Melt rheology and extrudate morphology studies of polystyrene/polybutadiene blends in the presence and absence of compatibilisers

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Abstract Melt rheology and extrudate morphology of polystyrene (PS)/polybutadiene (PB) blends were studied with special reference to the effect of blend ratio and two different types of compatibilisers, the random copolymers and block copolymers. Uncompatibilized blends had viscosities approximately intermediate between those of the component polymers. In the case of uncompatibilized blends both positive and negative deviation was observed in the viscositycomposition curve at different shear rate range studied. The viscosity of the compatibilized blend was found to increase when a few percent of block copolymer was added. However, at high concentrations of the compatibiliser, the viscosity values leveled off. This is due to interfacial saturation. Morphological investigations indicated that the size of the dispersed phase initially decreased when a few percent of the block copolymer was added followed by a clear leveling off at higher concentration. However, the random copolymer was not an effective interface modifier in agreement with microscopy study. These experimental results supports the earlier investigations of solid state NMR studies on these blends (Polymer (2005) 46(22):9385).

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Introduction

For the design and development of multiphase polymer blend systems, a thorough knowledge of the relationship between molecular characteristics of the component polymers, their rheological and interfacial properties, the melt processing conditions and the flow induced microstructure is essential [1, 2]. Rheological behavior of polymer blend is more complex and is influenced by miscibility, morphology, interfacial adhesion, interfacial area, processing conditions etc. Better knowledge of processing history will help to introduce suitable remedies for processing problems. Brydson [3] made a thorough review on the need of rheological studies in making a logical choice of the polymers and its processing conditions. Knowledge about the processability of a blend under high shearing action is essential for the fabrication of articles of good finish and dimensional tolerance. The complex rheological behavior and morphology of polymer blends have been investigated by several workers [4-12]. In our laboratory, the melt rheological properties of various thermoplastic elastomers have been studied by Thomas and coworkers [13–19].

Research into the compatibilisation of immiscible polymer blends has, during the past few decades, begun to focus on the role of block, graft co-polymers for studying rheological properties. Very often compatibilisation affects flow properties of the blends. Interactions occurring between blend components generally increase the viscosity of the system. Germain and coworkers [20], Joshi et al. [21] and Haddout and Villoutreix [22] have made contributions in this field. Puyvelde et al. [23] related rheology and morphology of compatibilised polymer blends. Potschke and coworkers [24] reported increase in melt viscosity as well as elasticity upon incorporation of reactive compatibiliser, styrene-acrylonitrile-maleic anhydride copolymer to polyamide 6(PA6) and acrylonitrilebutadiene-styrene (ABS) terpolymer. Brahimi et al. [25] showed that the complex viscosity of polyethylene/ polystyrene blends was very sensitive to the concentration and structure of the block copolymer. Yu et al. [26] made quantitative relationship between rheology and morphology in emulsions.

Thermoplastic elastomers resulting from PS/PB blends can be processed by conventional thermoplastic processing techniques. Hence, it is very essential to carry out melt rheology measurements to optimise the processing conditions. Several authors have reported the morphology-viscosity relationship of PS based blends [27, 28]. Some other reports on the rheological studies include the works of Ju et al. [29], Liao et al. [30], Lim et al. [31] and Sung et al. [32] and recent studies on compatibilised blends by Colbeaux et al. [33] and Filipe et al. [34]. Although there are several studies on PS/PB blends, till date no detailed investigations have been performed on the effect of two different compatibilisers (random and block copolymer of PS and PB) on the rheological properties of PS/PB blends. Therefore, the objective of the present study is to evaluate the role of the two compatibilisers on the viscosity of the blends. Additionally, the rheological behaviour of the blends in the presence and absence of compatibilisers will be correlated with the morphology of the system. The rheological studies (high shear) is extremely important to analyse the fabrication of these materials by injection moulding and extrusssion. Finally the effectiveness of the two compatibilisers on the rheology and morphology will be compared as discussed.

Experimental

Materials

Polystyrene, 678, SF (crystal grade) was supplied by Supreme Plastics, Mumbai.Cis-1,4 polybutadiene (PB)(Cisamer G.P.) was obtained from IPCL Vadodara, India. The glass transition temperatures of neat PB and neat PS are of -101 and 100 °C, respectively. A random copolymer of styrene and butadiene (SBR) having 30 percent of polystyrene content, $\bar{M}_w = 311792$, $\bar{M}_w/\bar{M}_n = 2.94$, T_g (-40 °C) and a triblock copolymer of styrene-butadiene-styrene (SBS) having 30% of PS were used as compatibilisers. SBR was obtained from Synthetics and Chemicals Ltd., Bareilly, U.P., India. SBS (Kraton D) was purchased from Shell chemicals, U.K. The characteristics of the materials are given in Table 1.

Preparation of blends

Blends of PS and PB were prepared in a Haake Rheocord 90 mixer. The temperature, rotor speed and mixing time were 180 °C, 60 rpm and 8 min, respectively. The melt mixed samples are denoted as S₀₀, S₃₀, S₅₀, S₇₀, and S₁₀₀, where S stands for melt mixing and the subscripts indicate polystyrene content in the blend. The compatibilised blends with 1,2.5,5 and 10 weight percentage of SBR and SBS with respect to minor phase PB are denoted as SR₇₀₀₁, SR_{702.5}, SR₇₀₀₅, SR₇₀₁₀ and SB₇₀₀₁, SB_{702.5}, SB₇₀₁₀ respectively. The mixing conditions were same as that of uncompatibilised blends.

Characterisation

Rheological measurements

Melt rheology of the blends was characterized by measuring their viscosities in elongational flow using a Capillary Rheometer (Rheoflixer SWO) with L/Dratio 30/1 and an angle of entry 180. End corrections such as Bagley correction and Rabinowitch correction were not applied on the data since a die of larger L/Dwas used. The measurements were made at 180 °C. The shear rates change from 50 to 1000 s⁻¹ was investigated. The shear rate (γ_w) values, flow behavior index (n') and the shear viscosity η were obtained directly.

Extrudate morphology analysis

The extruded samples collected in the rheological tests were cryo-fractured in liquid nitrogen. For PB rich blend, butan-2-one was used as etchant to dissolve the dispersed PS phase, and for PS rich blend, the dispersed PB phase was etched with *n*-heptane, to obtain better insight into the blend morphology. The fracture surface was dried, coated with a thin layer of

Table 1 Characteristics of materials used

Materials	Density (g cm-3)	Molecular weight \bar{M}_w
PS (atactic) PB(cis1,4) SBR SBS	1.04 0.94 0.97 0.95	$\begin{array}{l} 3.51 \times 10^5 \\ 1.25 \times 10^6 \\ 3.11 \times 10^5 \\ 2.24 \times 10^5 \end{array}$

gold. After gold coating, the morphology was examined with a Philips XL20 model scanning electron microscope operating at 20 kV. Micrographs of different magnifications were taken. The domain diameters were measured from different micrographs and several fields of view were taken for analyzing various average domain diameters as, [35] number-average, weightaverage, and volume-average diameter. The domain diameters of the dispersed phase were expressed in terms of number-average domain diameter \overline{D}_n as,

$$\bar{D}_n = \frac{\sum N_i D_i}{\sum N_i} \tag{1}$$

In these equations, N_i is the number of domains having diameter D_i .

The weight-average domain diameter,

$$\bar{D}_w = \frac{\sum N_i D_i^2}{\sum N_i D_i} \tag{2}$$

and the volume-average domain diameter,

$$\bar{D}_{\nu} = \frac{\sum N_i D_i^4}{\sum N_i D_i^3} \tag{3}$$

In these equations, N_i is the number of domains having diameter D_i . The polydispersity index [36], a measure of domain size distribution was calculated as, $PDI = \frac{\bar{D}_w}{\bar{D}_n}$.

Results and discussion

Effect of blend ratio

The viscosity-shear stress relationship for homopolymers and their uncompatibilised blends at 180 °C are shown in Fig. 1. PB has got higher melt viscosity than PS. Blends show intermediate values of component polymers. The melt viscosity of PB could not be obtained at high stress conditions. This is according to the reports, that flow through a capillary of crystallisable polymers like *cis* 1,4-polybutadiene [37], under certain conditions of temperature and shear rate becomes anomalous. In some cases the flow of the polymer stops on increasing the pressure above a certain level. It is observed that, the viscosity of all the blends decreased with increase in shear rates. The decrease in viscosity with increase in shear rate suggests pseudoplastic nature of the blends. This type of flow behavior arises from the randomly oriented and highly entangled nature of polymer chains, which on application of high shear get oriented and disentangled resulting in reduction in viscosity at higher shear stress [3]. The reduction in viscosity of the blends at higher shear stress is also due to shearing away of the dispersed particles in the blend. It can also be attributed to the decrease in particle size, as revealed in morphology analysis. The high viscosity at low shear rate provides the integrity of the extrudate during extrusion, and the low viscosity at high shear rate enables low injection pressure and less injection time. Ciesielska and Liu [38] made similar reports of decrease in viscosity with increase in shear rates in recycled expanded polystyrene/styrene-butadiene rubber (PS/SBR) blends.

Figure 2 shows the variations of melt viscosity for various blend compositions with weight percentage of PS at different shear rates. PB exhibits higher viscosity than PS. Variation of viscosity with blend composition is non-linear with negative deviation at regions of low PS content and positive deviation at regions of high PS content with respect to linear extrapolation between PS and PB extremes. At low shear rates, the high rubber blends have higher viscosities. It is observed that at all shear rates, as the weight percentage of PS increases, the viscosity of the blend decreases up to 30 wt.% of PS, followed by an increase at 50% PS and further addition of PS decreases the viscosity. It can be

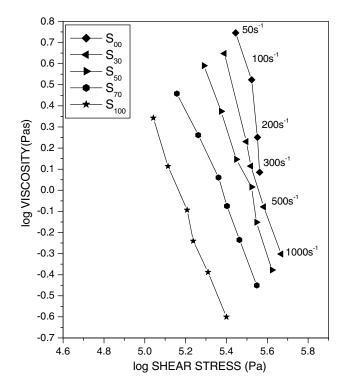


Fig. 1 Effect of shear stress on melt viscosity of PS/PB blends at 180 $^{\circ}\mathrm{C}$

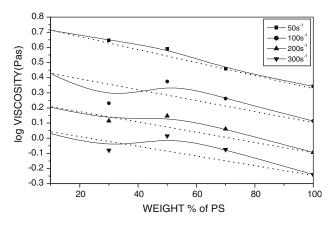


Fig. 2 Effect of blend composition on shear viscosity for PS/PB blends at 180 $^{\circ}\mathrm{C}$

seen that as the percentage of PS increases further, viscosity of the blend decreases at all shear rates. A sharp increase in viscosity at 50 wt. % PS can be attributed to the morphological changes, where the dispersed phase also become continuous with the blend (co-continuity).

Comparison with theoretical predictions

The experimental viscosity data of the blends has been correlated with various theoretical models. Utracki and Sammut [8] showed that a positive or negative deviation of measured viscosity from that calculated by the log additivity rule is an indication of strong or weak interactions between the phases of the blend.

$$\ln (\eta ap) \text{ blend} = \sum w_i \ln (\eta app)_i \tag{4}$$

where w_i is the weight fraction of the *i*th component of the blend. They indicated that the negative deviation shown by the immiscible blends is associated with heterogenous nature of the components, whereas a positive deviation indicates the miscibility of the blends resulting from the high solubility and homogenous nature of the components. The following models have been used to calculate the viscosity of the blend.

$$\eta_{\text{mix}} = \eta_1 \phi_1 + \eta_2 \phi_2 \text{ (model 1)}$$
(5)

where η_1 and η_2 are the viscosities of the components 1 and 2 and ϕ_1 and ϕ_2 are their volume fractions.

Hashin's [39] upper and lower limit models predict the viscosities by equations given below. This model is applicable to materials in which the components are connected parallel to one another so that the applied stress lengthens each component to the same extent. In the lowest- lower bound series model, the blend components are arranged in series (Reuss prediction) perpendicular to the direction of the applied force. The shear viscosity prediction is given by the inverse rule of mixtures as:

$$\eta_{\rm mix} = \eta_2 + \frac{\phi_1}{\frac{1}{\eta_1 - \eta_2} + \frac{\phi_2}{2\eta_2}} \,(\text{model2}) \tag{6}$$

$$\eta_{\rm mix} = \eta_1 + \frac{\phi_2}{\frac{1}{\eta_2 - \eta_1} + \frac{\phi_1}{2\eta_1}} \,(\text{model }3) \tag{7}$$

where η_1, η_2, ϕ_1 and ϕ_2 are the same as before.

Sood et al. [40] developed an altered free volume model to calculate the viscosity using equation,

$$\ln \eta_{\text{mix}} = \frac{\phi_1(\alpha - 1 - \gamma \phi_2) \ln \eta_1 + \alpha \phi_2(\alpha - 1 + \gamma \phi_1) \ln \eta_2}{\phi_1(\alpha - 1 - \gamma \phi_2) + \alpha \phi_2(\alpha - 1 + \gamma \phi_1)}$$
(model 4)
(8)

Figure 3 is a comparison between experimental viscosity data with model predictions for different blend compositions at a shear rate of 100 s^{-1} . It is evident from the figure that the viscosity of the blends showed a positive deviation from the additivity rule at low concentration of PS and a negative deviation at

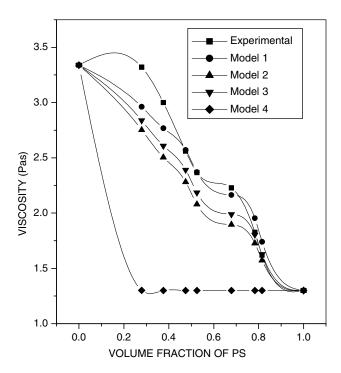


Fig. 3 Theoretical and experimental values of blend viscosity as a function of volume fraction of PS at 100 s^{-1}

high content of PS. The positive deviation indicates the miscibility of the blends resulting from the high solubility and homogenous nature of the components. A rather good qualitative agreement is observed with model 1 compared to any of the other models.

Effects of compatibilisation

Considerable advances have been made, from both theoretical and experimental point of view, in relating the presence of compatibilisers to structure development during flow. Compatibilisation increases the scale of dispersion and stabilises the blend morphology. A suitably selected compatibiliser locates at the blend interface, which leads to a reduction in interfacial tension and thereby permits the dispersion of the minor component in the matrix polymer and an enhancement in interfacial adhesion. Both SBS and SBR are non-reactive and have segments identical with each of the blend components.

Figure 4 shows the effect of modifying 70/30 PS/PB blend using SBS at 180 °C and at various shear rates. Compatibiliser incorporation increases the melt viscosity of the blend. But the increase is not regular. In all the cases viscosity decreases with shear stress indicating pseudoplastic behavior. The variation in viscosity is more pronounced at lower shear rates, than at higher shear rates, as is evidenced by the converging nature of flow curves.

Figure 5 shows the variation of viscosity as a function of SBS loading in 70/30 PS/PB blend at 180 °C and at 100 s⁻¹. Maximum viscosity is found for the blend compatibilised with 2.5 wt.% SBS. This particular composition at which viscosity is found to be maximum is said to be critical micelle concentration (CMC), above which there is a decrease. At this point the interface is saturated with the compatibiliser. The increase in viscosity has been attributed to the increased interaction between the PS and PB as a result of decreased interfacial tension and coalescence due to the introduction of the compatibiliser. The triblock copolymer can effectively localize at the interface of the binary blends and the arms of the copolymer can diffuse into the corresponding homopolymer phases. This has been confirmed by NMR studies made earlier on these blends [41]. Willis and Favis [42] and Varughese [43] have made similar reports of increase of viscosity upon addition of compatibilisers. The morphological observations, which will be discussed in the later part of the paper complements the rheological studies.

The effects of compatibilisation of 70/30 PS/PB blend using SBR at 180°C and at various shear rates is seen in Fig. 6. Flow curves of the compatibilised blends are basically similar to that of uncompatibilised blends. In all the cases viscosity decreases with shear rate indicating pseudoplastic behavior. The variation in viscosity is more pronounced at lower shear rates,

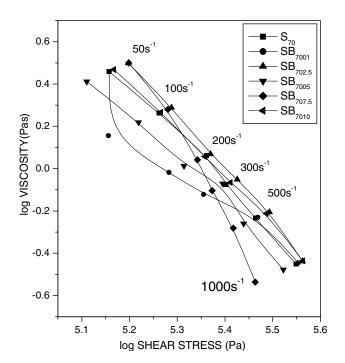


Fig. 4 Effect of shear stress on shear viscosity for blends compatibilised with SBS at 180 °C and at different shear rates

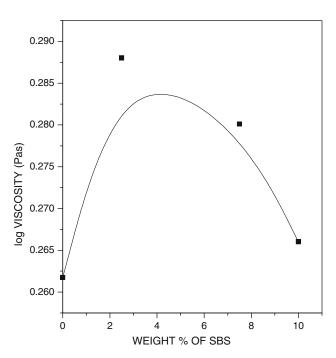


Fig. 5 Variation of shear viscosity with SBS loading for 70/30 PS/PB blends at 180 $^{\circ}{\rm C}$ and 100 ${\rm s}^{-1}$

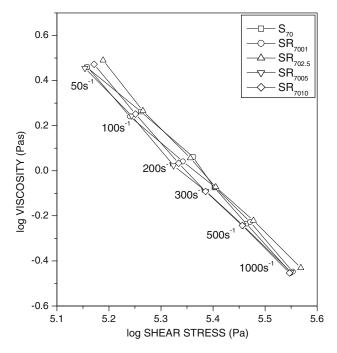


Fig. 6 Effect of shear stress on shear viscosity for SBR compatibilised blends at 180 °C at different shear rates

than at higher shear rates, as is evidenced by the converging nature of flow curves. Figure 7 shows the variation of viscosity as a function of SBR loading at 100 s^{-1} . Viscosity decreases by random copolymer addition, indicating that random copolymer is not effective in compatibilising the system. The random copolymer being asymmetric, cannot diffuse into the interface formed between the homopolymers effectively. Therefore, they can not compatibilise the polymer blends.

Flow behavior index

Table 2 lists the effect of blend ratio on the flow behavior index (n') values of the uncompatibilised

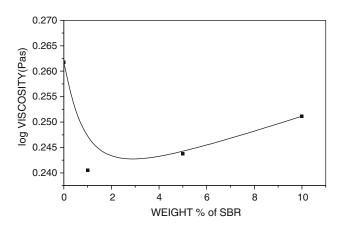


Fig. 7 Variation of shear viscosity with compatibiliser loading for SBR compatibilised blends at 180 $^{\circ}$ C and 100 s⁻¹

Table 2Flow behavior indexvaluesofuncompatibilisedandcompatibilisedblendsat180 °C

ex ed at	Blend	Flow behaviour index (n')
at	S ₀₀ S ₃₀ S ₅₀ S ₇₀ S ₁₀₀ SB ₇₀₀₁ SB _{702.5} SB ₇₀₁₀ SR ₇₀₀₁ SR ₇₀₀₁	0.15 0.23 0.25 0.27 0.27 0.34 0.28 0.32 0.31 0.31 0.29
	SR ₇₀₀₅ SR ₇₀₁₀	0.30 0.29

blends. Pseudoplastic materials are characterised by n' values less than one. The higher the value of n', the lower the pseudoplastic nature. All the mixes are non Newtonian. Among the homopolymers, PS is having relatively high n' values. Incorporation of PS to PB increases n' values in PS rich blends. Hence it is clear that less pseudoplastic behavior is observed for PS-rich blends. The flow behavior index values increase upon addition of both copolymers, indicating a slight decrease in the pseudoplastic nature of the compatibilised blends.

Extrudate morphology

Scanning electron micrographs (SEM) were taken to understand the effect of blend ratio, effects and efficiency of compatibilisation on the state of dispersion of rubber phase. The domain diameters were measured from several micrographs at random in each of the blend system. The various domain diameter values are calculated using Eqs. 1–3.

Effect of blend ratio and shear rate

The morphology that determines the physical properties of immiscible polymer blends can be controlled by rheological and thermodynamic factors [44]. It is observed that phase morphology of an immiscible blend changes with the composition of the blend, from a dispersion system to an inverted dispersion system through dual phase continuity [45]. The scanning electron micrographs of binary blends (S₃₀, S₅₀ and S₇₀) at 100 s⁻¹ is seen in Fig. 8(a)–(c), respectively demonstrate a two-phase morphology. In S₃₀, the minor phase of PS is dispersed as spherical inclusions in the continuous PB matrix (Fig. 8(a)). In S₅₀, both the phases are continuous as seen in Fig. 8(b). In S₇₀, the minor phase of PB is dispersed as droplets in the

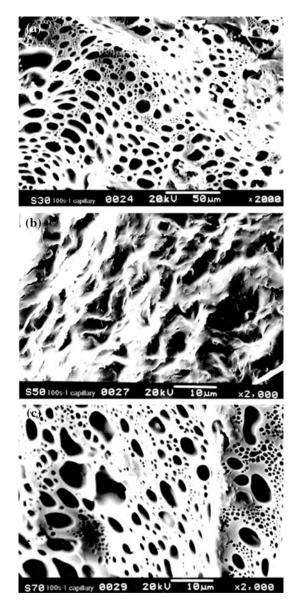


Fig. 8 SEM photographs of extrudates of PS/PB blends at 100 s^{-1} (a) 30/70 (b) 50/50 and (c) 70/30

continuous PS matrix as seen in Fig. 8(c). Average domain diameters of dispersed phase in uncompatibilised blends at 100 s^{-1} are seen in Table 3. In S₇₀, where PB is the dispersed phase shows larger domain

 Table 3
 Average domain diameters of dispersed phase in PS/PB

 blends at different shear rates

Blends	$\bar{D}_n(\mu m)$	$\bar{D}_w(\mu m)$	$\bar{D}_{\nu}(\mu m)$	PDI
$\begin{array}{c} {S_{30}} \left({100\;{s^{ - 1}}} \right)\\ {S_{70}} \left({50\;{s^{ - 1}}} \right)\\ {S_{70}} \left({100\;{s^{ - 1}}} \right)\\ {S_{70}} \left({300\;{s^{ - 1}}} \right)\\ {S_{70}} \left({1000\;{s^{ - 1}}} \right)\end{array}$	2.85	4.58	5.71	1.58
	3.76	6.67	10.78	1.62
	3.6	6.08	7.01	1.69
	3.1	4.03	4.98	1.30
	2.81	3.82	4.85	1.35

diameter than S_{30} , where PS forms the dispersed phase. This is due to shear-induced coalescence. It has been reported that the contact time needed for the drop coalescence increases when the matrix viscosity decreases, the drop diameter decreases, and the density difference between the matrix and the drop increases [46]. The SEM of PS/PB (70/30) at 180 °C and at 50, 300 and 1000 s⁻¹ shear rates are given in Fig. 9(a)–(c), respectively. The micrograph at 100 s⁻¹ is seen in Fig. 8(c). The extrudate morphology indicates that the particle size decreases considerably at higher shear rates. The dependence of shear rate on various domain diameters for S₇₀ blend is given in Table 3. The observed decrease in viscosity of the system with

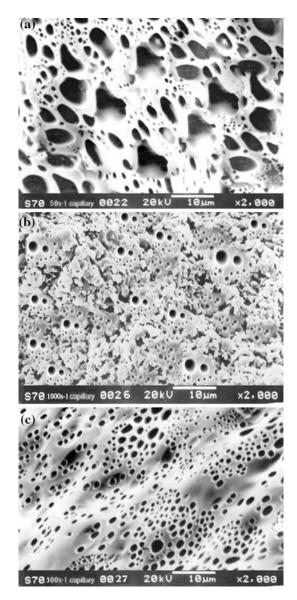


Fig. 9 Scanning electron micrographs of extrudates of PS/PB (70/30) blend at shear rates (a) 50 s⁻¹ (b) 300 s⁻¹ and (c) 1000 s⁻¹

increase in shear rates can be attributed to the decrease in particle size. The decrease in particle size is due to the increased deformation and consequent break down of the particles with increased shear rate.

Effect of compatibilisation

The extrudate cross-section morphology of PS/PB (70/ 30) blend compatibilised with SBS at a shear rate of 100 s^{-1} is seen in Fig. 10(a–c). The compatibilising action is evident from the changes associated with compatibiliser incorporation. Dispersed PB domains in PS matrix are visible from the micrographs. The various domain diameters seen in Table 3 show the better compatibilising action of the block copolymer,

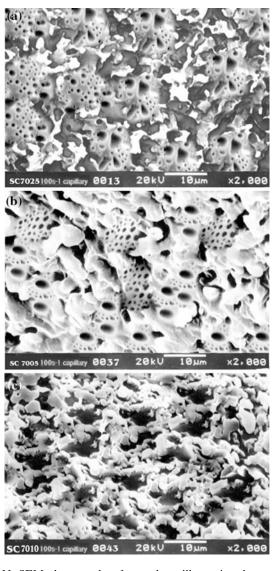
20kU SB 7010 100s-1 capillary 00 39 Fig. 10 SEM photographs of extrudates of PS/PB (70/30) blends illustrating the state of dispersion at different loadings of SBS at

 100 s^{-1} (**a**) 2.5 (**b**) 5 and (**c**) 10 %

which aids in finer dispersion of PB in the polymer matrix than that of a random copolymer. The morphological investigations show that the interface is saturated at 2.5 wt.% of the copolymer and there exist a critical concentration of the copolymer beyond which the copolymer forms micellar aggregation. We believe that as soon as the micelles are formed the copolymer already located at the interface will leave the interfacial region and will join the micellar aggregation. The coalescence tendency is highly suppressed in compatible blends, which is a critical requirement for a good compatibiliser.

The extrudate morphology depicting the effect of SBR addition on 70/30 PS/PB blend at a shear rate of 100 s^{-1} is seen in Fig. 11(a-c). In the absence of

Fig. 11 SEM photographs of extrudates illustrating the state of dispersion at different loadings of SBR at 100 s^{-1} (a) 2.5 (b) 5 and (c) 10 %



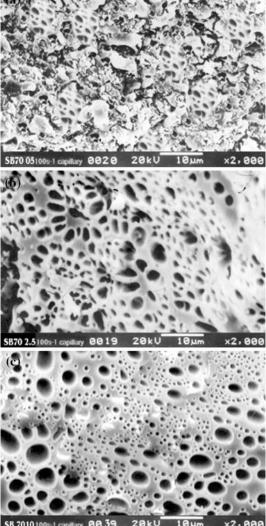


Table 4 Average domain diameters of dispersed phase in compatibilised PS/PB (70/30) blends at 100 s^{-1}

Blends	\bar{D}_n (µm)	\bar{D}_w (µm)	\bar{D}_{v} (μ m)	PDI
S ₇₀	3.6	6.08	7.01	1.69
SB702.5	1.23	1.70	4.72	1.38
SB7005	1.58	2.14	5.42	1.35
SB_{7010}	2.53	3.51	5.12	1.38
SR _{702.5}	2.36	3.87	5.78	1.64
SR ₇₀₀₅	2.61	4.39	10.34	1.68
SR ₇₀₁₀	2.70	4.48	9.17	1.66

compatibiliser, interface adhesion is very poor (Fig. 11(c)). This is evident from the detached domains of PB in PS matrix as seen in Fig. 11(c). The size of the dispersed phase is not appreciably affected by the addition of SBR. Also compatibilisation does not make the particle size uniform as seen for the different domain diameters in Table 4. This indicates that SBR does not considerably decrease the interfacial tension between PS/PB phases and aids in finer dispersion of PB in the polymer matrix. Thus the morphologies of binary and random copolymer compatibilised blends are almost similar, exhibiting elastomer domains dispersed in the PS matrix.

Conclusions

Melt rheological behaviour of PS/PB blends were investigated using a capillary rheometer. All the blends show a decrease in viscosity with increase of shear stress indicating pseudoplastic behavior. The viscosity of the system was found to increase with increase of rubber content. The variation in viscosity was correlated with the phase change of PS from a dispersed state to a continuous phase. The experimental viscosity values were compared with theoretical predictions. The experimental viscosity values were found to be close to series model beyond 50/50 PS/PB and did not fit well with any of the other models. The use of block copolymer, SBS as compatibiliser increases the melt viscosity of the blends. This increased viscosity has been attributed to the increased interaction between the PS and PB as a result of decreased interfacial tension and suppression of coalescence due to the introduction of the compatibiliser. This is associated with the localization of the triblock copolymer at the interphase. The arms of the copolymer diffuse into the corresponding homopolymer phases. The extrudate morphology was found to depend on shear rate, blend ratio and compatibilisation. The morphology of the blends showed a two-phase structure in which the minor phase was dispersed as domains in the major continuous matrix phase. A co-continuous morphology was observed at 50/50 PS/PB composition. The addition of the block copolymer led to finer dispersions of the particles of the minor component and a decrease in their size; this induced a significant change in the blend extrudate morphology. The important changes upon compatibilisation using block copolymer are fineness of the morphology and increase in blend viscosity. The random copolymer, SBR did not show much effect upon compatibilisation on account of its inability to localize at the interface. The conclusions derived from the rheological studies are in agreement with the microscopy observations and the earlier NMR studies.

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