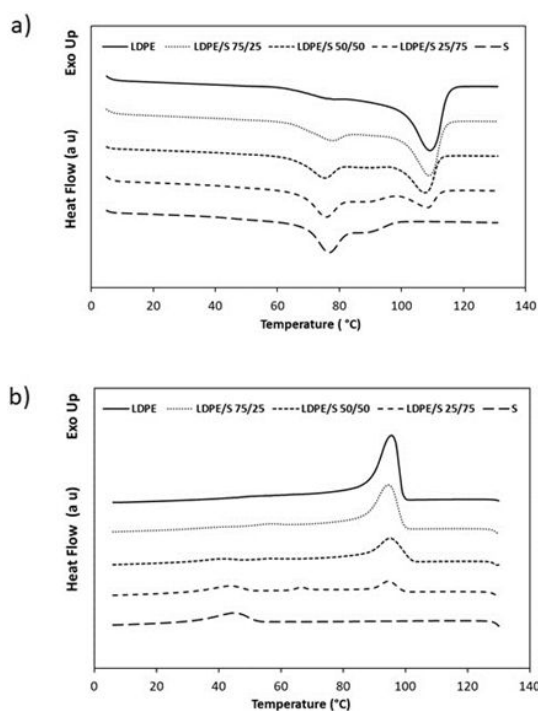|	Heating curve					Cooling curve			
	Surlyn®		LDPE			Surlyn®		LDPE	
	T_m (°C)	ΔH_m (J/g)	T_m (°C)	ΔH_m (J/g)	X_c (%)	Tc (°C)	ΔHc (J/g)	Tc (°C)	ΔHc (J/g)
LDPE	-	-	108.2	66.4	22.7	-	-	92.7	67.9
LDPE/S 75/25	75.0	7.9	107.5	45.6	20.8	-	-	92.5	48.7
LDPE/S 50/50	73.9	11.5	106.9	27.4	18.0	39.3	2.2	93.0	31.1
LDPE/S 25/75	74.0	13.7	106.7	10.8	14.8	39.4	6.9	92.6	8.6
Surlyn® (S)	74.2	16.3	-	-	-	42.5	15.7	-	
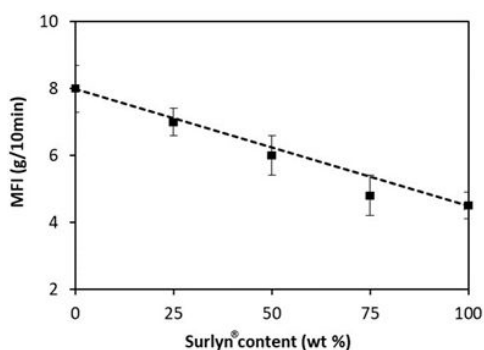
Figure 1. Melt flow index (MFI) (g/10 min) of neat polymers and blends

the DSC results. In the heating thermograms, LDPE/S blends presented two well-separated endothermic peaks, one peak at approximately 110 °C related to the melting of the LDPE crystals and another peak at approximately 90 °C related to the melting of crystals of polyethylene blocks of the Surlyn®[5]. This behavior indicated that LDPE and Surlyn® are immiscible even though the ionomer presents a similar olefin structure. The immiscibility is probably due to the difference in chain polarity of the two polymers[22]. Similar behavior was observed in many polyolefin/ionomer blends[13,21,22,30].

The endothermic peak position of the polyethylene blocks of the Surlyn® remains invariant. On the other hand, the magnitude of the melting peak related to the LDPE crystals decreased and was shifted to lower temperatures with increasing Surlyn® concentration. The degree of crystallinity of LDPE presented a decrease up to 35% with increasing Surlyn® concentration (Table 2), indicating that Surlyn® may have hampered the LDPE crystallization. Cho et al.[22] observed similar behavior in HDPE/ionomer blends.

In the cooling thermograms of the blends, two well-separated endothermic peaks are also presented. The crystallization temperature (T_c) of Surlyn® is more easily observed at concentration of 75 wt% of ionomer. The T_c of LDPE did not present significant differences in the presence of Surlyn®, as seen in Table 2. According to Horrion and Agarwal[21] this behavior may indicate that a co-crystallization phenomenon did not occur in this system.

Figure 3 shows the TG (a) and DTG (b) thermograms of neat polymers and blends. Table 3 summarizes the results obtained by TG and DTG analysis.

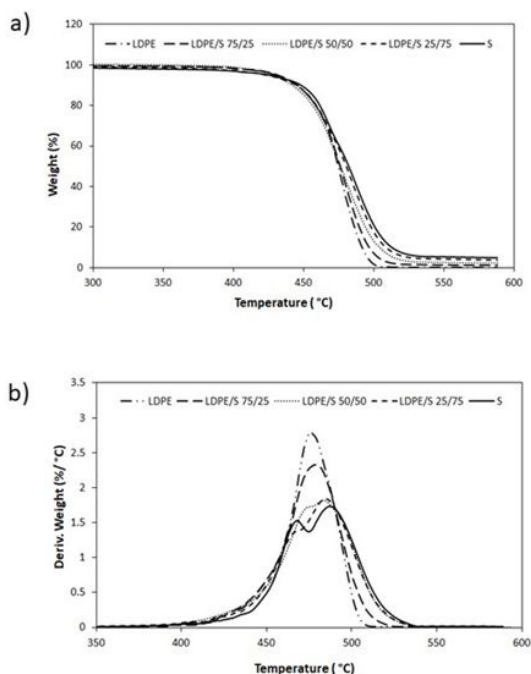**Figure 2.** DSC thermograms of neat polymers and blends. (a) heating curve; (b) cooling curve.

Surlyn® presented a shoulder in the main decomposition peak, probably related to the decomposition of the neutralized MAA comonomer fraction[31], whereas LDPE decomposition occurred in only one step. LDPE/S 75/25 blend decomposition occurred in only one step and the other blends presented an increase in the shoulder in the main decomposition with increasing Surlyn® concentration.

Surlyn® decomposition generates a residue of 5.8%, possibly due to the presence of ionic groups in its structure. Neat LDPE has practically no residue generation (0.1%). Regarding the blends, there is an increase in residue generation with increasing Surlyn® concentration (Table 3).

Compared to LDPE, the onset decomposition temperature (T_{onset}) of the blends slightly increased with increasing Surlyn® concentration as well as the decomposition rate. The decomposition rate can be quantified by the difference between the maximum decomposition temperature (T_{max}) and the onset decomposition temperature (T_{onset}), as shown

Table 3. Thermal properties of neat polymers and blends obtained by TG and DTG analysis.

	TG	DTG		
	Residue	T_{Onset}	T_{Max}	$\Delta = T_{max} - T_{onset}$
	(wt%)	(°C)	(°C)	(°C)
LDPE	0.1	448	477	29
LDPE/S 75/25	1.3	449	479	30
LDPE/S 50/50	2.4	450	473/483	33
LDPE/S 25/75	4.0	450	466/485	35
Surlyn® (S)	5.9	452	468/488	36

Figure 3. TG (a) and DTG (b) thermograms of the neat polymers and blends.

Figure 4. Heat deflection temperature (HDT) of neat polymers and blends.

in Table 3. The blends presented a higher delta value than neat LDPE, indicating that the ionomer may have delayed LDPE's decomposition rate.

The heat deflection temperature (HDT) of the neat polymers and blends are shown in Figure 4. The dotted line represents the additivity mixing rule calculated from HDT of the neat polymers. The HDT values of the blends decrease with increasing Surlyn® concentration, expected from the additivity mixing rule since the ionomer presents lower values of HDT.

3.3. Mechanical Properties

Figures 5 and 6 show the tensile and flexural properties of the neat polymers and blends as a function of ionomer content. The dotted line represents the additivity mixing rule calculated from the properties of the neat LDPE and Surlyn®.

Incorporating the ionomer promoted a significant increase in LDPE's tensile and flexural properties. Tensile strength and Young's modulus increased with increasing ionomer concentration up to 91% and 152%, respectively. Similar behavior was observed for flexural strength and flexural modulus that presented an increase up to 145% and 142%, respectively. This behavior may be related to the ionomer's higher tensile and flexural strength and higher stiffness when compared to LDPE. The properties presented similar values expected from the additivity mixing rule. It is important to note that the ionomer, even decreasing the degree of crystallinity of the LDPE as shown in the DSC results, could guarantee the increase in the stiffness of the blends.

The incorporation of the ionomer promoted a significant increase in LDPE strain at break up to 176%, being more significant for LDPE/S 50/50 and 25/75 composition, which presented a positive deviation from the additive mixing rule (Figure 4c). Nevertheless, this increase in ductility of the blends did not result in an increase in impact strength as can be seen in Figure 7. The impact strength at -70 °C decreased with increasing Surlyn® concentration, presenting values expected from the additivity mixing rule (dotted line) and can be related to the fact that the ionomer shows lower impact strength compared to LDPE.

The increase in strain at break with increasing Surlyn® concentration can be related to the ionomer's higher strain at break compared to LDPE and the blend's morphology. Figure 8 shows the micrograph of the impact fracture surface of LDPE/S blends with different Surlyn® content. Two phases are presented in all blends. LDPE/S 25/75 (Figure 8a)
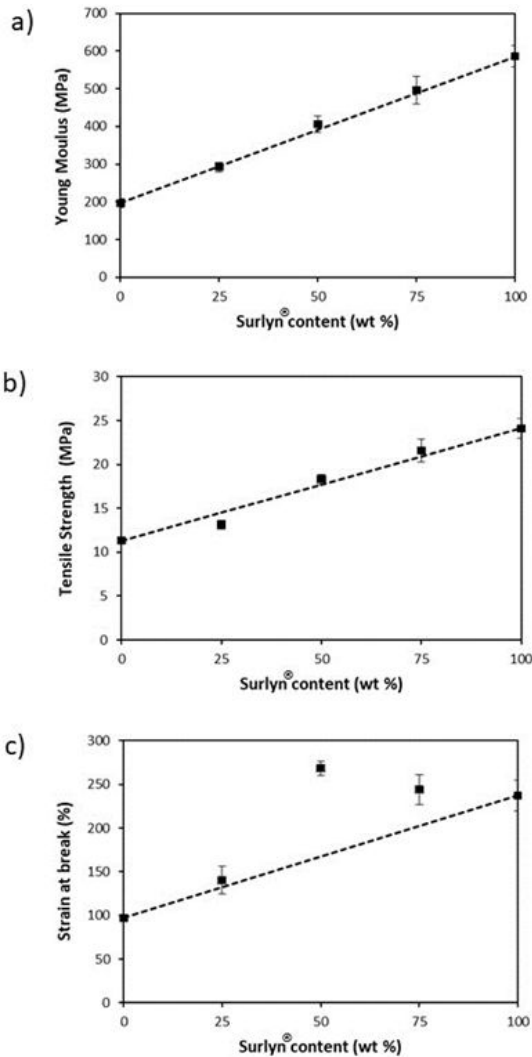
Figure 5. Tensile properties of neat polymers and blends. (a) Young modulus; (b) tensile strength; (c) strain at break.
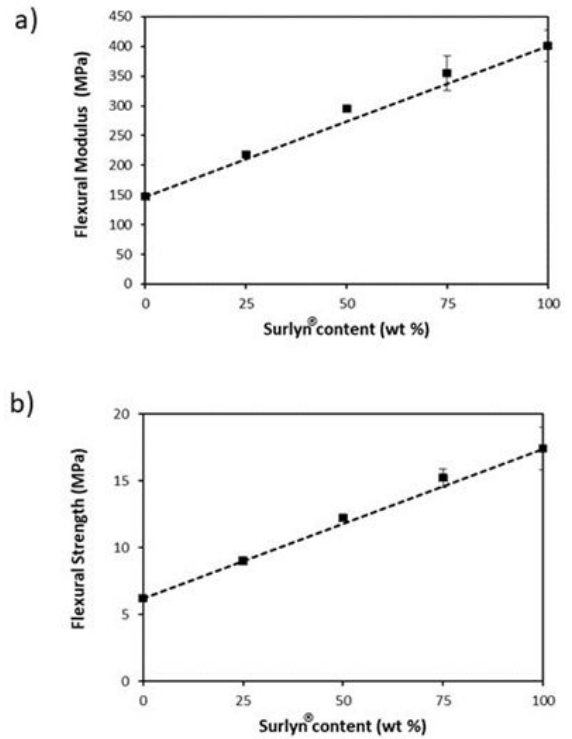

Figure 6. Flexural properties of neat polymers and blends. (a) flexural modulus; (b) flexural strength
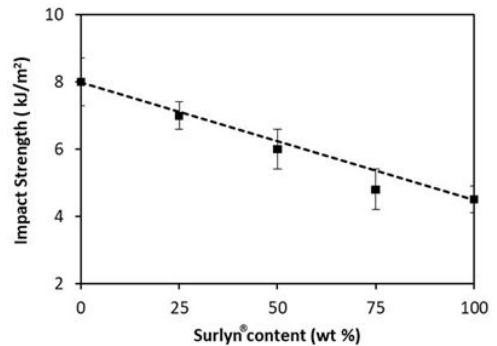
Figure 7. Impact strength of neat polymers and blends.

presented a morphology with Surlyn® domains dispersed in a LDPE continuous phase while LDPE/S 75/25 (Figure 8c) presented a morphology with LDPE domains dispersed in an ionomer continuous phase. LDPE/S 50/50 blend (Figure 8b) presented a lamellar morphology which probably facilitates the elongation of the phases as observed by Arruda et al.[32] in poly (lactic acid)/poly (butylene adipate-co-terephthalate) PLA/BAT 60/40 blend with chain extender incorporation. According to the authors, the dispersed phase changed from coarse sheets to elongated fibrils of the PBAT phase, which was probably responsible for the increase in elongation at break of 900% compared to PLA/PBAT 60/40 without chain extender. In addition, the decrease in the degree of the crystallinity of LDPE promoted by the ionomer incorporation probably resulted in a LDPE/Surlyn® interface able to deform. Boufarguine et al.[33] observed that poly(3-hydroxybutyrate-co-3-hydroxyvalerate) (PHBV) was dispersed in long thin lamellas in PLA/PHBV films produced using a multilayer

method. This morphology promoted a reduction in the glass transition temperature (Tg) of PLA mainly at the PLA-PHBV interface, resulting in a softened PLA region capable to deform.

In the case of the 25/75 LDPE/S blend in which the ionomer is the continuous phase, the elongation properties are similar to the neat Surlyn®. This behavior indicates that this ionomer property is less affected by incorporating a small amount of the LDPE dispersed phase, probably due to the network-like structure of ionomers promoted by the intermolecular ionic interactions[22,34].
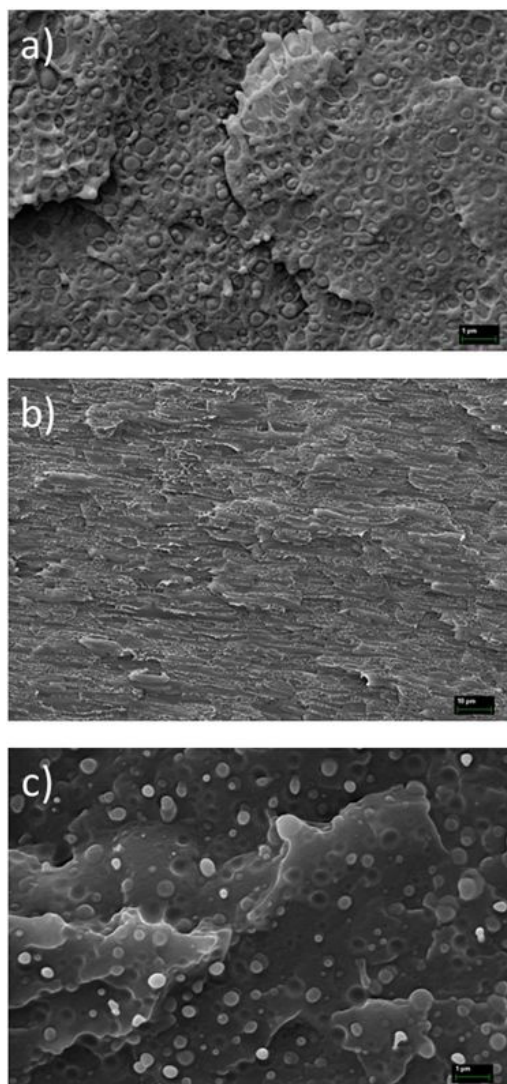
Figure 8. SEM micrographs of the fractured surfaces of the specimens of the blends: (a) LDPE/S 75/25; (b) LDPE/S 50/50; (c) LDPE/S 25/75.

4. Conclusions

In the present study, blends of LDPE and scraps of Surlyn® ionomer, with compositions ranging from 25 to 75 wt% ionomer were obtained by extrusion. The effects of the ionomer incorporation on the melt flow, on the morphology, and the mechanical and thermal properties of the blends were assessed.

MFI decreased with increasing Surlyn® concentration since the ionomer presents lower values of MFI. A slightly negative deviation from the additive mixing rule, may indicate a possible interaction between LDPE and Surlyn® molecules.

All blends' morphology showed two phases, indicating immiscibility of phases. DSC thermograms corroborate this behavior presenting two melting peaks in the heating curves. LDPE/S 25/75 and 75/25 presented a morphology with domains dispersed in a continuous phase while LDPE/S 50/50 blend presented a lamellar morphology.

Surlyn® incorporation promoted a slight decrease melting temperature and a significant decrease in the degree of crystallization of LDPE, indicating that the ionomer may have hampered the LDPE crystallization. In addition, Surlyn® incorporation resulted in a slight increase in the thermal decomposition temperature and delayed the decomposition rate of LDPE.

Tensile and flexural strength, stiffness and strain at break of the blends increased with increasing Surlyn® concentration. Impact strength at -70 °C decreased with increasing ionomer concentration. The mechanical properties values obtained were similar to the expected values from the additivity mixing rule since the ionomer presents higher tensile and flexural properties and lower impact strength compared to LDPE. The blends' thermal deflection temperature (HDT) values decrease with increasing Surlyn® concentration, expected from the additivity mixing rule.

In conclusion, Surlyn® ionomer scraps from cosmetic packaging production can be used in LDPE blends, mainly resulting in mechanical properties improvements, except impact strength. It may be an alternative for the destination of ionomer scraps if the final product obtained does not suffer impact stresses during its use.

6. References

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