RSM applied to PS/SBRr/SEBS Blends. Proper tool for maximized properties

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Abstract

Reusing rubber waste from footwear industry emerges as great practise in order to reduce environmental damage on natural ecosystems. Based on this target, the present work aimed to apply the response surface methodology (RSM) to polystyrene/styrene-butadiene PS/SBRr blends compatibilized with styrene-ethylene-butylene-styrene (SEBS). A mixture planning was applied to PS/SBRr/SEBS blends in order to investigate mechanical and thermomechanical behaviors as response variables. Scanning electron microscopy (SEM) was used to evaluate the morphology of investigated blends, which were melt extruded in a corrotational twin screw extruder and specimens were injection moulded. High impact strength, toughness, elongation at break and thermal deflection temperature (HDT) were achieved at SEBS rich region (∼10%). At PS rich region, tensile strength, Shore D hardness and Vicat softening temperature (VST) were more expressive. Related to morphological character, blends with 10% SEBS presented the highest interfacial adhesion, with high level of plastic deformation. Gathered results show that depending on the composition, properties significantly change, being RSM a proper tool to preview trends on mechanical and thermomechanical properties. In general, PS/SBRr/SEBS blends may be properly used as packaging and building applications.

Introduction

Post-consumer polymeric waste is a pollution source which seriously damages the environment, generally trending to be aggravated if efficient management and recycling policies are not adopted [1–3]. In this context, waste from elastomeric products used in the automobile and footwear industries has been drawn attention, since the vast majority are vulcanized, therefore lasting decades to be decomposed [4, 5].

The reuse of vulcanized rubbers is currently one of the main drawbacks in the recycling area, as consequence of chemical crosslinking between macromolecular chains, which hinder melting and reprocessing [6, 7]. Therefore, the final destination of vulcanized waste has been one of the main problems facing humanity, as its degradation occurs slowly, not only due to crosslinkings, but also due to stabilizers and other additives, making it difficult to be reused and heading to environmental damage when discarded [8, 9]. Currently, one way to reuse them is to be added as filler in thermoplastic or thermoset polymers, aiming at the production of new materials [10, 11].

Rubber waste from footwear and tire industries is a kind of raw material waste with good properties such as elasticity, flexibility, impermeability and abrasion strength [12]. Therefore, investigations aiming feasibilities of these vulcanized rubbers are primordial, as it provides material development with good technological performance, at the same time, enabling cost reduction in the final product. Thus, researches have been reported [13, 14] related to the development of polymer blends based on rubber and polystyrene (PS) residues, aiming to
Investigated system

Experimental planning aims to improve the performance of a system or process, seeking to maximize benefits of investigated system [15–17]. Macsiniuc et al [18] investigated mechanical properties and morphology of PS/SBRr blends compatibilized with styrene (ethylene butylene) styrene (SEBS). PS/SBRr blends presented rubber particles distribution relatively well dispersed in PS matrix, heading to impact strength improvement. Nevertheless, some particles were pulled out from PS matrix due to weak interfacial adhesion, most due to SBRr crosslinked character. Addition of SEBS compatibilizer proved to be effective, improving mechanical properties related to PS/SBRr. Morphological analysis indicated higher interfacial adhesion in PS/SBRr/SEBS system, suggesting strengthened phase interactions.

Mechanical, thermal, rheological and morphological properties of PS/SBRr blends were widely discussed [19–21]. Nevertheless, no studies have been found related to the use of response surface methodology (RSM) applied to PS/SBRr/SEBS. Thus, it becomes a relevant point to be explored, since the mechanical behavior trend of PS/SBRr/SEBS can be analyzed on a three-dimensional surface, granting the feasibility to verify synergist and antagonist effects within the blends. Generally, rubber addition to a rigid matrix reduces the elastic modulus. However, an antagonistic effect was observed by Rosas et al (2013) [22] where increasing the vulcanized rubber tire (VRT) content up to 10% to PS matrix the Young’s modulus increased compared to neat PS; nevertheless at contents (> 10% VTR), Young’s modulus gradually decreased; it is supposed to exist a critical concentration at which the stiffness increases and exceeding this the saturates translating in stiffness reduction. In principle, this antagonistic behavior in increasing stiffness with vulcanized rubber addition was only found at low concentrations.

Therefore, the present work aimed to develop PS/SBRr/SEBS blends using mixture planning; applying the mechanical and thermomechanical properties as variables, a response surface mapping within the interest region was made, which allowed to predict properties changes for sundry compositions.

Materials and methods

Materials

Crystal polystyrene (PS) commercially coded as U288, density 1.04 g cm$^{-3}$ and melt index 3 g/10 min (200 °C/5 kg), as pellets was supplied by Unigel SA (Brazil). Residue of styrene-butadiene vulcanized rubber (SBRr), from Alpargatas S.A. (São Paulo). Styrene-(ethylene-butylene)-styrene (SEBS) copolymer used as compatibilizing agent coded G1652 with 30% of styrene and melt flow rate (MFR) of 5 g/10 min (230 °C/5 kg) was supplied by Kraton. The chemical structures of these materials are presented in table 1.

Methods

Mixture planning

Experimental planning aims to improve the performance of a system or process, seeking to maximize benefits of investigated system [23]. When applying an experimental planning procedure, two types of variables should be recognized: independent variables (input data) and dependent variables (responses) [24]. In cases involving experimental mixture design, final properties are determined by their components’ proportion [25]. Experimental variables must be kept constant, such as: processing and injection conditions [26]. Additionally, when filling the program with independent variables, maximum and minimum component contents are informed; hence the system assembles a planning matrix with all compositions to be processed.

In this work, experimental planning will be used to PS/SBRr/SEBS blends, aiming at understanding how to vary the properties when changing proportions between phases. In this case, the program will not be fed with neat PS (100%) data.

Table 2 presents suggested planning matrix, containing 5 distinct compositions. Statistica 8.0$^*$ software was used for the mixture planning with three variables, being PS (maximum of 50% and minimum of 45%), SBRr (maximum of 50% and minimum of 45%) and SEBS (maximum of 10% and minimum of 0%), the input variables; as response variables: impact strength, tensile toughness, thermal deflection temperature (HDT), Vicat softening temperature (VST), elastic modulus, tensile strength, elongation at break and shore D hardness.

Blend processing

The blends’ components were dried and subsequently processed into a Coperion Werner-Pfleiderer model ZSK (D = 18 mm and L/D = 40) modular corrotational twin screw extruder with temperature profile in the zones; 190; 200; 200; 200; 200; 200 °C, 250 rpm screw rate and 4 kg h$^{-1}$ controlled feed rate, with screw profile configured with distributive and dispersive mixing elements. Subsequently, the materials were granulated and dried in a vacuum oven for 24 h at 60 °C.
The blends were injection molded on an Arburg Model Allrounder 207C Golden Edition injector to obtain impact, tensile and thermal deflection temperature (HDT) specimens according to ASTM D256, D638 and D648, respectively. The molding specifications were: temperature profile of 200 °C in all zones; mold temperature of 20 °C; injection and repression pressure, 1000 and 500 bar, respectively. After injection molding, the specimens were stored in a desiccator until the time for characterization. Figure 1 shows the tensile test specimens of the blends.

### Characterization

Izod impact strength test was performed on notched specimens, according to ASTM D256, in a Resil 5.5 J from Ceast, operating with hammer of 2.75 J, at room temperature (≈23 °C). Presented results are average of six specimens.
Tensile test carried out with injected specimens according to ASTM D638 using an EMIC DL 2000 universal test machine with elongation rate of 50 mm min$^{-1}$ and load cell of 20 kN at room temperature ($\sim 23 ^\circ$C). Presented results are average of six specimens.

Thermal deflection temperature (HDT) was performed according to ASTM D648 in a Ceast HDT 6 VICAT model with voltage of 1.82 MPa and heating rate of 120 $^\circ$C h$^{-1}$. The temperature was determined after the sample deflecting 0.25 mm. Presented results are average of three specimens.

Vicat softening temperature (VST) test was performed according to ASTM D1525 in a Ceast model HDT 6 VICAT using a heating rate of 120 $^\circ$C h$^{-1}$. The temperature was determined after the needle penetrating 1 mm into the specimens. Results were analyzed with an average of three specimens.

Hardness test was performed according to ASTM D2240 in Shore-Durometer Hardness Type ‘D’ with 10 N load controlled by calibrated springs through durometer standardized indices. Results were analyzed with an average of 7 measurements.

Scanning electron microscopy (SEM) analyzes were performed on fractured surface of impact specimens. A Shimadzu SSX-550 Superscan, was used at voltage of 30 kV under high vacuum. Investigated fractured surfaces were gold coated.

**Results and discussion**

**Scanning electron microscopy (SEM)**

Figure 2 shows SEM images of PS/SBRr and PS/SBRr/SEBS blends, which reveal immiscible character, with voids presence and dispersed particles. In addition, low interaction (black arrow) is noted, which suggests poor adhesion between PS and SBRr, most due to the difference in interfacial tension between blend components heading to the weak interface [27]. Observed morphology for this system corroborates the impact strength results, displayed at the bottom of composition triangle in figure 3.

Addition of 5 and 10% of SEBS modified the morphological blends profile, with significant reduction of voids, suggesting increase in the morphological structure stability. Therefore, morphological differences are observed depending on the blends components, for instance, blends with 47.5 and 50% of SBRr still have some voids, nevertheless only few are verified, this fact indicates that higher contents of SBRr head to a denser morphology voidless. Gathered morphologies clarify the lower impact performance of SBRr-rich blends compared to SEBS blends. At 5% SEBS evidences of plastic deformation is observed in the interface between PS and SBRr particles, with fibrillar structure indicated by the white arrow as presented in figure 2(d). At the same time, some particles are well adhered to the plane (black circles), which is important for the stress transfer. In these blends, some particles with poor adhesion are visualized, indicating higher proportions of SBRr may hinder compatibilizer migration to the interface or the SEBS amount is below than necessary. Indeed, when analyzing the impact strength response surface (figure 3(b)), it is noted that high concentrations of SEBS optimizes impact, indicating greater morphological stability.
In Figure 2(e), 10% SEBS compatible blend clearly shows the highest plastic deformation level, indicating synergistic effect. There is no evidence of porous structure, important fact to maximize energy dissipation. Well adhered particles are observed in the plane, indicating the interface between PS and SBRr did not break, suggesting reduction in interfacial tension; corroborating impact strength results, elongation at break and tensile
strength, which were higher for this system. Related to impact strength, its performance increase was intensified as SEBS content increased, reaching values in the range of 80–95 J m\(^{-1}\), as shown in figure 3.

**Impact strength**

Impact strength is one of the most important parameters in polymer selection for engineering applications and often used as a decision factor [28]. Commonly, PS’ impact is increased by adding an elastomeric phase, a rubbery phase usually assists in the toughening [29, 30]. Figures 3(a), (b) shows the response surface and the threshold graph for impact strength of the blends.

Blends impact decreased upon increasing of PS and SBRr contents (yellow and green zones in the threshold graph, respectively). Due to its brittle character, PS contributes to reduce impact, meanwhile, in high proportions, SBRr decreases interfacial adhesion with its vulcanized character. Indeed, it is reported [31–33] high addition of vulcanized rubber as particulate filler reduces properties as result from the poor phase adhesion and stress concentration around the rubber particles. Therefore, in this work, vulcanized rubber powder in PS/SBRr SEBS decreased its properties.

Nevertheless, impact strength may be adjusted through composition changing, i.e., higher PS, lower SBRr and lower SEBS contents (yellow zone close to 100% PS) providing values near to 75 J/m. This finding suggests adding higher SBRr amounts in PS/SBRr/SEBS molecular interactions are less prone to take place; whereas SEBS promotes more significant interactions when PS is in greater proportion, suggesting greater synergism for the system and corroborating to the morphological character as presented in figure 2(b). From these data is reasonable assume more significant interactions occur between PS/SEBS rather than SBRr/SEBS, which justifies the higher impact strength in PS (yellow) rich region compared to the highest SBRr (green) content.

In figure 5, it is observed when SEBS content is kept close to 10%, high impact values are achieved, as displayed in the red region of figure 3(b), with data ranging from 80 to 90 J m\(^{-1}\). There are strong indications SEBS is the proper component to improve impact, at high concentrations is believed to promote greater interactions between SEBS styrene molecules and PS and SBRr styrenic segments, albeit in greater proportion with PS. The results indicate addition of SEBS to PS/SBRr can increase adhesion between PS matrix and SBRr domains by modifying the interface. This hypothesis was confirmed with the morphological analysis discussed above, mainly in figure 2(e).

Important to pay attention increasing compatibilizer concentration obviously increases impact, nevertheless, this increase depends on PS and SBRr ratio, which is direct evidence that variables interact with each other and may increase or deteriorate impact strength, depending on defined blend composition. Therefore, if impact is required, the dark green region of figure 3(b) should be avoided as it provides lower impact values, SBRr added should be controlled to not deteriorate impact in PS/SBRr/SEBS blends.

**Tensile properties**

In figures 4(a), (b) the response surface and threshold graph is displayed for tensile strength of investigated blends, it can be observed tensile strength decreased upon increasing content of SEBS or SBRr, which is related to elastomeric dispersed particles and consequent increase on the macromolecular flexibility, in this case, blends...
deform at lower stresses, as observed in the SBRr and SEBS rich regions (green area). Only when PS content is greater than 0.75% the tensile strength reaches higher values, in accordance to the small dark red area of figure 4(b). The increase in the benzene side groups hinders molecular mobility, requiring higher tensile load to fracture the specimens [34].

Figure 4(b) shows a critical region (dark green) to obtain lower tensile strength (14.6 MPa), using high concentrations of SEBS and combination of low PS and SBRr contents. There are strong indications that initiates a process of SEBS saturation in the system, helping to reduce the tensile strength. In fact, SEBS composition contains 70% of elastomeric phase and by increasing the compatibilizer content in blends, there is a trend to increase the system flexibility. This approach corroborates impact results, whereas the compatibilizer content increases the greater the impact strength, i.e., with higher dissipation energy level, while reducing the tensile strength.

Literature [35] has reported that increasing an elastomeric component content up to 13% in PS matrix it reduces tensile strength to approximately 14.5 MPa as the system becomes more flexible. However, consideration should be given to the added rubber type to toughen PS. In PS/SBRr/SEBS blends they were formulated with contents higher than 45% SBRr, and yet tensile strength was higher than 14.6 MPa.

Figure 4(b) shows that when using lower SEBS, PS (>0.75%) and SBRr (<0.75%) ratios, critical values are achieved in compositions near to the lower PS vertex, i.e., dark red zone, indicating tensile strength maximization. A light red band (>0.25 and <0.5%) can be observed in this case for higher SBRr levels, where tensile strength around 15.8 MPa was obtained.

Figures 5(a), (b) illustrates the response surface and threshold graph for elastic modulus of the blends, the highest elastic modulus values were found for compounds localized near the dark red vertex, corresponding to the highest SBRr contents. At the same time, a wide range of red region is verified. As the SEBS content increases to 0.75%, the elastic modulus is kept at high standard, suggesting the compatibilizer promotes adhesion. However, when adding SEBS content higher than 0.75%, it tends to elastic modulus decaying, indicating that compatibilizer content has already saturated within the system, therefore, increasing flexibility. Distinct behavior is observed when PS concentration increases, high elastic modulus is obtained when using PS in the range of 0.25%–0.5%, but when exceeding the 0.5% PS limit content there is a continuous reduction as verified in the green region. This result was not expected, since blends with higher PS content were expected to have higher stiffness and, upon increasing SBRr and SEBS contents, stiffness would decrease. It is believed this behavior may be associated with the crosslinkings in SBRr phase, providing rigidity to the system, as well as the presence of mineral fillers. Indeed, the final properties of vulcanized rubber generally depend on the crosslink density as also of the type and fillers amount in the formulation. Generally, the higher the crosslinking content, the vulcanized rubber tends to be harder and stiffer. Therefore, it is reasonable to suggest that used SBRr in this work may contain a high level of crosslinking, as well as the presence of loads, impacting the different behavior on elastic modulus presented in figure 5. At the same time, it is observed that even using high SBRr content within intermediate SEBS composition (0.5 to 0.75%), the elastic modulus remains in high levels, there may be proper adhesion with SBRr and PS, contributing to this synergistic effect.
In figure 6 elongation at break data is shown whereas low values were found in SBRr-rich blends as presented in the large green region of the triangle, in this case high SBRr contents weak the interfacial adhesion. In a first view, the elongation behavior in the SBRr rich region seems contradictory, since the great characteristic of rubbers is high elastic deformation. However, in the present study, the elongation of neat SBRr was not analyzed, but rather a mixture of vulcanized powder and polystyrene. In this case, there is phase separation between PS and SBRr, as seen in the morphological analyzes. Then the elongation level at rupture will be dependent on the degree of interfacial adhesion. Indeed, literature \[25, 26\] reported vulcanized rubber addition does not significantly conduct to increased elongation at break due to poor interfacial adhesion. However, upon addition of suitable compatibilizer, improvements on this property are reached. Maximum elongation is observed for SEBS rich blends, according to the small red region. Apparently, SEBS is paramount to improve the adhesion lack between PS and SBRr phases, heading to increased elongation at break. An interesting fact was verified, although PS is inherently rigid with low deformation, in PS rich blend with low SEBS and SBRr contents high elongation was reached according to the light red region near PS vertex. In principle, critical compositions are required for this property, suggesting depending on the performed formulation the property can be optimized. From composition triangle can be noted that high PS content, with low SBRr (<12.5%) and SEBS below to 0.25%, the elongation trends to values close to the SEBS rich blends (>0.75%).
Aquired toughness data are presented in figure 7, whose behavior is similar to that found in impact strength, i.e., addition of SEBS resulted in its significant improvement. Higher toughness was obtained in SEBS-rich mixtures (>0.75%) which can be seen in the large dark red region of the triangle, whereas lower toughness (<190 J m$^{-3}$) was found in blends with higher PS and SBRr contents, which are located in the yellow and green region of figure 7. Upon a maximum SBRr content, according to dark green region, toughness trended to decrease attaining lower performance (170 J m$^{-3}$). Undoubtedly, high SBRr content should hinder proper adhesion to PS matrix and microvoids are likely developed in the interface [25, 26] which result in decreased toughness which agrees with impact strength and elongation at break results.

**Shore D hardness**

In figures 8(a), (b) the response surface and threshold graph for Shore D hardness of the investigated blends is presented. Higher Shore D hardness was observed for higher PS contents, which agrees with the red region in the composition triangle. Generally, rubber addition trends to decrease the hardness due to flexible character reached [36]. In figure 8(b) can be verified that Shore D hardness changes with SBRr/SEBS ratio, upon high content of SBRr and low of SEBS (<0.25%), blends trend to keep high Shore D hardness, as seen in the yellow and orange regions; in this case, the prominent factor is the vulcanized residue of SBRr, which presents crosslinked structure providing stiffness. At high SEBS contents (>0.5%), Shore D hardness is reduced, as...
displayed in the green region. Unlike the previous case, SEBS is non-vulcanized thermoplastic elastomer and therefore acts softening blends surface, as well as reducing the penetration resistance. This finding is reinforced when analyzing the region of low SEBS content and high PS and SBRr, where the highest Shore D hardness is found (dark red to yellow zone in the composition triangle).

Shore D hardness test evaluates the polymer surface hardness, being dependent on the type of polymeric matrix. Generally, the continuous phase makes a major contribution to the Shore D hardness of polymer blends. As PS is a rigid polymer (Elastic modulus in the range from 2.8 to 3.5 GPa) it has high Shore D hardness, thus contributing to the found high values in the red region of the composition triangle, as shown in figure 8(b). As SEBS content increased, Shore D hardness tended to decrease, suggesting this parameter does not depend on the degree of interfacial adhesion. This finding is reinforced by the SEBS rich region (dark green), with the lowest hardness values. At the same time, as SEBS is a thermoplastic elastomer and has low elastic modulus, it contributed to decrease the Shore D hardness of compatible blends compared to PS/SBRr blend (compounded only with crosslinked SBRr). Shore D hardness is believed to be largely dependent on the surface characteristics of the blends and, therefore, overlapping the effect of interfacial adhesion, which justifies the different behavior of PS/SBRr blend, with a higher hardness value compared to the PS/SBRr/SEBS blends.

The Shore D hardness test is considered non-destructive, leaving only small impressions on the specimen surface. In this case, it is reinforced the hypothesis that this property is affected by surface characteristics and, on the other hand, does not depend on the degree of interfacial adhesion between the constituent phases of the blends, justifying the reduction of Shore D hardness upon SEBS increasing.

Adding vulcanized rubber powder up to 50% by weight in PS did not significantly change Shore D hardness, as shown in figure 8(b), although such statement cannot be generalized to all vulcanized rubber powder, as performance will be proportional to the crosslinking amount.

**Heat deflection temperature (HDT)**

HDT is relevant analysis in polymer science as it measures thermomechanical stability [37], it becomes required property for material selection, as changes commonly are verified in the product structural stability when simultaneously submitted to stress and temperature. Figures 9(a), (b) presents the response surface and threshold graph for HDT of the blends. Blends presented high HDT values, ranging from 76 to 79 °C, this high performance is associated with polystyrene, which has high glass transition temperature (∼100 °C), as result, PS contributed to keep blends HDT at high levels. Although not significant, HDT decreased increasing SBRr content, resulting in the small green area represented in the triangle of figure 9(b); it is interesting noting HDT behaves differently when changing ratios between PS and SEBS; at higher PS content (yellow region near vertex), HDT increase is verified. Literature [38–40] has reported HDT depends on the rubber content, it is a property that significantly depends on the continuous phase.

HDT of PS/SBRr/SEBS blends was influenced by SEBS content, according to the red area. This finding suggests the compatibilizing agent had a positive effect on HDT, possibly improving interfacial adhesion between components and increasing compatibility. Increasing SEBS content seems to have greater influence on
thermomechanical resistance than in the case of higher PS contents. This is direct evidence that chemical interactions favored this property.

It is noteworthy that HDT depends on PS and SEBS ratio, for instance, at low SEBS and SBRr contents in rich PS region (yellow region), a proper thermomechanical performance is observed. At the same time, it should be paid attention even though SBRr reduces HDT at high concentrations (green region), the decrease is not intense. Vulcanized rubber generally contains mineral fillers in its composition [41, 42], therefore crosslinked rubber residue and reinforcing mineral fillers probably protected HDT against severe loss.

Vulcanized rubber generally contains mineral fillers in its composition [41, 42], therefore crosslinked rubber residue and reinforcing mineral fillers probably protected HDT against severe loss.

Vicat softening temperature (VST)

Figures 10(a, b) shows acquired data for VST, where higher data is observed for the blends whit high PS content, as displayed in the dark red region of the composition triangle, reaching 82 °C. Rubber addition trends to decrease hardness and consequently contributes to lower VST. When adding higher SBRr and SEBS contents, blends become more flexible, thus reducing VST as shown in figure 10(b). However, it can be verified at higher SEBS content (>0.75%), intense reduction in VST is verified, according to the small dark green area in the triangle of figure 10(b), thus high thermoplastic elastomer concentrations denigrates VST, this finding is reinforced when analyzing the region of low SEBS content (<0.25%) and high PS (>0.75%) and SBRr (≤0.5%) ones, in which VST are the highest (dark red to orange range of the composition triangle).

Conclusions

RSM was used to elucidate mechanical performance of PS/SBRr/SEBS blends, this is a helpful technique for blends development since guides to reach optimal compositions thus attaining maximized properties. In this work, improved impact strength, elongation at break, toughness and HDT were obtained for SEBS rich blends; whereas surface hardness-dependent properties were obtained in neat PS rich ones. Nevertheless, upon detailed analysis of threshold graphs intermediate properties can be reached, aiming at proper ratio between composition and properties. To predict aimed properties within wide compositional range is the great contribution acquired with RSM technique.

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