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Influence of maleic anhydride grafting on the rheological properties of polyethylene terephthalate–styrene butadiene blends

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Abstract

The rheological properties of polyethylene terephthalate (PET)–styrene butadiene rubber (SBR) blends are studied in this work. The shear viscosity was measured in a special capillary device mounted on an injection-molding machine, which provides data on pressure and flow rate. Comparison of the viscosity of the PET–SBR physical blend with that of the blend of PET with maleic anhydride-functionalized SBR (SBRg) provides information on the effect of grafted maleic anhydride chains at the rubber–thermoplastic interface on the rheological properties of the blend. Shear viscosity is a function of the amount of compatibilizer and applied shear stress present in the preparation of the blends. A reduction in the particle size due to increasing screw speed was correlated with a reduction in the shear viscosity and an increase in the impact properties of the blend. © 2001 Elsevier Science Ltd. All rights reserved.

Keywords: Maleic anhydride grafting; PET–SBR blends; Rheology of polymer blends

1. Introduction

An adequate polymer-blend process requires information on, and understanding of, the blend flow behavior under production process conditions. The knowledge of the relationship among melt viscosity, elasticity, shear rate, pressure and process temperature is important for the design of molds and appropriate process equipment selection, as well as for assessment of the optimum process conditions.

Polymer blends should merge the best properties of each of their components, but the production of polymer systems by mixing different precursor polymers complicates their rheology. The problem of blend production has been eased by the use of extruders in which chemical reactions take place to induce compatibility among the blend ingredients. Reactive extrusion has the advantage of controlling temperature, mix distribution and dispersion, residence time, and reaction under pressure, and also ensures the continuity of the process.

Polymeric blends are dispersions of polymer drops embedded in a melted polymeric matrix. Their mechanical and rheological properties depend on shape, size, interparticle distance and orientation of the dispersed phase. It has been found that the addition of interfacial agents can improve the properties of immiscible polymer blends, because these modifiers decrease the interfacial tension between the phases in contact, inhibiting coalescence among particles [1–3].

Attention has been given to the influence of the dispersion state on the rheological properties of blends produced by reactive extrusion, using compatibilizers or functional chemical groups grafted onto one of the components of the blend. (For instance, the reactive extrusion of mixtures of polyethylene terephthalate (PET) with other polymers like PET-PP [4], PET-LCP [5,6], PET-PC [7], and other systems prepared by similar methods such as PMMA-PVDF [8], PE-PS [9], PP-EMA-EVA [10], PP-MAH-DCP [11] and PP-SBS [12]) Likewise, mathematical, theoretical and process tools have been developed for the design of new blends [13–15] and also new devices have been developed for measuring rheological properties of PS-$CaCO_3$ [16] and PP-fillers [17].

It has been shown that the PET–styrene butadiene rubber (SBR) blend leads to incompatibility, which is reflected in its low-impact resistance when compared to that of pure PET [18]. To induce compatibility and to improve impact resistance, SBR grafted with maleic anhydride (MAH) provides polar groups that are responsible for an increase in the PET–SBR interfacial interaction, which leads to improved compatibility and better mechanical and impact

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properties. This chemical modification has a considerable effect on the rheological properties of the PET–SBRg blend.

Most analyses on compatibilized blends by interfacial agents deal with large effects observed on the morphology of the resulting system, especially with regard to particle size and phase characteristics. However, not enough attention is given to the relationship between the variables involved in the preparation of the blend and its resulting rheological properties. In this work, attention is given to the preparation procedure-viscosity relation of the PET–SBR blend, where the SBR was chemically modified by grafting MAH groups. Melt viscosity measurements of the resulting blends were studied as a function of the processing variables inherent in the preparation of the blends. These are screw speed (which provides different particle sizes and dispersion degrees of rubber particles in the PET matrix), MAH percentage in the mixture (which provides various levels of interfacial activity) and SBR concentration in the PET matrix. Viscosity determinations were carried out in a capillary rheometer designed to operate on an injection thermoplastic machine. The advantage of this device is that the viscosity measurements are obtained online at the processing conditions and under the process history of the material, which involves variables such as cylinder temperature, injection speed and dosage speed in the range of shear rates of an industrial process.

2. Experimental procedure

2.1. Materials

PET from Celanese had a density of 1.425 g/cm^3 according to ASTM D792, a melting point of 249°C, and crystallinity of 37% [19,20]. A glass transition temperature of 74°C was determined by Differential Scanning Calorimetry (DSC) with a heating speed of 10°C/min under a nitrogen atmosphere. An average molecular weight of 26,000 g/mol was determined by high temperature GPC using *m*-cresol. An intrinsic viscosity of 0.850 dl/g [21] was measured with an Ubbelhode viscometer. SBR from Dynasol (Solprene 416) had a density of 0.919 g/cm^3 according to ASTM D792, a styrene block content of 30% [22] and an average molecular weight of 105,500 determined by high temperature GPC using 1,2,4 trichlorobenzene. A glass transition temperature of −89°C was determined by DSC with a heating speed of 10°C/min under a nitrogen atmosphere. The MAH was recrystallized twice using chloroform at 53–54°C (previously dried with P_2O_5) under stirring with reflux. The resulting solution was vacuum-filtered and the soluble portion was cooled up to precipitation. The precipitated material was dried under vacuum and crystals of MAH were obtained. Benzoyl peroxide (BPO) was used as received. Two additives to prevent oxidation were used: Irganox 1076 for the rubber and Irganox 1010 for PET, from Ciba-Geigy.

2.2. Equipment

A Haake Rheocord 90 TW100 twin-screw conical counter-rotating extruder with length of 331 mm was used in the reactive extrusion process. Ground samples were dried in a Pagani dehumidifier with molecular meshes. The rheological measurements were made in a Demag Ergotech 50 injection-molding machine. Particle diameter and interparticle distances were measured in post-quenched samples in a Jeol JSM-70 scanning electron microscope, using an image analyzer system. The average diameters of and distances among particles were evaluated, taking into account 200 particles for each sample. The particle diameter was measured in molded samples after quenching, following the procedures generally employed for these purposes, which it is assumed that there is little change in the particle size and particle position before and after the quenching operation.

2.3. Grafting reaction and blending procedure

The reaction to obtain grafted rubber (SBRg) and the procedure to prepare the PET–SBRg blends have been reported elsewhere [18,23,24]. In the latter reference it was shown that the molecular weight of the grafted polybutadiene chain does not diminish with the grafting reaction, and that the grafted groups can attach either to a 1,2 vinyl position or along the backbone (position 1,4) of the polybutadiene chain. At low MAH contents, it was further found that the grafting reaction takes place mostly in the 1,2 vinyl position, while for higher MAH contents, the grafting takes place along the polymer chain. In this work, the extruder screw speed for grafting was adjusted to 70 rpm and that for blending was set at 50 rpm. The MAH concentration was 2 phr in most cases and the initiator concentration, BPO, was 3% with respect to MAH amount. Extrusion temperature for the grafting reaction was set at 160°C and the extrusion temperature for blending was 270°C. Evaluation of degradation effects was made by preparing the blends at different screw speeds and, particularly in the case of PET, comparisons were made among samples with different thermorheological histories.

3. Results

3.1. Procedure to obtain the PET–SBR blends

The previously dried components are mixed in a twin-screw conical counter-rotating extruder and the screw rotational speed (rpm) is varied to obtain blends made according to different processing conditions. Various particle sizes and inter-particle distances were obtained in the dispersed phase. The viscosity measurements were carried out in a capillary rheometer mounted on an injection machine (see Fig. 1). To assess the suitability of this method, the viscosity of PET was determined primarily by extrusion capillary

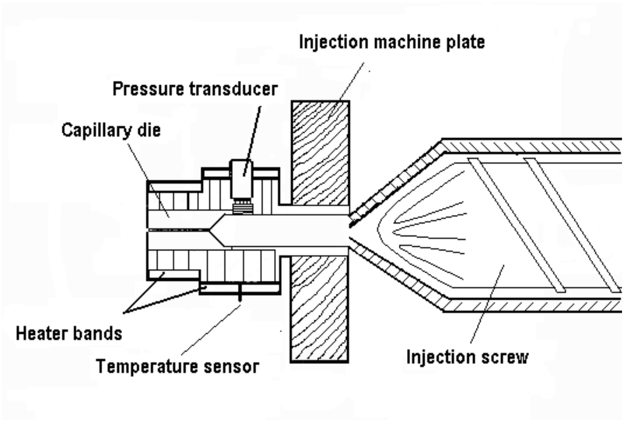

Fig. 1. Schematic representation of the capillary rheometer mounted on an injection-molding machine.

rheometry and subsequently the viscosity was measured in the injection-molding machine. The same capillaries were used in both techniques and the Rabinowitsch correction was applied. The viscosity values obtained through both methods are shown in Fig. 2. It is observed that in the injection method the viscosity line lies slightly under the extrapolation of the extrusion curve. Different thermo-mechanical histories and residence times of the fluid in the devices may explain this departure.

3.2. *Effect of SBR concentration on melt viscosity. PET–SBR physical blends (without grafting)*

Results of the viscosity variation with the shear rate for the PET–SBR blends without grafting, and for several rubber concentrations, are shown in Fig. 3(a) and (b). As mentioned in the preparation procedure, blends were produced at a screw rotational speed of 50 rpm. In Fig. 3(a), rubber contents lie in the range 3–15 phr, whereas in Fig. 3(b) rubber concentration varies from 20 to 50 phr. The viscosity of PET alone was determined either by using the polymer as received or in samples already extruded once. As observed, the PET online viscosity presents a substantial decrease due to thermo-mechanical degradation at the processing conditions [25].

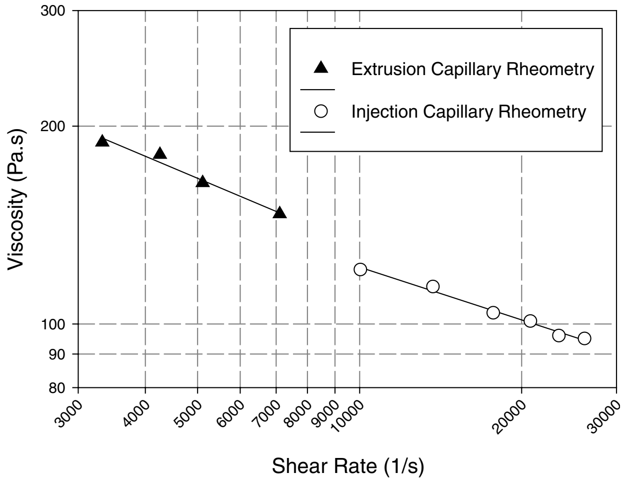

Fig. 2. Comparison between sets of viscosity data obtained in the extruder and in the injection-molding machine, respectively.

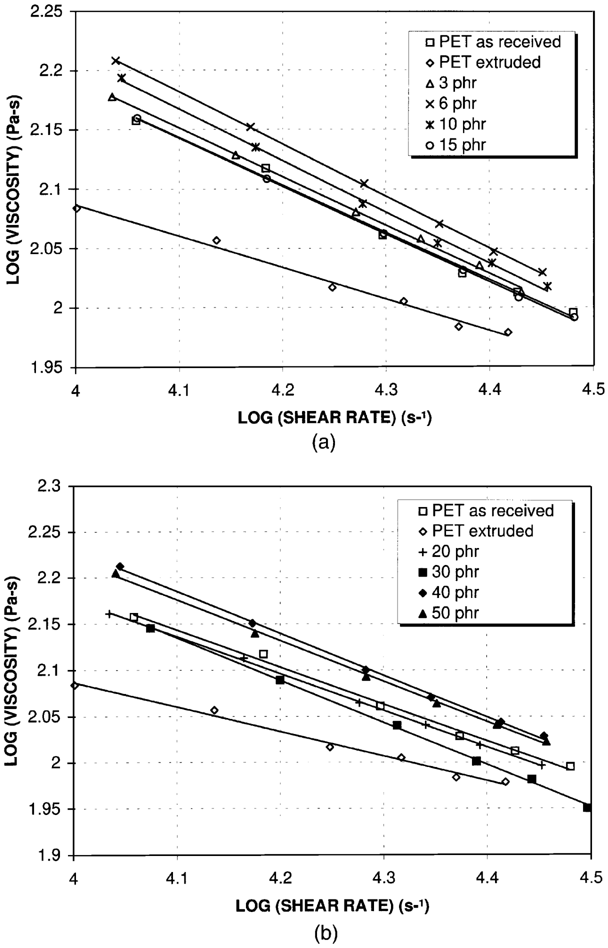

Fig. 3. (a) PET–SBR viscosity as a function of shear rate for various SBR contents in the blend (3–15 phr). Blends were prepared at 50 rpm screw rotational speed. (b) Same as (a) with SBR contents of 20–50 phr.

It is interesting that the viscosity variation with shear rate in the physical blends is such that the viscosity curves lie above the PET curve. The rubber particles induce an increasing resistance to flow, as observed in polymer-filler systems [26]. Results seen in these plots reflect a low degree of dispersion of the rubber particles in the matrix, with presumably high coalescence to form agglomerates.

3.3. *PET–SBR blends with grafted rubber*

The blend of PET and SBR grafted with MAH groups (PET–SBRg) possesses lower viscosity than the physical blend (PET–SBR), as shown in Fig. 4(a) and (b) for low and high rubber contents, respectively. Blends were prepared at an extrusion speed of 50 rpm with a MAH content of 2 phr. As the rubber amount increases (from 3 to 20 phr) the viscosity diminishes gradually down to the

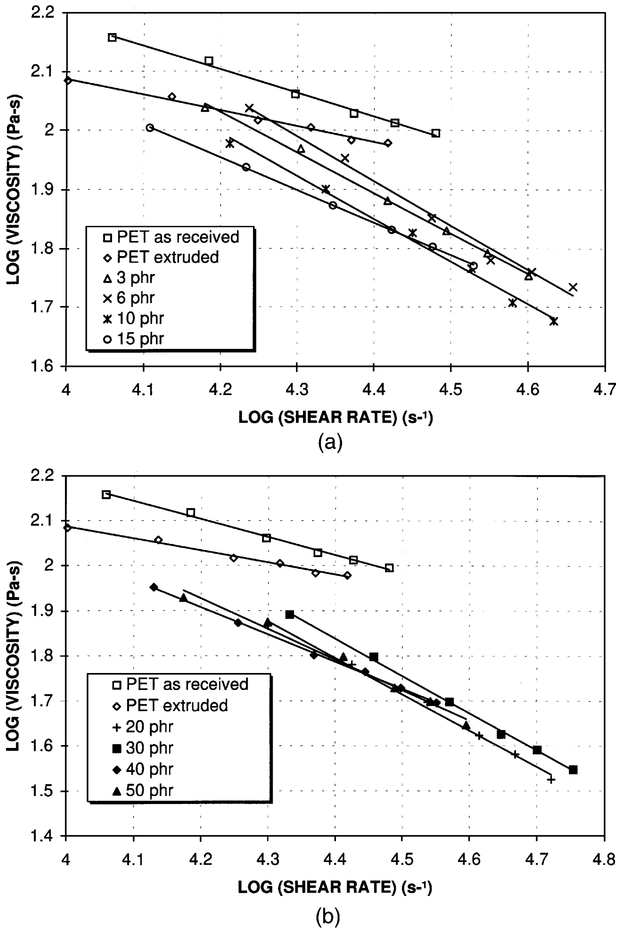

Fig. 4. (a) PET–SBRg viscosity as a function of shear rate for various SBRg contents in the blend (3–15 phr). Blends were prepared at 50 rpm screw rotational speed with a MAH content of 2 phr. (b) Same as (a) with SBRg contents of 20–50 phr.

highest concentrations (30–50 phr), where it levels off. MAH grafting provides polar groups, which are also reactive groups that may induce an alcoholysis reaction with the terminal hydroxyl groups of PET, producing the corresponding co-polyester PET–SBRg. The MAH grafting may produce a better interfacial adhesion of the rubber to the PET matrix, and simultaneously inhibits coalescence, resulting in an improved dispersion. The effect of reduced coalescence is combined with the breakage of agglomerates due to high shear stresses, which leads to an overall decrease in the blend viscosity, similar to the effect of processing aids [27].

3.4. Rheological properties

Variation of the viscosity with shear rate suggests the application of the power-law model for the specific range of shear rates studied. The variation of the power-law index for PET–SBRg and PET–SBR is shown in Table 1. Power-law indexes for blends with rubber without grafting are practically invariant, attaining a mean value of 0.55–0.57, while in blends with grafted rubber large variations are observed. The PET–SBRg indexes are lower than those of the PET–SBR physical blends, implying a high pseudoplasticity in the compatibilized blends. These observations suggest that the overall rheological behavior is substantially influenced by increased matrix–particle interaction.

Table 1
Power-law index from PET–SBR blends, with and without MAH grafting (2 phr MAH)

Rubber concentration (phr)	PET–SBRg	PET–SBR
0	0.73	0.73
3	0.31	0.59
6	0.24	0.56
10	0.27	0.56
15	0.44	0.59
20	0.19	0.60
30	0.17	0.54
40	0.40	0.54
50	0.32	0.56

3.5. Influence of MAH concentration on the rheological properties

In Fig. 4(a) and (b), the results of viscosity are presented considering a fixed MAH concentration of 2 phr. However, it is important to analyze the effect of the amount of grafting on the overall blend viscosity. It may be advanced that increasing the amount of graft provides better particle dispersion and coalescence inhibition. The effect of varying the MAH concentration used in the SBR grafting process for a fixed rubber concentration (10 phr) is shown in Figs. 5 and 6(a). The interfacial action of the grafted groups is clearly shown, as the viscosity is plotted with the shear rate (Fig. 5) and with the MAH content (Fig. 6(a)). In Fig. 5, the first group of curves, corresponding to 0.5, 1.0 and 1.5 phr of MAH concentration, lies above the PET viscosity curve,

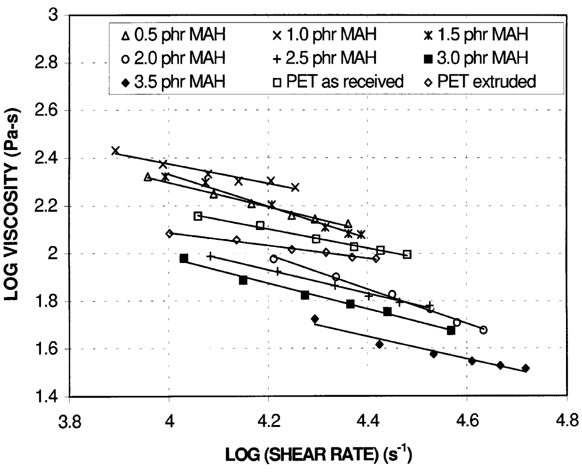

Fig. 5. Viscosity of PET–SBRg blends as a function of shear rate. Concentration of SBRg is 10 phr. Blends were prepared considering several MAH contents at 50 rpm screw rotational speed.

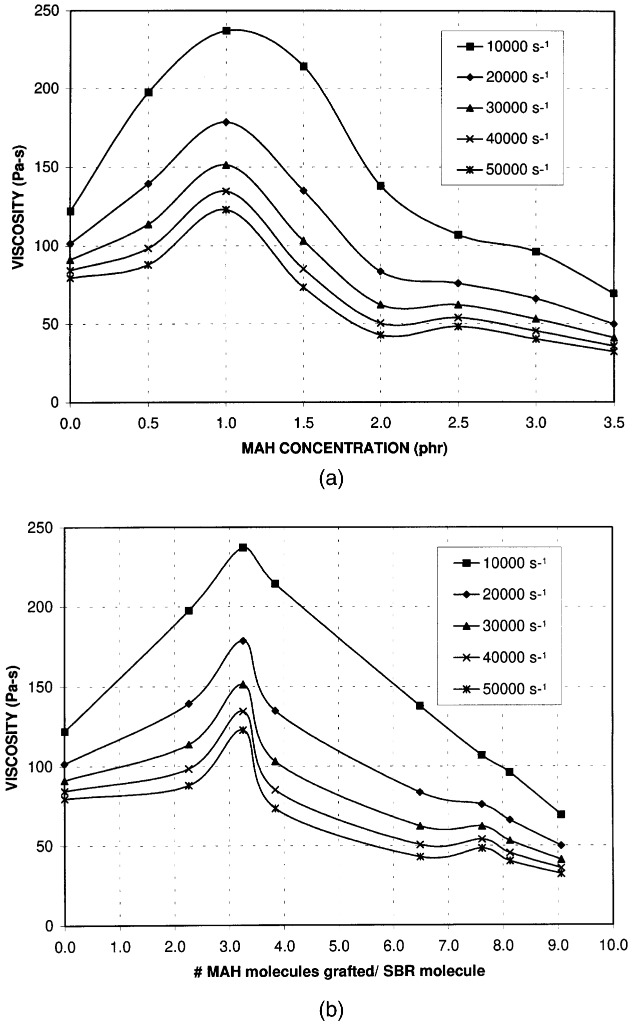

Fig. 6. (a) Viscosity of PET–SBRg blends as a function of the amount of MAH used in the preparation of the blends, for various shear rates. Concentration of SBRg is 10 phr. Screw rotational speed is 50 rpm. (b) Viscosity of PET–SBRg blends as a function of the amount of MAH molecules grafted per SBR molecule. Same conditions as in (a).

Table 2
Effect of MAH concentration on the resulting percentage of grafting and impact resistance. SBR content in blend: 10 phr

MAH added in grafting process (phr)	% MAH grafted/ Available sites in SBR	Izod impact notched (J/m)
0	0	38
0.5	0.212	77
1.0	0.305	69
1.5	0.360	64
2.0	0.608	104
2.5	0.714	68
3.0	0.762	75
3.5	0.849	45

while the second group has larger MAH concentrations (2–3.5 phr) and smaller viscosity than that of PET. To illustrate the variation of viscosity with MAH concentration for a given shear rate, data from Fig. 5 is plotted in a different form in Fig. 6(a), where some points have been interpolated and extrapolated, without changing the qualitative trends of the results. A steep drop in viscosity is observed between 1 and 2 phr of MAH, becoming lower than the viscosity of PET alone, for most shear rates. The MAH concentration used in the grafting reaction is proportional to the percentage of MAH grafted in the unsaturated butadiene chain, as illustrated in Table 2. Lower viscosity may be the result of better dispersion with inhibited coalescence, induced by the effect of the grafted groups on SBR, which begins at a critical concentration of MAH, this is, for concentrations larger than 2%. It is precisely at this concentration that the impact strength attains a maximum, as shown in Table 2. In this table, simultaneously, it is shown that the amount of MAH used in the grafting process scales with the amount of MAH grafted.

To illustrate quantitatively the action of the grafted groups on the resulting viscosity, Fig. 6(b) shows the viscosity plotted against the number of MAH molecules grafted per SBR molecule, calculated from the knowledge of the rubber molecular weight, rubber proportion in SBR and percentage of grafting, from Table 2. The resulting variation is qualitatively similar to that shown in Fig. 6(a), illustrating further that a small number of grafted molecules of MAH in the polybutadiene chain produces a large effect on the rheological properties of the resulting blend.

3.6. Effect of extrusion speed used in the preparation of the blends on blend viscosity

With the purpose of relating method of preparation of the blends to the resulting viscosity measured in the injection-molding machine, blends were prepared at various extrusion speeds. As was demonstrated in a previous study [18], the highest value of Izod-impact resistance of the PET–SBRg blend is obtained at a rubber concentration of 15 phr. For this specific rubber concentration, and with 2 phr of MAH, the extrusion speed is varied with the purpose of relating the shear stress, which produces different particle size and inter-particle distances, with the resulting viscosity. In Fig. 7 is shown that for different extrusion speeds (rpm) and therefore different rubber particle sizes, the viscosity curves from different blends are separated into two groups. Those with higher viscosity correspond to larger particle sizes (extruded at 20, 30 and 40 rpm) and those of smaller particle size (extruded from 50 to 125 rpm) show lower viscosity. These data are supported by measurements of particle diameter taken in fractured samples (analyzed elsewhere, see Ref. [18]) shown in Fig. 8, where the experimentally determined particle diameter is plotted with extrusion speed. Between 40 and 50 rpm, a steep drop in the diameter is observed, from 20 μm down to around 6 μm. Further increases in the screw speed do not change the particle size.

To illustrate the variation of viscosity with extrusion

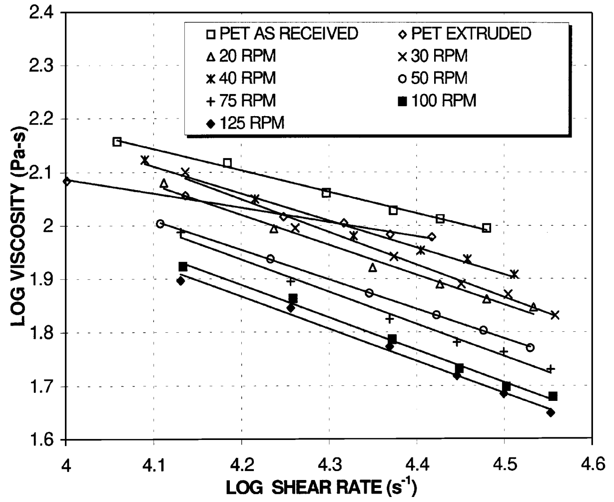

Fig. 7. Viscosity of PET–SBRg blends in function of shear rate. SBRg content is 15 phr, and the MAH concentration used in the blends preparation is 2 phr. Blends were prepared at several extrusion speeds.

speed used in the blend preparation for a given shear rate measured in the injection-molding machine, data from Fig. 7 is re-plotted in Fig. 9. A pronounced viscosity decrement within the 40–50 rpm interval is apparent, illustrating the close relationship between particle size and viscosity of the blend, i.e. between the shear stress acting in the blend preparation and the resulting viscosity of the blend. High particle deformation may lead to breakage, and hence lower particle size. Injection molding provides very high shear rates that induce high deformations and particle breakage, and this is reflected in the steep drop in particle diameter shown in Fig. 8.

It is interesting that other properties improve with reduction in particle size and better dispersion. For example, impact strength increases steeply in the same range as the viscocity decreases (see Table 3). In the region where the viscosity is high, impact properties are smaller than those of PET alone (20–40 rpm), corresponding to the dispersion of large particles (or agglomerates) with large inter-particle distances. Consequently, processing speeds of more than 50 rpm in the compatibilized blend correspond to small particle size, small inter-particle distance, high impact strength and low viscosity. The blend viscosity remains practically unchanged for higher speeds, like the particle diameter variation shown in Fig. 8.

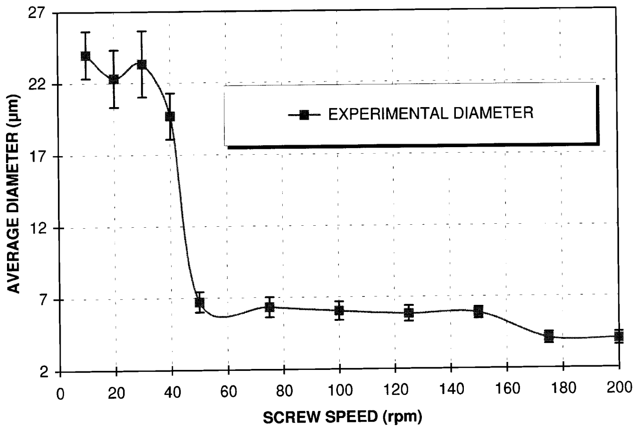

Fig. 8. Average particle diameter of the PET–SBRg blend plotted as a function of the extrusion speed used in the preparation of the blends. SBRg content is 15 phr and the MAH concentration is 2 phr.

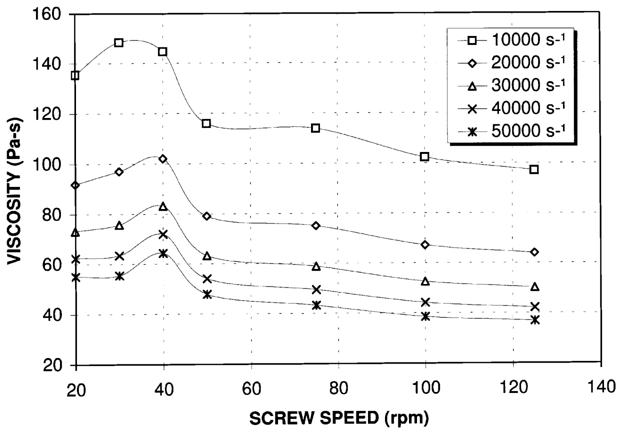

Fig. 9. Viscosity of the PET–SBRg blends plotted as a function of extrusion speed for several shear rates. Same conditions as in Fig. 7.

4. Discussions

The two principal results of the interfacial modification in the PET–SBR blends studied in this work are summarized in Figs. 6(a) and 9. These are for blends prepared keeping the SBRg concentration constant (10 phr in the systems shown in Fig. 6(a) and 15 phr in those of Fig. 9). Degradation effects in the systems shown in Fig. 6(a) are similar, because all samples undergo the same thermorheological history; the viscosity variation with increasing MAH content is thus due to the amount of grafted groups. Blends shown in Fig. 6(a) were prepared at 50 rpm, which fixes the

Table 3
Effect of screw speed on particle diameter, interparticle distance and impact Izod resistance. SBRg concentration in blend: 15 phr. MAH content is 2 phr

Extrusion screw speed (rpm)	Average particle diameter (μm)	Average interparticle distance (μm)	Izod impact notched (J/m)
10	24	48	24
20	22	39	31
30	23	34	28
40	20	23	28
50	7	20	123
75	6	17	119
100	6	15	119
125	6	14	117
150	6	14	118
175	4	11	138
200	4	10	135

particle size, and therefore differences in the viscosity of these blends are due to factors other than particle diameter. Coalescence inhibition, variations in the MAH grafting place along the molecular chain and better dispersion caused by the grafting may account for the observed reductions in blend viscosity with increasing MAH content. On the other hand, over an extended rpm range, there is a relationship between particle size (Fig. 8) and viscosity (Fig. 9). Smaller particle diameters correspond to lower viscosity and higher impact properties (Table 3).

In the present work, the reduction in particle size observed in Fig. 8 is observed in both grafted and ungrafted rubber blends, (plot of reduction in particle size for ungrafted blends is not shown). Particle size is 7 μm for the grafted blends and 8 μm for the ungrafted systems, and both were prepared at the same speed (50 rpm). This confirms that differences in the viscosity between the systems shown in Figs. 3(a and b) and 4(a and b) are not attributable to particle size, but to the presence of the compatibilizer. A decrease in the interfacial tension, estimated from the measured interfacial concentration of graft copolymer produced in the reactive mixing process (see Ref. [29]), is too small to account for the observed droplet sizes as being the result of emulsification. Instead, they are the result of the shear stress applied in the preparation of the blends.

Thermodynamic and rheological variables are involved in the process to induce compatibility in immiscible blends by a grafted polymer. Specifically, the blend morphology is determined by two competing process: droplet breakup (as the blend is subjected to shear and extensional deformations in the mold injection process) versus droplet coalescence (which occurs because of the convective character of shear flows). Both coalescence and breakup behavior can be effected in systems where the grafted groups place themselves at the interface of the two polymers. Smaller particles are produced by breakup if the shear stress is large enough to produce a deformation substantial enough to break agglomerates and particles, as observed in Fig. 8. On the other hand, it has been shown that a very small number of grafts, insufficient to reduce the interfacial tension considerably, may be extremely effective in suppressing coalescence [28–30]. This is clearly shown in Fig. 6(b), where the number of grafts in the polybutadiene chain is very small in comparison to the potential number of reactive places in the polymer chain.

When grafted rubber is used, the degree of compatibility of the blend components increases due to higher interfacial adhesion resulting from larger interactions between reactive groups in both SBRg and in PET. The SBR-grafted particles adhere to the PET matrix, allowing high deformations by the flow stress that leads to particle breakage. The imposed deformation and orientation have been determined in other systems, such as polyisobutylene and polydimethylsiloxane, and characterized using the capillary number [31,32]

$$Ca = \frac{\eta\dot{\gamma}R_0}{\sigma}. \qquad (1)$$

In this equation, η is the viscosity of the continuous phase, $\dot{\gamma}$ is the shear rate, R_0 is the undeformed particle radius and σ is the interfacial tension. To characterize the magnitude of deformation of particles, the deformation parameter $D = (a - b)/(a + b)$ is defined, where b is the smallest and a is the larger axis of the particle. According to the Taylor's theory [33,34], this parameter is a function of Ca and the viscosity ratio $\lambda = \eta_d/\eta_c$, where η_d and η_c are the dispersed and continuous phase viscosity, respectively. For low values of Ca,

$$D = Ca\frac{19\lambda + 16}{16\lambda + 16}. \qquad (2)$$

Particle deformation is thus proportional to the capillary number of that expresses the balance between flow forces, which tend to deform the particles, and surface tension forces, which tend to drive back the particle to an equilibrium shape. Since the action of the grafted groups on the rubber phase is to promote adhesion and compatibility to the PET matrix (surfactant action), the interfacial tension should diminish with the number of grafts, increasing the capillary number and allowing higher deformations. However, Eq. (2) does not take coalescence effects into account.

Variations in rubber composition may affect the balance between disintegration and coalescence. Increasing the content of the dispersed phase results in an increase in the particle size due to coalescence, because of the larger number of particles and particle–particle collisions. Reports [35] show that coalescence can start at values of the dispersed phase as low as 1%. In Fig. 9, blends were prepared at various speeds with grafted rubber. From 10 to 30 rpm, the particle size is larger than 22 μm, as shown in Fig. 8. The initial increase in viscosity observed from 20 to 40 rpm is related to the coalescence of large particle domains, when the rubber concentration is high (15 phr). These agglomerates are disrupted as the speed is increased to 50 rpm, producing a drop in the viscosity due to smaller particle size combined with coalescence inhibition. Note that the mechanism of restriction to coalescence works with small particle sizes produced for speeds faster than 50 rpm. In conclusion, results suggest that the effects of interfacial modification on SBRg–PET blends are the reduced coalescence and a reduction in particle size. The latter was produced by the shear stresses used in the preparation of the blends.

Coalescence inhibition by interfacial modification depends on the amount of interfacial agents, in this case, on the amount of grafted MAH groups, and also on molecular aspects, such as variations in the MAH grafting place along the polymer chain. In Fig. 6(a), for low MAH concentrations the grafting reaction takes place mostly in the 1,2 vinyl position. For higher MAH contents, the grafting occurs along the polymer chain (see Ref. [24]). Variations in the grafting site lead to different surface properties, in such a way that interactions with the PET matrix will in turn be modified, and hence the coalescence and flow behaviors.

5. Conclusions

Rheological measurements were carried out using a capillary rheometer mounted in an industrial thermoplastic injection machine. This method was tested by comparison with results obtained by extrusion capillary rheometry. The flow behavior of PET–SBR blends was affected extensively by the following factors: SBRg concentration, MAH content in SBRg, particle diameter and distance among particles in the dispersed phase. The PET–SBR blend viscosity is found to be larger than that of PET alone, and is attributed to coalescence of particle domains and agglomerates. On the other hand, the viscosity of PET–SBRg blends changes with the amount of MAH used. Coalescence inhibition, variations in the MAH grafting site along the molecular chain and better dispersion caused by the grafting may account for reduction in the blend viscosity. Improved adhesion between blend components leads to a more effective action of the shear stress to break agglomerates and to lower the mean particle diameter and inter-particle distance. The morphology of blends is thus modified. Consequently the resistance to flow is reduced and the impact strength is substantially increased.

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