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Phase morphological study on SEBS compatibilized PS/LDPE blends

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Abstract

The co-continuous phase morphology of polystyrene (PS)/low density polyethylene (LDPE) blends compatibilized with poly(styrene-*block*-ethylene/butylene-*block*-styrene) triblock copolymers (SEBS) with varying molecular weights has been investigated. The blend samples were prepared in a mini-twin screw extruder. The barrel length and diameter are 224 and 16 mm, respectively. The diameter of the capillary die is 1 mm. The concentration of the blends was 70/30 wt% of PS/LDPE while that of the SEBS used was 5 wt% of the blend. The mixing temperatures used were 180, 250, and 280°C, and a screw speed of 60 rpm. The morphology of the blends was investigated using an AFM technique. Average droplet diameters of the blend samples were determined using an OM technique. The co-continuous morphology has not been obtained in all the blends, although the mixing temperature used is as high as 280°C. The experimental results indicated that the model prediction of the co-continuous morphology proposed by Willemse and co-worker was not applicable to the blend systems studied. Only droplet-type dispersion was observed. This is considered to arise from the processing conditions and the mixing device used. The blend compatibilized with the high molecular weight SEBS had higher dispersed phase size than that of the blend compatibilized with the medium and low molecular weight SEBSs. This behaviour is likely to arise from coalescence during melt processing.

Keywords : Block copolymer, Co-continuous morphology, LDPE, PS, SEBS

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1. Introduction

An effective means to obtain new materials is blending of thermoplastic immiscible polymer. The properties of these blends are to a large extent determined by the morphology, i.e. the size, shape and distribution of the component [1]. Generally, the morphologies obtained in the immiscible polymer blends can be divided into three classes, i.e. dispersed, stratified and co-continuous morphologies. In this work, we are interested in the co-continuous morphology as this morphologies show a combination of the characteristics of both the blend component [2]. This morphology is usually a non-equilibrium structure that is generated during melt processing. Therefore, it is an unstable morphology and starts changing through filament breakup and retraction as soon as the melt blend comes out from the mixer [3]. It has been reported that this morphology could be obtained in polymer blends with low concentrations of the minor blend component provided appropriate processing conditions are applied [2]. The continuous minor phase is represented as an assembly of rod-like particles randomly oriented and at their maximum packing density inside the matrix (major component). To form the co-continuous morphology, at this packing density, all the rods have to touch each other and coalesce at their crossover points [2]. The maximum packing density of randomly oriented rods (ϕ_{\max}) depends on the aspect ratio L/B , where L is the length and B is the diameter of the particle. The empirical equation of the ϕ_{\max} for randomly oriented stiff rods is given as [4].

$$\frac{1}{\phi_{\max}} = 1.38 + 0.0376 \left(\frac{L}{B} \right)^{1.4} \quad (1)$$

According the model proposed by Willemse *et al.* [2], the existence and stability of the elongated structure in the blend are determined by the value of the capillary number (Ca) where in this case, the Ca is defined as.

$$Ca = \frac{\eta_m \dot{\gamma} B}{2\Gamma} \quad (2)$$

where η_m is the viscosity of the continuous phase, $\dot{\gamma}$ is the shear rate, and Γ is the interfacial tension. As the aspect ratio (L/B) increases, the Ca decreases. When a critical value ($Ca = 1$) is reached, the interfacial stress destabilizes the elongated particles and breakup occurs (dispersive mixing). This indicates that the minimum value of B , for which cylindrical particles are still stable, can be found from Eq. (2) by setting $Ca = 1$. In order to relate the Ca to the ϕ_{\max} of the elongated structure, ($Ca = 1$) together with the condition for the conservation of the volume of the extended particles.

$$\frac{B}{2R_o} = \left(\frac{2}{3} \right)^{\frac{1}{3}} \left(\frac{L}{B} \right)^{\frac{1}{3}} \quad (3)$$

are combined with Eq. (1). The final relation, which is the so-called *Willemse model* [2,5], is given as.

$$\frac{1}{\phi_{d,cc}} = 1.38 + 0.0213 \left(\frac{\eta_m \dot{\gamma}}{\Gamma} R_o \right)^{4.2} \quad (4)$$

where R_o is the radius of the spherical particle before deforming into a long cylindrical shape. Eq. (4) gives the lower limit of the range of the volume fraction of the minor phase ($\phi_{d,cc}$) where fully co-continuous morphologies are possible, for a specific polymer system (η_m and Γ) and for the specific blending conditions ($\dot{\gamma}$) [5].

In the presence of compatibilizers, which are normally block copolymers, it is possible to obtain immiscible polymer blends with co-continuous morphology and improved mechanical properties simultaneously [4]. Therefore, the formation of this morphology at low concentrations of one polymer in the other, in the presence of compatibilizer, can be of significant importance for the design of novel materials. Polystyrene (PS) is a glassy polymer having properties which are adequate for many applications. It is normally used in service below its glass transition temperatures, T_g . In general, the PS

is prone to brittle fracture. Because of such problem, low density polyethylene (LDPE) which is a rubbery polymer above its T_g , is incorporated into PS matrix phase. However, the PS and LDPE are highly immiscible homopolymers. Introducing small amount of a block copolymer into the PS/LDPE blend system would enhance a finer morphology and increase in the adhesion between PS and LDPE phases. This can lead to improvements in the mechanical behavior of the PS. Over the years, the poly(styrene-*block*-ethylene/butylene-*block*-styrene) triblock copolymers (SEBS) triblock copolymers have been intensively studied as compatibilizers for many immiscible polymer blends and commonly reported that the main function of the SEBS during processing lies in reducing the interfacial tension between two immiscible polymer phases resulting in a finer distribution of the minor phase of the blends studied [5-7].

The co-continuous morphology has been observed in blend of PS and high density polyethylene (HDPE) in the presence of SEBS (G1652) prepared in a conical twin-screw extruder. According to the work of Xu and Chan [8], this morphology was obtained in the PS/HDPE blend having 80/20 wt% when the SEBS (G1652) content of 4 wt% was introduced in to the blend as a compatibilizer. These compatibilized blends have shown to provide better improvement in both impact and tensile properties [8-9]. Further increase in the content of the SEBS (G1652), i.e. 8-10 wt% of the blend, led to a change of co-continuous to droplet type morphologies. These results demonstrated that the SEBS play the role of a compatibilizer. This morphology has been also observed in the PS/LDPE blends with 30 wt% of LDPE [10]. The SEBS (G1652) with the content of 10 wt% of the blend was employed as a compatibilizer. The blends were prepared in a conventional mixer under mild processing condition, i.e. mixing at 200°C with a rotor speed of 35 rpm for 10 min. However, as the

LDPE content of the blend was increased to 40 wt%, the co-continuous structure still existed but there were also many round particles dispersed in the PS matrix phase. With an increase of the LDPE content to 60 wt%, phase inversion appeared, i.e. the LDPE became the continuous phase. The compatibilized PS/LDPE blends with 30 - 40 wt% of the LDPE content gave the highest elongation and fracture strength. As mentioned before, the co-continuous morphologies are unstable with annealing. When immiscible polymer blends are in the liquid state at a given temperature, i.e. annealing temperature, not only change the dimensions of the phase domains, but also the range of volume fractions where the co-continuous morphologies are obtained [1].

It is, therefore, difficult to control these phase morphologies during further processing, e.g. compression and injection molding. Over the years, the capability of a block copolymer to stabilize the co-continuous morphology has been extensively studied. Harrats *et al.* [11] have reported that poly(styrene-*block*-hydrogenated butadiene) (P(S-*block*-B)) and poly(styrene-*block*-hydrogenated butadiene-*block*-styrene) (P(S-*block*-hB-*block*-S)) exhibited ability both enhancing and stabilizing the co-continuous morphology in the PS/LDPE with 20 wt% LDPE. However, at a given thermal annealing condition, the P(S-*block*-hB) diblock copolymer showed to be more efficient compatibilizing activity than the P(S-*block*-hB-*block*-S) triblock block, particularly at a low concentration of about 2 wt% of the blend. The superiority of the diblock copolymer over the triblock copolymer might be due to its ability to quantitatively locate at the interface of PS/LDPE.

The objectives of this work are to investigate whether the SEBS triblock copolymers can cause co-continuous morphology in the PS/LDPE blend and to examine if the model prediction proposed by Willemse *et al.* [2] is applicable to the compatibilized blend studied.

2. Experimental

The PS used was provided by BP Chemical Company. The LDPE is a commercial product of DSM. Table 1 shows molecular characteristics of the PS and LDPE used. All homopolymers were obtained in pellet form. As the molecular weight of the block polymer is shown to be a crucial factor governing the co-continuous morphology, three different molecular weight SEBSs were used. The SEBSs used are commercial products of Shell Development Company: Kraton G1650, G1651, and G1652. The number average molecular weight (M_n) values of the SEBS were obtained from the supplier and shown in Table 2.

Table 1 Molecular characteristic of PS and LDPE

Properties*	PS	LDPE
M_w	2.50×10^5	1.21×10^5
M_n	1.11×10^5	9.53×10^3
Polydispersity (M_w/M_n)	2.20	12.30

*Determined by GPC, RAPRA Technology Ltd. UK.

Table 2 Molecular characteristic of SEBSs

SEBS	Total M_n	Mid-Block (PEB)*, M_n	End-Block (PS), M_n	PS (%)
G1651	240,000	160,000	39,000	33
G1650	100,000	71,100	14,500	29
G1652	50,000	39,000	8,000	29

*PEB = Poly(ethylene-butylene)

The PS/LDPE blends were prepared in a mini-twin screw extruder (PRISM Co., UK). The barrel length and diameter are 224 and 16 mm, respectively. The diameter of the capillary die is 1 mm. The mixing temperatures used were 180, 250, and 280°C and a screw speed of 60 rpm. As aforementioned, the droplet type morphologies have been found to improve the impact properties while co-continuous morphologies provide better tensile

properties [2]. In a previous work [12], the relatively higher impact strengths were obtained from uncompatibilized PS/LDEP blends having 30 wt% of LDPE content prepared at a mixing temperature of 180°C and screw speed of 60 rpm. These blends exhibited the droplet type morphology. It was expected that better improvement in both impact and tensile properties would be achieved in the SEBS compatibilized PS/LDPE blend exhibiting the co-continuous morphology. The concentration of the SEBS employed was 5 wt% of the blend. Emulsifying curves of PS/LDPE blend compatibilized with SEBS triblock copolymer reported in the work of Kunyawut [13] indicating that this concentration was enough to reduce the interfacial tension between the blend components to its lowest value at a given temperature. Further increase in the amount of SEBS would result in undesired effect of micelle formation and an increase in viscosity of the matrix phase [14-15]. In this work, therefore, the concentration of the blend components used was 70/30 wt% of PS/LDPE and that of the SEBS triblock copolymers was only 5 wt% of the blends. Practically, in an extrusion procession, higher screw speed provides stronger shearing resulting in better blending. However, high shearing can result in cleavage molecular chains leading to decrease of the molecular weight of the blend components [10]. In order to minimize the number of variables and processing conditions, the screw speed used was kept at 60 rpm based on the impact properties observed in our previous work [12]. The mixing temperature was only varied as the interfacial of the blend components decreases significantly with temperature [1,3,7]. According to the so-called *Willemse model*, the interfacial tension of the blend components is among the most crucial parameters affecting the volume fraction of the minor phase as $\phi_{d,cc} \propto \eta_m \dot{\gamma} R_o / \Gamma$ where the co-continuous morphologies exist [5]. Note that, as the resident time in the extruder employed was of about 3-5 min, thermal degradation of the blend components at

relatively high mixing temperatures, i.e. 250°C and 280°C, was expected to minimize.

After extruding, the strands were cooled in air to room temperature. The extruded strands about 10 cm in length were immersed in liquid nitrogen for 5 min and then kept in plastic bags. The morphology of the blends was obtained using AFM (NanoScope IIIa MSP, Digital Instruments Co., California, USA). The PS can be identified as the matrix phase while the dispersed phase is LDPE using the tapping mode AFM. The LDPE droplets were identified by their lamellar structures using high-resolution images.

Average droplet diameters of the blend samples were determined using OM. Pellets of blend samples were extracted using warm THF in which the PS phase was dissolved while droplets of the LDPE were suspended in THF. A drop of this solution was placed on a glass slide and the solvent was allowed to evaporate at room temperature. The glass slide was stored in an oven at room temperature for several days before performing OM. This would allow the LDPE droplets to return to their original shape and size from swelling. An optical microscope (Optiphot-2, Nikon Co. Ltd., Japan) set for phase contrast was used to determine an average phase size and size distribution of the minor phase. The image analysis was performed using a commercial image analysis software. The blend morphology was characterized by its size distribution function $f(d)$ where d is the droplet diameter. In particular, use will be made of the number-average and volume-average diameters, d_n and d_v , defined as.

$$d_n = \frac{\sum_i f(d_i)d_i}{\sum_i f(d_i)} = \frac{\sum_i n_i d_i}{\sum_i n_i} \quad (5)$$

$$d_v = \frac{\sum_i f(d_i)V_i d_i}{\sum_i f(d_i)V_i} = \frac{\sum_i n_i d_i^3}{\sum_i n_i d_i^2} \quad (6)$$

where d_i is the diameter of a generic drop and n_i is the total number of the droplets of diameter d_i . In each blend, a number of order 800 droplets were analyzed. The measurements of particle size diameter were repeated three times on several samples and it was found that d_n and d_v were reproducible to $\pm 15\%$.

3. Results and discussion

A morphological investigation was first carried out on the blends prepared at 280°C based on the idea that the co-continuous morphology is likely to be found in the blends prepared under relatively high intensity of processing conditions, i.e. high mixing temperature and high screw speed. Since the SEBS triblock copolymers have relatively high viscosity, mixing under high temperature allows the block copolymer to segregate to the interfaces effectively leading to high degree of reduction of the interfacial tension [6]. Under given mixing conditions, the co-continuous morphology is likely to be obtained in a blend system with low interfacial tension [5]. The morphology of these blends obtained from AFM micrographs shown in Figure 1 revealed that no co-continuous morphology was obtained. Although the contrast of the micrographs of the blends was low due to technical problems, only droplet-type dispersion was observed. As seen in Figure 2, the LDPE droplets were identified by their lamellar structures while this is not the case for the PS which is a glassy polymer [16].

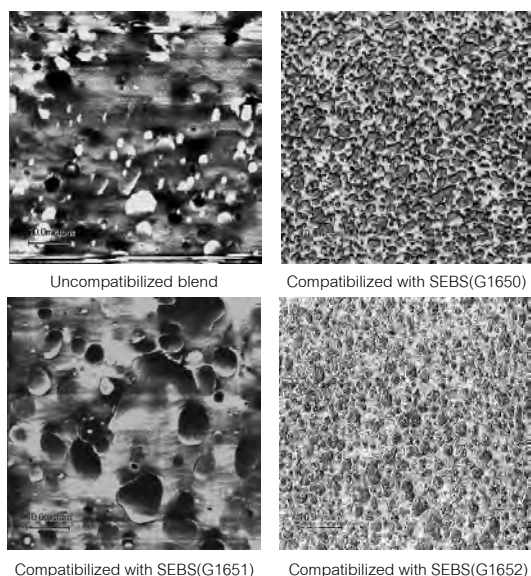


Figure 1 AFM micrographs for PS/LDPE (70/30 wt%) uncompatibilized and compatibilized with 5 wt% of SEBS: prepared in the PRISM mini-twin screw extruder at 280°C/60 rpm

The effect of mixing temperature on coalescence of the minor phase was also investigated. It can be seen in Figure 3a and 3b that both d_v and d_n of the SEBS(G1651) compatibilized blend increase with an increase in mixing temperature whereas these two average diameters for the rest of the blends are not highly dependent on the mixing temperature. The lower limit volume fraction in which the co-continuous morphology will occur for the PS/LDPE blends studied may be estimated [2]. The lower limit volume fraction predicted decreases significantly with an increase in shear rate. When the interfacial tension of the blend component is reduced, through addition of a block copolymer, at the same applied shear rate the critical volume drops dramatically. If the shear rate is considered to be determined by the screw speed used, at a screw speed of 60 rpm and in the presence of SEBS, the blends with 30 wt% of LDPE would exhibit co-continuous morphology according to the model prediction proposed by Willemse *et al.*

[2]. However, this morphology is not observed. An explanation may be that during processing the degree of coalescence of the threads of the dispersed phase is not high enough to form co-continuous structures owing to the high degree of dispersive mixing during processing.

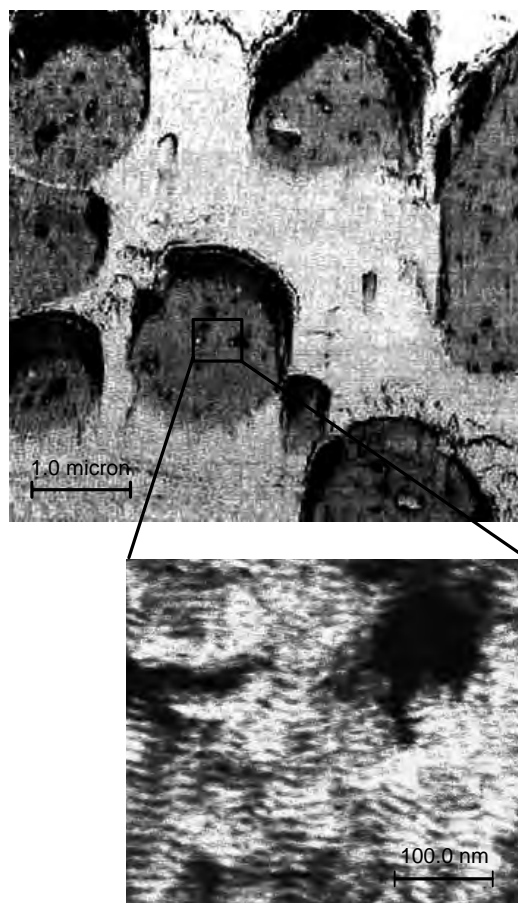


Figure 2 Lamellar structure of the dispersed LDPE particles for the PS/LDPE (70/30 wt%) blend compatibilized with 5 wt% of the SEBS (G1650): prepared in the PRISM mini-twin screw extruder at 280°C/60 rpm

According to the thermodynamic approach for the “wet” and “dry” brushes formed by a block copolymer at the interface [16], it is expected that phase inversion and/or co-continuous morphology should be obtained in the SEBS (G1651) compatibilized blend because the M_n of the PEB

mid-blocks is higher than the M_w of the LDPE. Note that the polydispersity of polymer is estimated by the ratio of M_w to M_n . If the minimum value of polydispersity of the PEB mid-blocks is assumed to be unity, the M_w of the PEB mid-blocks is still higher than that of the LDPE (see Table 2). Although the morphology obtained in the SEBS (G1651) compatibilized blends exhibited relatively large dispersed phase size as can be seen in Figure 3, this morphology is still far away from being phase inverted or co-continuous morphology as compared with the co-continuous morphology which is shown in Figure 4. The results obtained in this study are in general agreement with the suggestion of Hermes and Higgins [16] that phase inversion and/or co-continuous morphology are not always governed by equilibrium thermodynamics.

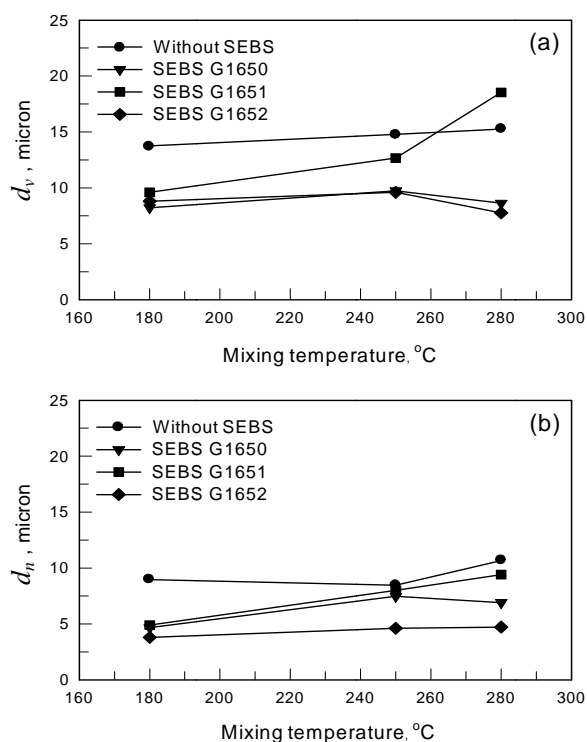


Figure 3 Volume and number average diameters of PS/LDPE (70/30 wt%) blends in the absence and presence of SEBS (5 wt% of the total blend): prepared at screw speed of 60 rpm

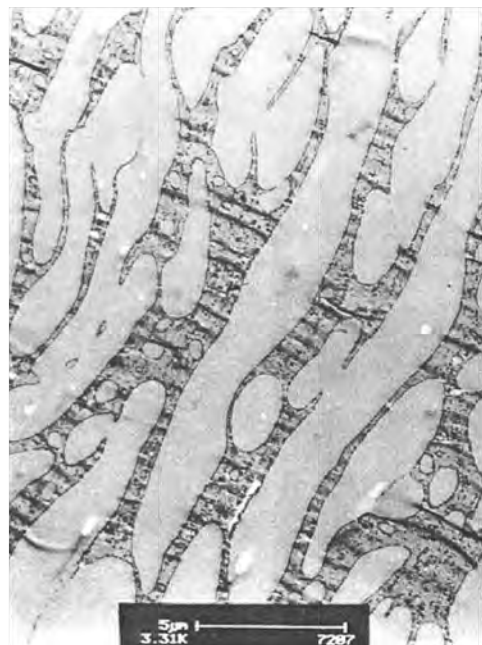


Figure 4 TEM micrograph of PS/LLDPE (75/25 wt%) blend compatibilized with PS-b-hPB: 5 wt% of the blend), Total $M_w = 5.30 \times 10^4$, M_w (hPB blocks) = 3.20×10^4 : prepared at mixing temperature of 300°C and screw speed of 250 rpm (from Hermes and Higgins [16])

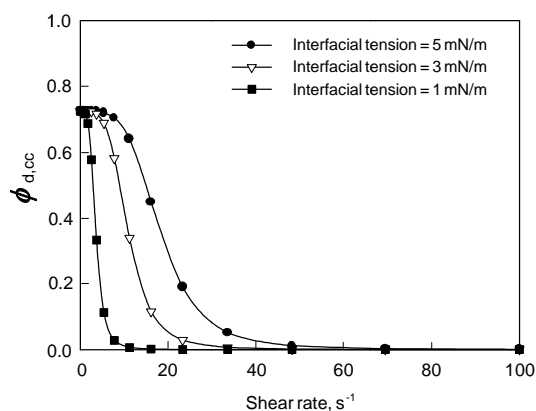


Figure 5 Prediction of PS/LDPE lower limit concentration for co-continuous morphology as a function of shear rate

The lower limit volume fraction in which the co-continuous morphology will occur for the PS/LDPE blends studied may be estimated using Eq. (4). At a temperature of 280°C the viscosity of the PS (η_m) is about 740 Pa.s [6]. Assuming the radius of

the dispersed particles is about 1 μm , by varying shear rate and the interfacial tension, the values of $\phi_{d,cc}$ are obtained. Plots of $\phi_{d,cc}$ as a function of shear rate are shown in Figure 5. Since the viscosity of the PS was found to be a rheological function varying with shear rate [9], in order to evaluate the influence of interfacial tension, an average value of two viscosities measured at angular frequency (ω) = 0.1 rad/s (1,080 Pa.s) and at ω = 100 rad/s (400 Pa.s) reported by Kuniyawut [6] was employed. Note that, according to Cox-Merz rule, the steady shear viscosity corresponds to the complex viscosity if the shear rate ($\dot{\gamma}$) in s^{-1} is plotted on the same scale as the angular frequency (ω) in rad/s. This can be stated as $|\eta^*(\omega)| = \eta(\dot{\gamma})|_{\dot{\gamma}=\omega}$ [17]. This empirical rule seems to hold up quite well for most polymers. Using this rule, it is possible to determine viscosity data up to 500 s^{-1} by applying an oscillatory motion to dynamic rheometers [18].

As seen in Figure 5, the lower limit volume fraction predicted by Eq. (4) decreases significantly with an increase in shear rate. When the interfacial tension of the blend component is reduced, through addition of a block copolymer, at the same applied shear rate the critical volume drops dramatically. At a temperature of 180°C, the interfacial tension of the PS/LDPE is about 5.8 mN/m [19] while that of the PS/LDPE in the presence of SEBS (G1652) is reduced to about 1.1 mN/m [20]. This clearly indicates that the SEBS triblock copolymers have the effect of reducing the interfacial tension between the PS/LDPE homopolymers. According to the work of Burkhardt *et al.* [21] and Wu [22], the number of revolutions per unit time of the screw of a co-rotating twin screw extruder can be used as an effective shear rate by assuming that the shear rate in the channel is due to drag flow. This approach is

somewhat crude but convenient. The shear rate of an extruder estimated from the screw speed has been widely used by many researchers [23,26]. Therefore, if the shear rate is considered to be determined by the screw speed used, at a screw speed of 60 rpm and in the presence of SEBS, the blends with 30 wt% of LDPE would exhibit co-continuous morphology according to the model prediction proposed by Willemse *et al.* [2]. However, this morphology was not observed. An explanation may be that during processing the degree of coalescence of the threads of the dispersed phase is not high enough to form co-continuous structures owing to the high degree of dispersive mixing during processing. Note that the PRISM mini-extruder used in this study has three sections of kneading blocks in the screw set. This screw configuration used in the PRISM mini-extruder is designed to provide a high degree of dispersive mixing. The SEBS triblock copolymers are also considered to play a role in preventing droplet coalescence through the so-called *staple structure* [24] formed at the interface where the PS blocks are miscible with the PS phase and the PEB blocks miscible with the LDPE phases [14]. The quantitative segregation of the SEBS to the PS/LDPE interface was indicated by reducing of the interfacial tension resulting in the smaller dispersed phase dimension of LDPE. However, with larger dispersed phase size obtained as seen in Figure 3, the high molecular weight SEBS (G1651) was shown to be less effective ability to suppress droplet coalescence than the low molecular weight ones. This is because its critical micelle concentration is very low. It can be expected that the long chain SEBS (G1651) gets struck in micelles under the mixing flow field [25].

Regarding the work of Hermes and Higgins [16], the phase inversion and co-continuous morphology observed in the PS/LLDPE blend compatibilized by the PS-*b*-hPB diblock copolymers may occur because of poor efficiency of dispersive mixing of the DSM mini-twin screw extruder used since there are no kneading blocks in the screws. Therefore, it can be expected that during melt mixing at the high temperature of $300 \pm 2^\circ\text{C}$ and high screw speed of 250 ± 5 rpm molten PS and LLDPE would experience a high degree of distributive mixing ($Ca \gg Ca_{crit}$). Together with the extremely short mixing time (less than 1 min), incomplete mixing might result. In reality, the formation of co-continuous morphologies more likely proceeds by a sheet forming mechanism involving breakup and local coalescence of sheets of the minor component [26-27]. The co-continuous morphology is a non-equilibrium state in quiescent conditions and will change form because of the interfacial tension driven mechanism, including Rayleigh distortions [28-29], retraction and end-pinching [30-31]. Therefore, in the presence of a block copolymer, the reshaping mechanism would be effectively prevented in some ways, as mentioned before, in order for the co-continuous morphology to exist.

4. Conclusion

The co-continuous morphology of the PS/LLDPE blends (70/30 wt%) compatibilized with 5 wt% of the SEBS triblock copolymers having various molecular weights has been studied. This morphology has not been obtained in all the blends prepared, although the mixing temperature used is as high as 280°C . The model prediction of the co-continuous morphology proposed by Willemse *et al.* was not applicable to the blends studied. Only droplet-type

dispersion was observed. This is considered to arise from the processing conditions and the mixing device used. The blend compatibilized with the high molecular weight SEBS had higher dispersed phase size than that of the blend compatibilized with the medium and low molecular weight SEBSs. This behaviour is likely to arise from coalescence during melt processing.

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