Toughening of polystyrene using styrene-butadiene rubber (SBRr) waste from the shoe industry

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Abstract

The vulcanized rubber waste from the shoe industry causes environmental damage when it is incinerated or inappropriately discarded, turning this into a problem of major concern. Therefore, this study had as the main objective the Polystyrene (PS) toughening using different contents of white vulcanized styrene-butadiene rubber (SBRr) waste produced in the shoe industry. The mixtures were initially prepared in a co-rotational double screw extruder and, thereafter, the extruded granules were injection molded. Analyzed were the rheological, mechanical, thermomechanical properties and morphology of the produced blends. The rheological results showed a viscosity increase as the SBRr concentration was augmented, leading to a higher stability when compared to pure Polystyrene. Verified was an increase of impact resistance of 189% to the blend that contained 50% of SBR rather than pure Polystyrene. On the other hand, the traction properties, hardness Shore D, thermal deflection temperature (HDT) and Vicat softening temperature of the blends tended to decrease when compared to pure Polystyrene results. However, as this SBR waste is made up of a complex mixture of SBR, filler, processing additions, curing agents and stabilizers, it probably acted in the sense of not causing such a drastic reduction of the properties, even using a high concentration of SBRr waste. The morphologies obtained with the SEM method (Scanning Electronic Microscope) were quite different and typical of immiscible blends. The results show that it is possible to obtain a new material with good properties, valuing a discarded industrial waste and avoiding environment aggression.

Keywords: reuse, rubber waste, toughening, polystyrene.

1. Introduction

Despite the great benefits provided by the technological development in the last decades, an expressive increase of the industries' solid waste volume has generated a negative contribution to the environment, since most of the times, the product's final destiny is inappropriate or unacceptable from the environmental point of view. In this sense, the pollution problem is increasingly worrysome, specially related to the rubber waste produced by the tire and shoe industry (Ramarad *et al*., 2015; Luna *et al*., 2015a).

According to the statistics from the International Rubber Study Group, the world consumption of natural and synthetic rubber in 2016 was 27.2 million tons, generating a large amount of waste during the development of materials for everyday use, as well as post-consume rejects.

The vulcanized elastomer SBR is the world's most consumed synthetic rubber, as it is used in the manufacturing of most of the tires, in addition to belts, hoses, shoe soles, sealing profiles, cylinder coatings and cable isolation

(Weber *et al.*, 2011). SBR is a non-polar material that shows good resistance to attrition; good properties under low temperatures; satisfactory mechanical performance and high resistance to natural aging, as well as compatibility with most of the elastomers (Grison *et al.,* 2010). Thus, this material generates two counterpoints: on one hand the rubber waste from the shoe and tire industry are a source of aggression when discarded in the environment, and on the other hand, a loss of feedstock with great properties that could be reused in other applications.

One of the greatest difficulties to reuse vulcanized elastomers lies in the fact that these materials have in their structures the presence of crosslinks that make them infusible and insoluble in organic solvents, blocking their reprocessing as thermoplastics (Garcia et al., 2015; Luna *et al.,* 2015b). Nowadays, the search for technologies that make possible the reuse of this waste is one of the main concerns of the scientific community and industry as well as environmentalists.

One of the technologies used for rubber waste reuse is through the development of mixtures of these materials with thermoplastics, in other words, the development of polymeric blends. In this case, the ground wastes of rubber in micrometric form are blended with a polymeric matrix during the processing, acting as flexible fillers (Costa *et al.,* 2012). In general, rubber is added to thermoplastic polymers with fragile mechanical behavior with the objective of toughening them, e.g. the Polystyrene (Coutinho *et al.,* 2008).

Polystyrene (PS) is a thermoplastic polymer, easily synthetized, processed and recycled. It is relatively resistant to degradation, but has low impact strength and consequently, needs to be toughened for some applications (Calleja *et al.*, 2004). The addition of vulcanized rubber waste from tires and shoes in a black powder form is presented in literature as a way of toughening the polystyrene, being profitable from both economic and environmental points of view, turning it possible to have a new material with great properties (Macsiniuc *et al.,* 2014; Veilleux e Rodrigues, 2016;).

Studies of the use of vulcanized SBR rubber materials with white coloration without development of projects such as polymer matrix or polystyrene (PS) are still very scarce or almost nonexistent. Besides that, currently, the reuse of this waste is even harder, due to the addition of new components to the mixtures and additives to increase their performance and lifespan, where the real formulation is unknown due to their secret nature (Sousa *et al.*, 2015). Therefore, from the scientific and technological point of view, the production and development of PS/White rubber waste blends can boost new advances in the production of materials with better properties that contribute to a sustainable environmental cycle.

Therefore, this research had the goal of toughening the polystyrene (PS) with different contents of white vulcanized SBR waste from the shoe industry.

2. Materials and methods

Materials

The crystal polystyrene (PS), commercialized under the U288 code, density of 1.04 g/cm³ and melt flow rate (MFI) of 3 g/10 min (tested under the condition: 200°C/5 kg), provided in granule form and manufactured by Unigel S.A. (Brazil) was used as the polymer matrix. The filler used, or dispersed phase, was vulcanized SBRr waste in a white powder form, coming from São Paulo Alpargatas S.A., shoe industry, based in Campina Grande, Paraíba - Brazil.

Blends Preparation

Before preparing the blends, the powdered SBRr waste from the industry was sifted in number 18 mesh (1mm), being used just the powder that passed through the mesh. The pure polystyrene and the prepared blends PS/SBRr (80/20, 70/30, 60/40 and 50/50%) in mass were processed in a modular double screw co-rotational extruder, ZSK model (D = 18 mm e L/D = 40), made by Coperion ZSK, under 190°C temperature in 1 and 2 zones and 200°C in the other zones. Screw's rotating speed was 250 rpm with controlled feed rate of 4 Kg/h, with the screw profile configured for distributive and dispersive element mixtures. After the extrusion blend processing, the material was granulated and dried in a vacuumless oven for 24h under a 60°C temperature, and subsequently, in a vacuum oven for 24h under the same temperature. The polystyrene and the extrusion obtained blends were injection molded in an Arburg injector, Allrounder 207C Golden Edition model, to obtain impact, traction and HDT proof bodies. The molding temperature was 200°C, and the mold temperature was 20°C.

Characterization Techniques

Torque Rheometry

The rheological curves were obtained in a Haake PolyLab QC mixer from Thermo Scientific, with roller-kind rotors, under 200°C and 50 rpm rotation speed, under air atmosphere.

Mechanical Properties

The resistance test to Izod impact was performed on test bodies notched accordingly to D256 ASTM rule, in a Ceast device, Resil 5.5 J model, operating with a 2.75 J hammer in ambient temperature. The results reported are the average of eight samples.

The traction tests were realized on test bodies injected according to the D638 ASTM standard, using an EMIC DL 2000 universal test machine with a charging speed of 5 mm/min and a 2 kN load cell. The tests were run at ambient temperature and the results reported are the average of eight samples

The resistance to penetration test was realized accordingly to the D2240 ASTM standard, in a Shore Durometer Hardness Type "D" device, with 1 kN load controlled by springs calibrated by standardized dentiometers for the durometer. The indenter was pressed over the sample during 15 seconds at five aleatory points of the sample.

Thermomechanical Properties

The heat deflection temperature (HDT) was obtained according to the D648 ASTM standard, in a Ceast HDT 6 Vicat device with 455 kPa of tension and a 120°C/h heating rate. The temperature was determined after the sample had suffered a deflection of 0.25 mm. The results reported are the average of three samples.

The Vicat softening test temperature was performed according to the D1525 ASTM standard, applying a 120°C/h heating rate. The temperature was determined after the needle had penetrated 1mm inside the proof bodies. The results reported are the average of three samples.

Morphological Analysis

The SEM (Scanning Electronic Microscope) analysis was obtained with a VEGAN 3 TESCAN device with a 30 kV voltage and under high vacuum conditions. The fracture surfaces of the impact samples were analyzed. The surfaces were coated with a gold layer (sputtering – Shimadzu Metallizer–IC-50, using a 4mA current).

3. Results and discussion

Rheological Properties

Figures 1 (a, b, c e d) illustrate the graphics of the rheological behavior of pure polystyrene and of the binary PS/SBRr blend with 20, 30, 40 and 50% of SBRr mass, respectively.

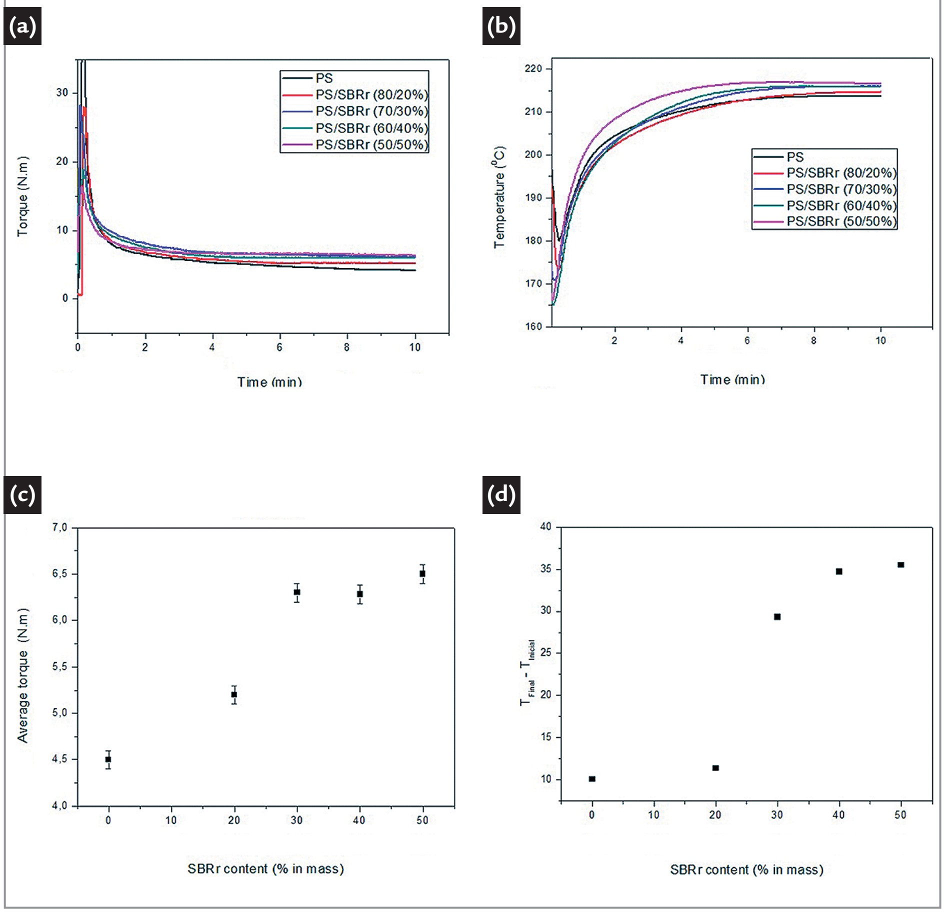

Figure 1
Rheological behavior of pure PS and binary blends (PS/SBRr): (a) torque versus time; (b) temperature versus time; (c) average stabilized torque; and (d) mechanical work.

As seen in Figure 1a, after 2 minutes of processing, the pure PS and binary blends torque tends to become almost constant, with little oscillations around an average value. This behavior indicates a tendency to torque stabilization, and consequently, viscosity stabilization for the process conditions used, that is, 50 rpm speed and 200°C temperature. It is observed that pure PS showed lower torque; that is, a lower viscosity when compared to PS/SBRr blends, which indicates lower energy consumption for its processing, even being a viscous polymer, with 3 g/10 min low melt flow rate. It is also possible to visualize that the torque value of the PS/SBRr blends tended to augment with the increase of SBRr rate in the mixture, leading to a subtle viscosity elevation, which can be assigned to the high concentration of vulcanized rubber powder, acting as a filler. For the 50/50% SBRr composed blend, the viscosity increase was more pronounced, and for the 30 and 40% SBRr, compound blend, torques were similar between them. In other words, even with a 10% increase of SBR, it had little influence on torque between these two blends. In general, it is probably possible to infer that the occurrence of a synergic effect is related to the increase of viscosity of the blends, which is the action of a vulcanized filler over pure PS and possible interactions between similar chemical groups, such as the SBRr blocks and PS molecules (Yousefi, 2012).

Figure 1b shows the temperature curves versus time, obtained during the mixtures of pure PS torque and blend torque in the rheometer. A temperature decrease inside the mixture chamber in the beginning of the process has been verified; it relates to the thermal energy absorption by the materials during the solid state feeding. As the mixture goes on, it is notable that the temperature tends to get higher, due to the mechanical energy dissipation and to the chamber's own heating. The blend that contained 50% of SBRr shows a higher final processing temperature, whereas the pure OS shows the lower temperature. Thus, PS requires a lower energy consumption during the processing time, and the 50% SBRr blend, a higher one. Meanwhile, the mixtures with 20, 30 and 40% of SBRr composition presented intermediate temperatures for those respective materials, as expected, due to the influence of the content on SBRr additivity.

Figure 1c illustrates the average stabilized torque values in a range of 5 to 10 minutes for the pure PS and the blends depending on the SBRr content. It is noticeable that an increase in SBRr concentration elevates the average torque and, consequently, the blend's viscosity, when compared to PS. The 30% and 40% SBRr blends have nearly the same average torque. Thus, at first sight, it is possible to say that, even having different SBRr levels, the viscosity of the blends are similar. The temperature curve based on time, Figure 1b, shows that the numbers of the mixture temperatures are very close. This confirmation emphasizes the hypothesis that the 30% and 40% SBRr blends have close viscosities at the end of processing's temperature range, around 216°C.

Figure 1d represents the difference between the final and initial temperatures of PS and the blends inside the chamber during the whole process, reflecting the entire mechanical work done. One can observe that PS showed the lowest temperature variation during the processing, reflecting in smaller mechanical work between all the materials. For the blend that contained 20% of SBRr, the temperature variation during the processing was about 11.4°C, suggesting that the contribution for the temperature increase from the mechanical energy conversion is similar to PS. The blends with 30 and 40% SBRr showed variations of 29.5 and 33.5°C, respectively, demonstrating that throughout the process, the blend with 40% SBRr imposes higher mechanical work during the processing. The blend containing 50% of SBRr presents higher temperature variation, around 35°C and, consequently, higher viscous heating when compared to the other blends.

In general, the change in the viscosity form of the TP with the increase of the percentage of SBRr content, observed in the torque rheometry is in agreement with the formulation of these rubbers, since they impose greater difficulty during the processing. Generally, these residues (SBRr) are composed of a complex mixture having inorganic fillers and other additives. In addition, it has a cross-linked structure, which prevents melting and reprocessing. Similar findings were also verified by Ciro *et al.* (2015).

Mechanical Properties

The Table 1 illustrates the results of pure PS and binary PS/SBRr mechanical properties in relation to SBRr level increase.

Materiais	Impact Strength (J/m)	Tensile Stress (MPa)	Elastic Modulus (MPa)	Hardness Shore D
PS	23.0 ± 0.9	42.7 ± 1.4	1216.9 ± 55.0	82.0 ± 1.4
PS/SBRr (80/20)%	37.5 ± 1.9	31.3 ± 0.5	939.6 ± 20.0	77.5 ± 0.8
PS/SBRr (70/30)%	56.1 ± 1.9	25.1 ± 1.0	913.3 ± 13.7	75.4 ± 0.9
PS/SBRr (60/40)%	60.9 ± 1.2	19.8 ± 0.8	742.5 ± 7.8	73.8 ± 0.8
PS/SBRr (50/50)%	66.5 ± 0.9	17.2 ± 0.8	686.5 ± 17.9	71.5 ± 0.9

Table 1
Mechanical properties
of pure PS and binary blends.

According to Table 1, it is observed that pure PS shows low resistance to impact, due to its fragile behavior, given mainly to the high glassy transition temperature (~100°C) that this polymer presents. It is verified that the addition and increase of quantity of a flexible material, such as SBRr waste, to a rigid matrix (PS) causes an augment of impact resistance. From Table 1, it is seen that the blends impact resistance increased substantially with the quantity increase of SBRr waste, with a gain of 189% of this property for the PS/SBRr blend (50/50%) compared to pure PS. The impact resistance elevation with the increasing of rubber content can be attributed to the rubber particle capacity to trigger toughening mechanisms, allowing a better distribution of tensions and, consequently, retarding material fracture. The results of impact resistance, in Table 1, corroborates with the results of torque rheometry, that is, when raising the viscosity of the blends, better results of impact resistance were observed.

Table 1 demonstrates that for pure polystyrene, there are high resistance values and an elasticity module under traction, which are typical of fragile polymers. It also can be seen that there is a progressive fall in the resistance under traction of the PS/SBRr blends with the increase of the SBRr concentration. This behavior is attributed to the chemical structure of the SBRr elastomeric waste that has butadiene in its chain, providing flexibility to the mixtures. The PS, when hardened with rubber and under tension, suffers a change in its matrix phase tension state, leading to a concentration or dissipation of this energy by the dispersed elastomeric particles. The smaller the value of the blend traction resistance, the more energy dissipated by the material (Tang *et al.*, 2001), corroborating with the blends impact resistance results, where it is observed that the more the SBRr quantity increase, the higher the mixture impact resistance is. This means that it has a higher energy dissipation level and, consequently, smaller resistance under traction.

The introduction of SBRr into the PS glassy matrix promoted a reduction of the elastic module of all blends, becoming evident a more pronounced decrease at each 10% increase of SBRr waste. The 20% SBRr use has given a module reduction of 22.8% to the blend when compared to pure PS, whereas 50% of SBRr decreased the module by 43.6%. However, when it is considered that is a vulcanized rubber waste, composed of a complex SBR mixture (styrene-butadiene co-polymer), together with processing additions and fillers, using 50% of it's weight of SBRr did not provoke a very drastic reduction on the elasticity module, as, for instance, in Ibrahim and Kadum (2012), where there was observed a higher than 50% reduction on polystyrene elastic module when toughening it with 50% of styrene-butadiene-styrene thermoplastic rubber (SBS).

Related to the hardness of the systems, it can be seen in Table 1 that PS displayed the highest hardness, that is, the highest resistance to penetration. In general, the Shore hardness values obtained for all blends tended to reduce with the increase of SBRr quantity. This behavior is due, possibly, to rubber incorporation as a dispersed phase in a polystyrene matrix, and thus leading to a lower stiffness and softer material. For 20% SBRr in PS, there was observed a hardness decrease of 5.5% versus pure PS, whereas for 50% of SBRr, this property reduced around 12.8%. Therefore, it is possible to observe that even using high SBRr concentrations in PS, the Shore hardness decrease is not so relevant as in some studies considering PS blends with 20% of low-cis polybutadiene (PB_b) (Coutinho *et al.*, 2008), in which was found approximately 22.2% reduction of this property. Probably, this is due to the use of a reticulate SBRr waste that has mineral fillers in its composition, which favoured a better penetration resistance and helped to avoid a drastic fall in the PS/SBRr blend hardness. The behavior of PS/SBRr blends in Shore D hardness are consistent with the tensile modulus of elasticity, where a decrease in stiffness was accompanied by a decrease in Shore D hardness.

Thermomechanical Properties

The polymer properties, as all materials, are dependant on temperature. This assumes a special importance to pieces that suffer static mechanical efforts as well as high temperatures simultaneously. As a result, the heat deflection temperature (HDT) and VICAT temperature of a polymer are of great importance, allowing the conception of products for the automobile industry, for example. The Figure 2 (a, b) shows the HDT and Vicat temperature of pure PS and of the PS blends containing 20, 30, 40 and 50% by weight of SBRr, respectively.

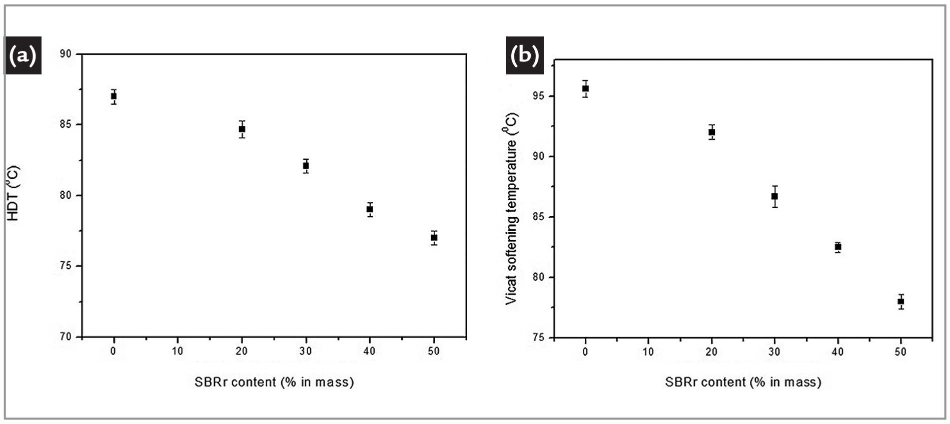

Figure 2
Thermomechanical behavior of pure PS and binary blends (PS/SBRr): (a) heat deflection temperature (°C); (b) Vicat softening temperature (°C).

The heat deflection temperature doesn't show any variation with most of the variables (particle size, molar mass, graphitization, etc), but only with the rubber quantity added (Grassi e Forte, 2001). It can be seen in Figure 2a that the maximum value reached for the HDT was 87°C for pure PS, probably because it is a stiff material and, consequently, needs a higher temperature for proof body deflection. It is observable as well that there is a decrease in this property for all blends as the SBRr waste quantity is increased, being that the decline is more evidenced for the 50/50% SBRr composition. This fact is assigned to the presence of a high elastomeric component content in the mixture, which gives it high mobility, due to the presence of butadiene groups, acting to make the systems more flexible, generating an HDT behavior loss. However, it is interesting to note that there was no drastic reduction of the HDT of the blends, even using 50% by weight of a rubber residue (SBRr), the decrease was around 13% compared to the PS, surpassing even polymers of engineering as polyamide (~55°C). Therefore, it is suggestible that the addition of a rubber waste that has crosslinks and reinforced mineral fillers, does not provoke a drastic loss of this property. The results of the heat distortion temperature (HDT) are in agreement with the elastic modulus (Table 1).

The rubber incorporation into polymers tends to reduce hardness, contributing to decreasing the Vicat softening temperature (Tasdemir e Karatop, 2006). In Figure 2b, it is observable that pure PS showed the highest Vicat softening temperature, which is due to its high stiffness, requiring a higher temperature for needle penetration. On the other hand, a decrease in the blend's Vicat temperatures can be seen when compared to pure PS, being the difference more noticeable at each SBR increase. It is presumed that when adding to PS a flexible filler, it favours a hardness reduction, causing a lower resistance to penetration as the temperature gets higher. It is noted that the Vicat softening temperature results are consistent with Shore D hardness results (Table 1).

It is observable that HDT and Vicat results are striking and representative, since all the standard deviations are very low. The high precision of all blends is due to all the samples having been submitted to silicone oil that promotes a great heating transference homogeneity in the samples.

Morphological Analysis

The Figure 3 shows the photomicrographs of the pure PS and blends fracture surfaces obtained by SEM.

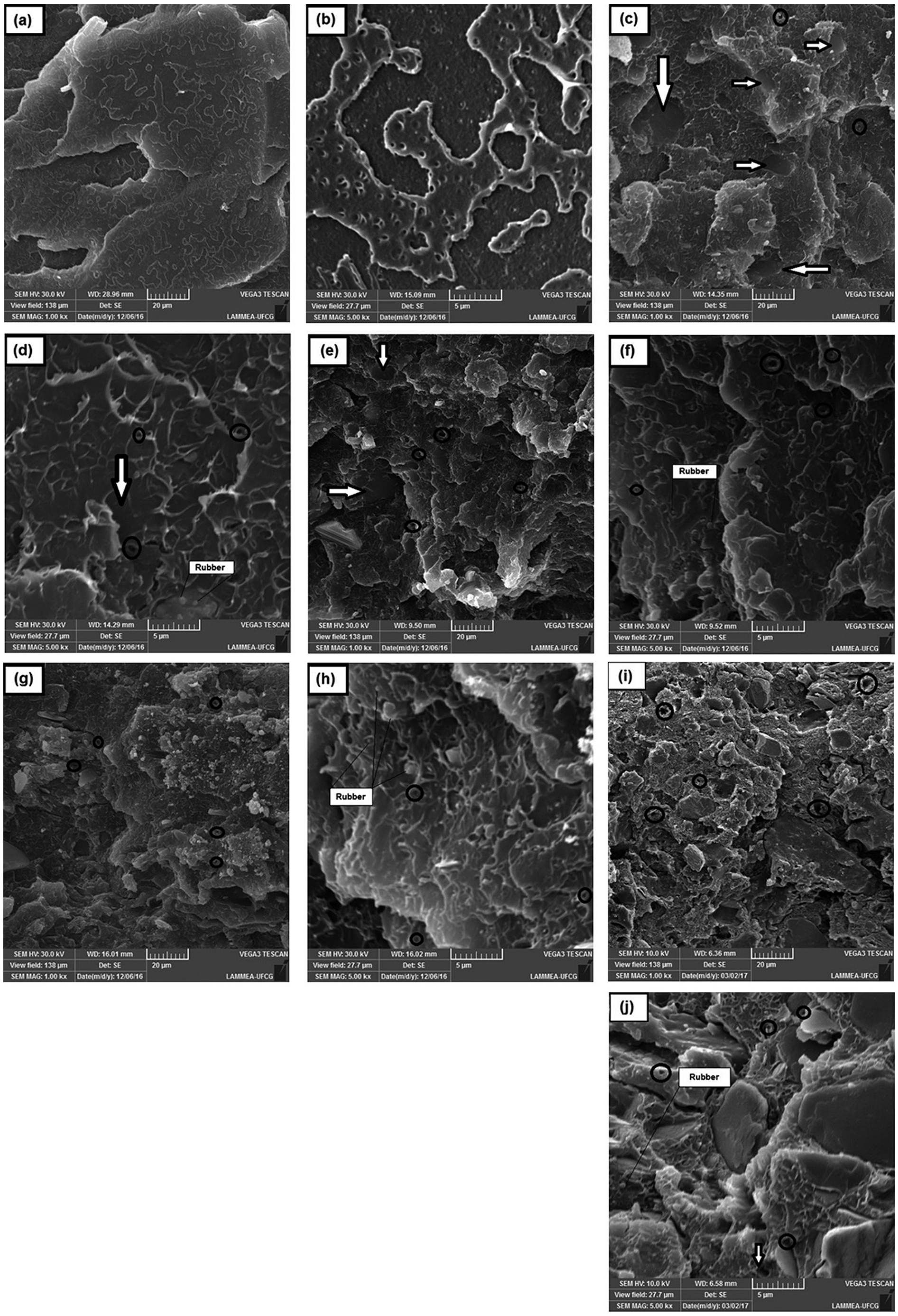

Figure 3
SEM micrographs of the samples fracture surfaces with 1000x and 5000x zoom, being: (a, b) Pure PS; (c, d) PS/SBRr (80/20%); (e, f) PS/SBRr (70/30%); (g, h) PS/SBRr (60/40%); (i, j) PS/SBRr (50/50%).

Figures 3 (a, b) presents the pure PS photomicrographs, which is smooth and homogeneous, associated to the fragile nature of this low impact resistance polymer. Besides that, it is possible to verify stretch marks correspondening to the marks left by the cracks that caused rupture of the proof body, and that was nucleated from the notch.

All micrographs, Figure 3 (c, d, e, f, g, h, i, j), for the blends reveal a morphologically typical surface of immiscible blends, showing PS and SBRr phase segregation. Besides this, all blends showed voids caused by the SBRr particle displacement over the PS matrix (marked by dark circles in the micrographs), probably as a consequence of low adhesion between the phases. Nevertheless, the SBR concentration effect produces different surface characteristics. In the 20% SBRr blend, Figures 3 (c, d), it is possible to verify a more homogeneous and smooth aspect in the regions (white arrows), characterizing a low tenacity, fragile blend, corroborating with the impact results (Table 1). With 30% SBR, according to the morphologies (e, f), it is still possible to observe fragile aspect regions (white arrows), yet with a noticeable reduction of these regions, which permits greater plastic deformation. Also, it is possible to see the presence of geometrically spherical rubber particles, with smooth surfaces, indicating weak interfacial adhesion (as indicated in the white chart). On the other hand, the 40% SBRr blend almost does not present fragile regions, making more visible the rough surface rubber particles that are irregular and heterogeneously sized. It is noticeable too that the material shows a highly rugged surface, having irregular voids (dark circles in the micrographs), probably associated with SBRr segment diffusion in contact with PS, causing, even limitedly, an interlacing between them. The 3h morphology of this blend containing 40% of SBRr shows some particles barely adhered to the PS matrix.

The 50% SBRr blend shows a morphology with a high level of plastic deformation, Figures 3 (i, j), show that the dispersed phase is not easily verified. There are clear evidences of a more ductile rupture mechanism, that is, with a more visible plastic deformation. It is observed in the 3j morphology that there is an increase in phase separation, start of delamination and very heterogeneous voids. Also, seen is a lack of interface adhesion, increase of heterogeneity of particle sizes and increase of drawn particles during the impact test. If we compare the morphology with the impact results, we shall see that blend impact resistance increases due to an augment only of the SBRr elastomeric phase, probably because the SBRr is anchored to the PS matrix exactly for the miscibility between styrene groups; the reason that promoted these elevations in Table 1.

4. Conclusions

The influence of white vulcanized rubber waste concentration in polystyrene toughening was investigated. It was verified that a kind of reuse of these elastomeric wastes, after turning them into powder, is as a filler to harden the polystyrene. In general, it is observable that the use of white rubber waste powder in polystyrene mixtures becomes convenient from both scientific and technological points of view, since it makes possible the production of materials with good elastic properties. Besides that, it also allows the development of a lower price material, as this waste is normally incinerated or discarded. Due to the inherent difficulties to reuse the rubber waste, the results confirm that it is possible to reduce environmental responsibility generated by this industrial sector, producing materials with desired properties.

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