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Rheological Behavior of Immiscible PS/LDPE Blends: A Comparative Study of the Palierne Emulsion Model

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Abstract

The rheological behavior of polystyrene (PS)/low density polyethylene (LDPE) blends have been investigated. Blends with 10, 20 and 30 wt% LDPE were prepared using a co-rotating twin-screw extruder at a melt mixing temperature of 180 °C. SEM micrographs revealed that all blends exhibited droplet-type dispersion morphology. Rheological measurements were performed at the same temperature as the mixing temperature. The complex viscosity of the blends laid between those of the PS and the LDPE and decreased with increasing LDPE concentration. The plots of tanδ of blends with 10 and 20 wt% LDPE suggested that the elasticity of these blends was comparable to that of the pure PS. By contrast, the elasticity behavior of the blend with 30 wt% LDPE was shown to be similar to that of LDPE. Palierne model predictions were in very good agreement with the experimental data for the blends having 10 and 20 wt% LDPE. For the 30 wt% LDPE blend, agreement with the prediction and the experimental data was not as good. Within the frequency range of the rheological measurements, the second plateau at low frequency which results from an increase of the elasticity was not observed in any of the blends studied. This is expected to be due to the long relaxation times of the relatively large LDPE droplets of these blends.

Keywords: LDPE, Palierne model, PS, rheological properties, viscoelastic behavior

Introduction

The rheological behavior of polymer blends is generally very complex. In the case of immiscible blends, the rheological properties depend in a complex manner on composition and the properties of the blend components. Several models have been proposed in order to describe the linear viscoelastic behavior of an immiscible polymer blend under linear oscillatory flow. The most successful model, proposed by Palierne [1], has been extensively examined and found to be applicable to the viscoelastic properties in many immiscible polymer blend systems [2-8]. However, failures of the model have also been found in concentrated suspensions of non-deformable particles. These were attributed to particle-particle contacts, which are neglected in the model [9-11]. The Palierne model treats the blend as an emulsion formed from two viscoelastic incompressible materials, where the droplets forming the dispersed phase are spherical in equilibrium [1,2]. It takes into account the deformation and relaxation of the shape of the dispersed phase and considers only the hydrodynamic interaction between dispersed particles. The main assumption is that the droplet deformation remains small. According to the Palierne model, a linear viscoelastic constitutive equation for the emulsion is expressed as a function of the linear viscoelastic properties of the blend components, the size distribution of the dispersed phase, and the interfacial tension. Variation of the interfacial area in the deformed particles gives rise to an elastic contribution proportional to the ratio of interfacial tension over radius for the particles. The simplified expression for the complex modulus of the emulsion is given as [2];

$$G_b^*(\omega) = G_M^*(\omega)\frac{1+3\sum_i \phi_i H_i(\omega)}{1-2\sum_i \phi_i H_i(\omega)} \tag{1}$$

and the $H_i(\omega)$ is given as;

$$H_i(\omega) = \frac{\left(4\frac{\Gamma}{R_i}\right)\left[2G_M^*(\omega)+5G_I^*(\omega)\right]+\left[G_I^*(\omega)-G_M^*(\omega)\right]\left[16G_M^*(\omega)+19G_I^*(\omega)\right]}{\left(40\frac{\Gamma}{R_i}\right)\left[G_M^*(\omega)+G_I^*(\omega)\right]+\left[2G_M^*(\omega)+3G_I^*(\omega)\right]\left[6G_M^*(\omega)+19G_I^*(\omega)\right]} \tag{2}$$

where G_I^*, G_M^* and G_b^* are the complex moduli of the dispersed phase, matrix phase and emulsion at frequency ω, respectively. Γ is the interfacial tension which is assumed to be independent of the local shear and ϕ_i is the volume fraction of dispersed particles of radii R_i. The distribution of particle sizes can be replaced with a volume-average particle size if the polydispersity of the dispersed phase, d_v/d_n, is not higher than 2.3, where d_v and d_n are the number-average and volume-average diameters, respectively [1,2]. If the values of interfacial tension and volume-average radius of the dispersed particles are known, the complex modulus of the blend can be calculated directly from the complex moduli of the dispersed and matrix phases which are normally obtained from rheological measurement. Expressions for storage and loss moduli (G' and G'', respectively) deduced from complex modulus are given elsewhere [12,13]. In many multiphase polymer blends, a plateau of G' at low frequency has been observed [9,14]. According to the Palierne model, the plateau will exist and can be determined experimentally if the longest relaxation time of the emulsion (λ_D) of the system (the relaxation time of the shape of a deformed droplet), the plateau relaxation time (λ_p) and the relaxation time of the matrix phase (λ_m) are not too close together. By assuming that the droplet has a spherical shape, λ_D and the plateau storage modulus (G_p) may be estimated using the following equations;

$$\lambda_D = \left(\frac{R\eta_m}{4\Gamma}\right)\frac{(19K+16)(2K+3-2\phi[K-1])}{10(K+1)-2\phi(5K+2)} \tag{3}$$

$$G_p = \frac{20\Gamma\phi}{R}\left(\frac{1}{[2K+3-2\phi(K-1)]^2}\right) \tag{4}$$

where $K = \eta_d/\eta_m$, η_d and η_m are the viscosity of the dispersed and matrix phases, respectively [15].

PS is a glassy polymer with properties which are adequate for many applications. However, it is normally used in service below its glass transition temperatures, T_g, where it is prone to brittle fracture. Incorporation of LDPE which is a rubbery polymer above its T_g into the PS matrix phase leads to improvements in the mechanical behavior of the PS [11]. From the processing point of view, one of the most fundamental questions is how to determine or predict the rheological properties of a given polymer blend system. Good understanding of the rheological properties of the PS/LDPE blends would help in smooth operations and processing. The objectives of this study are to investigate the rheological properties of PS/LDPE blends and to examine if the Palierne emulsion model is applicable to the blends studied.

Materials and methods

The PS was provided by BP Chemical Company (M_w = 250,000, polydispersity = 2.2). The LDPE (Stamylan LD2100 TN00) is a commercial product of DSM (M_w = 121,000, polydispersity = 12.3). The molecular weights of the homopolymers were determined using GPC technique performed by RAPRA Tech., UK. All homopolymers were obtained in pellet form.

The blends were prepared using a co-rotating twin-screw extruder (PRISM, UK). The barrel length and diameter are 224 mm and 16 mm respectively. The diameter of the capillary die is 1 mm. The concentrations of the blends used were 90/10, 80/20 and 70/30 wt% of PS/LDPE. The blend components were dry mixed before feeding into the hopper of the extruder. In order to minimize the effect of processing conditions, at the desired screw speed (60 rpm), the temperature of the barrel wall inside the extruder (the feed and mixing zone and at the die) was maintained constant at 180±5 °C using 3 automatic temperature controllers. After extruding, the strands were cooled in air to room temperature. The cooling time was about 10 min. Extruded strands about 10 cm in length were immersed in liquid nitrogen for 5 min. They were, then, stored in a dry place until required for morphological investigation.

The morphology of the blends was obtained from their fracture surfaces using a SEM model JSM-5300 (JEOL, Japan). The two phases are distinct for each composition at which the LDPE droplets were dispersed in the PS. The LDPE phase was identified by polarized light microscopy of a thin section of a strand of the blend [16]. White spot areas observed are the LDPE due to its semicrystalline property while the large dark area is the PS. Average droplet diameters of the blend samples were determined using optical microscopy (OM). Pellets of blend samples were extracted using warm tetrahydrofuran (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. In order to allow the LDPE droplets to return to their original shape and size from swelling, the glass slide was stored in an oven at room temperature for several days before performing OM. An optical microscope model Optiphot-2 (Nikon, Japan) set for phase contrast was used to determine the average phase size and size distribution of the minor phase. The blend morphology was characterized by its size distribution function $f(d)$, where d is the droplet diameter. The number-average, d_n, and volume-average diameters, d_v, are defined as;

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

$$d_v = \frac{\sum_i f(d_i)V_i d_i}{\sum_i f(d_i)V_i} = \frac{\sum_i n_i d_i^4}{\sum_i n_i d_i^3} \tag{6}$$

where d_i is the diameter of a generic drop, V_i is the volume fraction and n_i is the total number of the droplets of diameter d_i. In each blend, a number of order 1,000 droplets were analyzed. The measurements of particle size diameter were repeated three times on several samples.

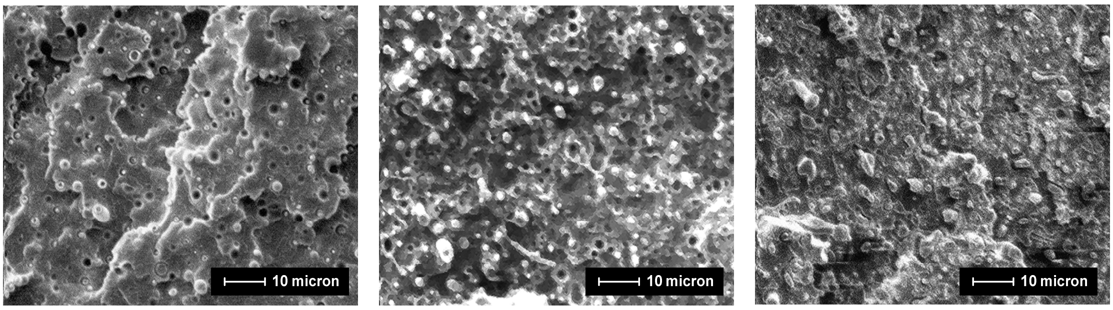
The rheological measurements were performed on a controlled strain rheometer using a Parr Physica model UDS200 (Physica Messtechnik, Germany) with a cone-plate geometry (25 mm diameter and 2° cone angle). For each polymer and blend, test specimens were prepared from compression molded plaques. The molded plaques of 2 mm thickness were prepared using the same conditions as those prepared for impact tests (BS EN ISO 2818: 1994). Discs of 20 mm diameter were cut from the plaques. Since the gap between the cone and the plate of the rheometer is relatively small, the discs of 20 mm diameter were able to fill the gap space with only a small amount of the melt sample exposed to the air when the level of the cone was moved downwards to the set point. Therefore, the distortion of the free surface causing the serious flow irregularity problem sometimes called edge fracture was minimized [17]. Frequency sweep measurements (0.01 - 100 rad/s) were performed at a constant temperature of 180 °C. The strain was maintained below 30 % to stay within the linear viscoelastic range which was tested by strain sweeps. Inspection of the samples after testing indicated that the amount of degradation was minimal and it was restricted to the edges of the sample not in contact with the cone and plate. The measurement time (including sample loading) was about 30 min. Therefore, the change of phase morphology of the blends caused by droplet coalescence occurring under quiescent (annealing) conditions was investigated. For the investigation under quiescent conditions, granules of the blends investigated were annealed in the rheometer chamber at 180±2 °C under dry nitrogen atmosphere. The annealing times used were 5, 15 and 30 min. Upon removal from the chamber, the annealed granules were immediately quenched in liquid nitrogen for 10 min in order to freeze-in the existing morphology. They were stored at room temperature in a dry place until required for morphological investigation.

Results and discussion

SEM micrographs of cryogenic fracture surfaces of the blends (**Figure 1)** clearly show that these blends having 10, 20 and 30 wt% of LDPE exhibit only the droplet-type dispersion morphology after extrusion. Hemispherical embeddings and hollows representing removed particles are clearly visible on the fracture surface. The frequency-distribution histograms for the blends prepared illustrated in **Figure 2** indicate that although the size distributions of these blends are different, their particle polydispersity values (d_v / d_n) are similar. The polydispersity of the dispersed phase of all the blends prepared is lower than 2.3. Hence, in Eq. (2), particle polydispersity can be used instead of the particle size distribution. **Figure 3** summarizes the effect of annealing time on droplet coalescence of the blends. An increase in d_v and d_n is observed with annealing time. The significant increase in the value of d_v and d_n is, however, found after 5 min of annealing in most of the blends studied. The increase in the dispersed particle size during a short time of annealing has been reported by Guo *et al.* [18]. After 15 min of annealing, no further significant change in the particle diameters is observed. The particle polydispersity values of all the blends studied are still lower than 2.3 (**Table 1**). As seen in **Figure 3**, the blend with 10 %wt LDPE has a higher rate of coalescence than those with 20 and 30 wt% of LDPE. According to the so-called *drainage model*, the small droplets have much more probability to coalesce than the large ones [19]. This is because the time required for film drainage from a pair of the small droplets would be shorter than that of the large ones leading to a higher coalescence. On the other hand, without external forces applied, the larger particles forming a large fraction in the blend are unlikely to move easily toward one another. The droplet coalescence results observed in this work are in general agreement with the work of several researchers [3,9,20].

Table 1 Polydispersity % PS/LDPE and annealing times.

PS/LDPE (wt%)	Annealing times (min)			
	0	5	15	30
90/10	1.58	1.69	1.51	1.43
80/20	1.53	1.56	1.48	1.55
70/30	1.49	1.55	1.52	1.50



PS/LDPE (90/10 wt%) PS/LDPE (80/20 wt%) PS/LDPE (70/30 wt%)

Figure 1 SEM micrographs of PS/LDPE blends with different LDPE concentrations.

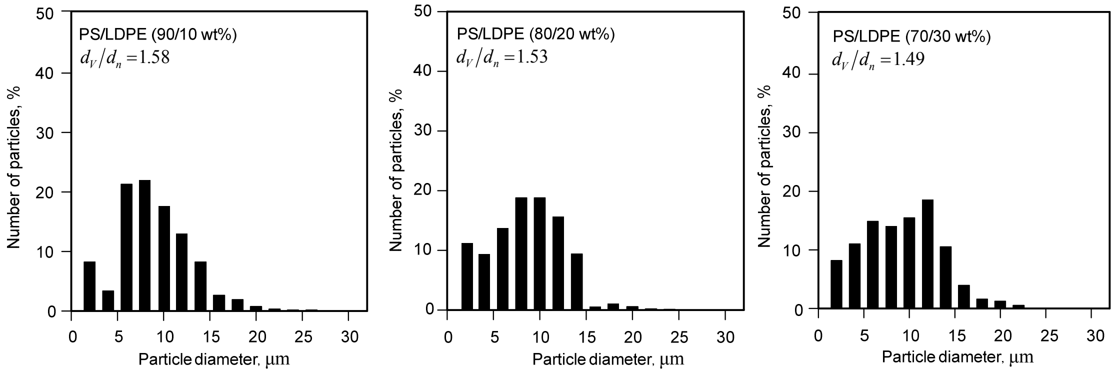


Figure 2 Particle frequency-distribution histograms for PS/LDPE blends from optical microscopy.

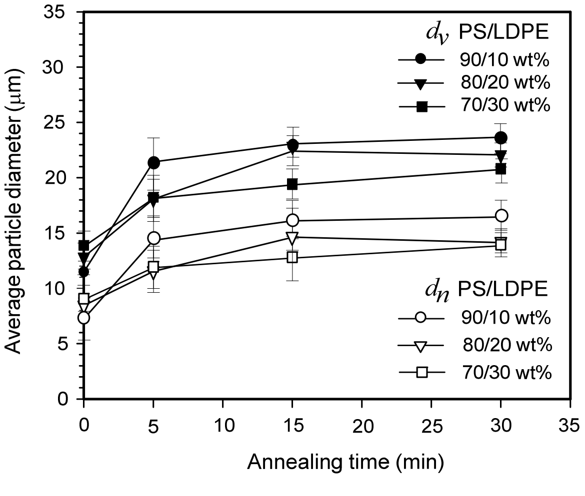


Figure 3 Volume-average and number-average diameters as a function of annealing time of the PS/LDPE blends.

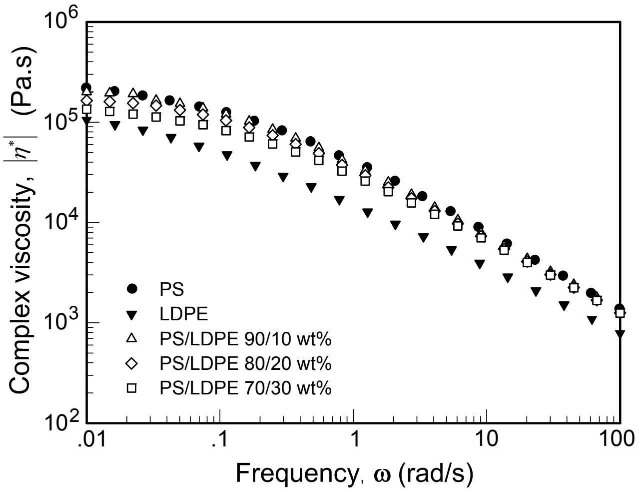


Figure 4 Complex viscosity as a function of frequency for PS/LDPE blends and the blend components (PS and LDPE) measured at 180 °C.

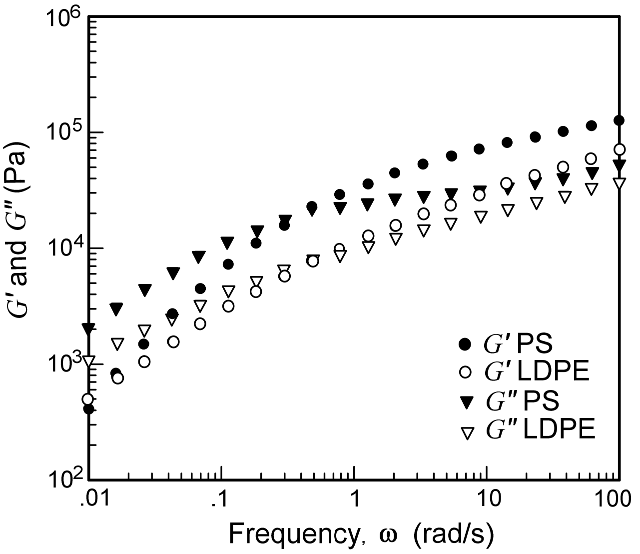


Figure 5 Frequency dependence of storage G' and loss G'' moduli for the PS and LDPE samples measured at 180 °C.

The complex viscosity versus frequency for the PS and LDPE samples at 180 °C, i.e. the mixing temperature, is illustrated in **Figure 4**. According to this figure, the shear thinning behavior of the LDPE is more significant than that of the PS over the ranges of frequency studied. **Figure 5** shows the frequency dependence of the storage (G') and loss (G'') moduli for both PS and LDPE measured at the same temperature. It can be seen that the values of both G' and G'' of the LDPE are lower than that of the PS over the whole frequency range. Therefore, it can be expected that G' and G'' of the blend will decrease with increasing concentration of LDPE. **Figure 6** shows the frequency dependence of tanδ of the PS/LDPE blends and the blend components. The tanδ, shown in this figure, is the ratio of the loss modulus to the storage modulus which is used in characterization of viscoelastic materials in the molten state [17]. If tanδ < 1, the elastic contribution predominates whereas, if tanδ > 1, the viscous contribution predominates. According to **Figure 6**, the frequency at which the LDPE samples exhibit elastic predominance is lower than that of the PS samples at the same measuring temperature. This indicates that the LDPE sample has a higher frequency sensitivity of the elastic contribution than the PS sample. Note that, at high frequency (1 - 100 rad/s), the elastic property of the PS tends to make a greater contribution than that of the LDPE.

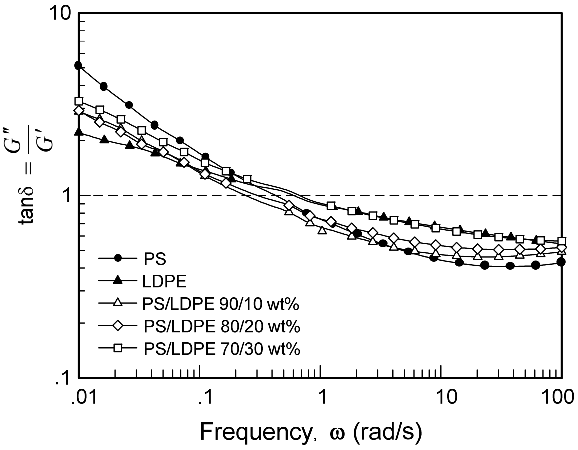


Figure 6 Frequency dependence of tanδ for the PS/LDPE blends and the blend components (PS and LDPE) measured at 180 °C.

It is widely known that the rheological properties of immiscible polymer blends are strongly influenced by their morphology. During the melt blending process, the less viscous component will form droplets and be dispersed into the more viscous one [21]. In this study, the PS/LDPE blends are observed to exhibit a droplet-type dispersion morphology as expected, and in good agreement with the work of Van Oene [22]. In an immiscible polymer blend, in which the disperse phase has a lower viscosity than the matrix phase, the viscosity of the blend is normally lower than that of the major phase. Moreover, it has been reported that viscosity decreases with growth of the dispersed phase particle size (under identical measuring conditions) [23]. With an increase in the size of the dispersed phase, the contribution due to deformation of the dispersed phase of the low viscosity component increases. This lowers the overall level of the viscosity of the blend melt. The frequency dependence of the complex viscosity of the PS/LDPE blends measured at 180 °C is expressed in **Figure 4**. It can be clearly seen that the complex viscosity of the blends lies between those of the PS and the LDPE. As expected, the complex viscosity decreases with an increase in LDPE concentration. As clearly seen in **Figure 6**, the plots of tanδ of the blends having 10 and 20 wt% LDPE lie close to the PS at higher frequency indicating that the elasticity of these blends behave like that of the PS. By contrast, at high frequency, the plot of tanδ of the blend having 30 wt% LDPE lies nearly over the plot of tanδ of the LDPE. This indicates that the elasticity behavior of the blend having 30 wt% LDPE is shown to be similar to that of the LDPE. At low frequency (0.01 - 0.1 rad/s), the plots of tanδ of all the blends lie between those of the blend components (PS and LDPE) indicating that there is an increase of elasticity attributed to a relaxation process of the dispersed droplets of the LDPE when slightly sheared [24]. As the plots of tanδ of the blends having 10 and 20 wt%

lie close to that of the LDPE, it can be expected that the increase of elasticity obtained in these blends would be higher than that of the blend having 30 wt% LDPE. Moreover, the increase of elasticity of these blends can be confirmed by an increase of their storage modulus at low frequency. **Figure 7** shows the storage G' and loss G'' moduli of the PS/LDPE blends with different compositions and of the individual blend components at 180 °C. It can be seen that at low frequency the storage modulus of the blends with 10 and 20 wt% LDPE are slightly greater than those of the PS and the LDPE. However, the storage modulus of the blend having 30 wt% LDPE and the PS are the same indicating that the increase of the elasticity of the blend having 30 wt% is rather small and cannot infer from the storage modulus. According to emulsion models, the increase of the elasticity can be employed to evaluate the interfacial tension between the phases of the blend components [2,5,25-34]. However, as will be shown later, this is not the case for relatively small increase of the elasticity obtained for the PS/LDPE blends studied.

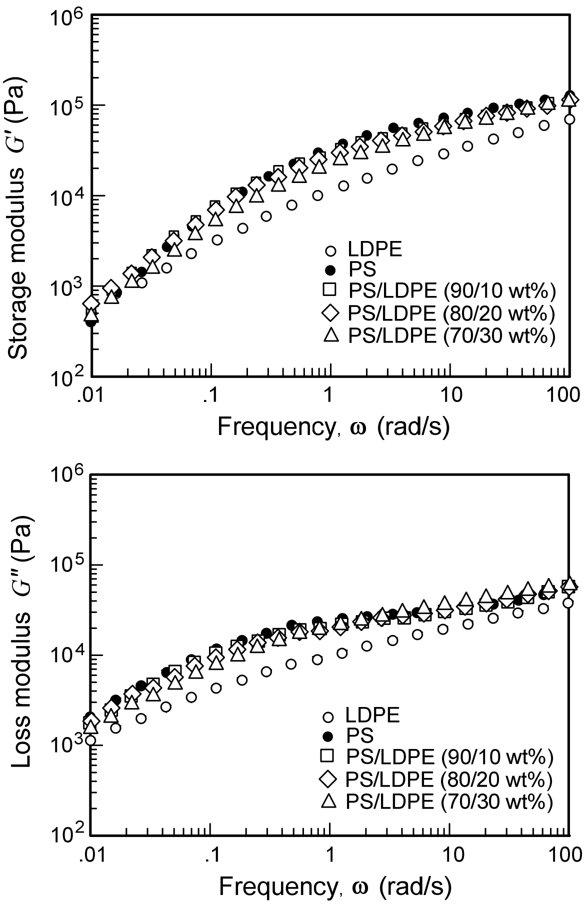


Figure 7 Frequency dependence of storage G' and loss G'' moduli of the PS/LDPE blends measured at 180 °C.

The morphology of the test samples changed due to coalescence during the rheological measurement as the first measuring data point is obtained about 20 min after the measurement (**Figure 3**). To take droplet coalescence into account, it is appropriate to use the rheological data measured at 180 °C to compare with the Palierne model predictions. As seen in **Table 1**, the droplet polydispersities of all the blends after annealing are well below 2.3 indicating that these blends have narrow particle size distribution. Therefore, the volume-average particle size and volume fraction of LDPE in the PS/LDPE blends are employed for estimation of G^* with Eqs. (1) and (2). In this work, the interfacial tension between the PS and LDPE used is obtained from the literature which is about 5.8 mN/m at 180 °C [34].

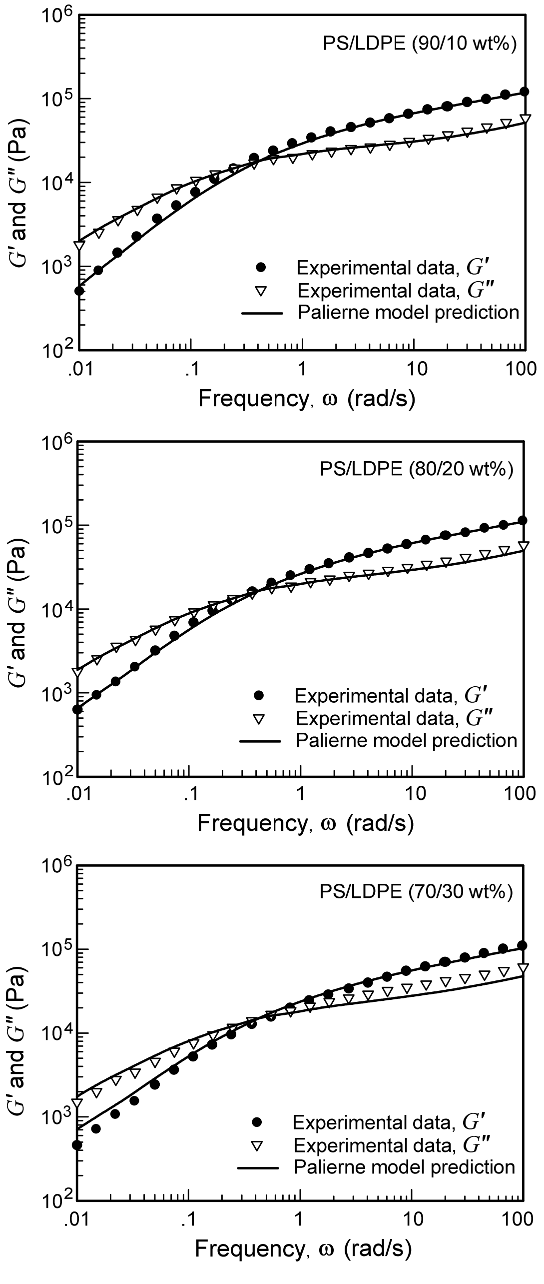


Figure 8 Comparison between experimental data and the Palierne model predictions for the PS/LDPE blends at 180 °C.

Model predictions for G' and G'' deduced from G^* and their comparison with the experimental data for the PS/LDPE blends are illustrated in **Figure 8**. Very good agreement between the model predictions and the experimental data is obtained for the PS/LDPE blends with 10 and 20 wt% LDPE. For the PS/LDPE blend with 30 wt% LDPE, the fit is less satisfactory and a deviation of the model predictions from the experimental data in the low frequency region for G' and in the low and high frequency regions for G'' is seen. This is attributed to the higher contribution of LDPE which the model cannot take into account effectively [9-11]. This is in agreement with the expected limitations of the model [35,36]. Droplet coalescence which is shown to be more pronounced in this immiscible blend system (see **Figure 3**) and resulted in a change of morphology would also be associated with a less satisfactory agreement between the model predictions and experimental data.

Table 2 Droplet relaxation time (λ_D) and plateau modulus (G_P) of PS/LDPE blends estimated using the Palierne model and corresponding relaxation frequency (ω_D) [2].

PS/LDPE (wt%)	**Estimated parameters**		
	λ_D (s)	G_P (Pa)	$\omega_D \approx 1/\lambda_D$ (s^{-1})
90/10	1170.0	30.4	8.6×10^{-4}
80/20	1215.0	59.6	8.2×10^{-4}
70/30	1250.0	94.2	8.0×10^{-4}

As seen in **Figures 8**, well defined secondary plateau at low frequency are not observed in the storage modulus G' curves of the model predictions and experimental data of any of the PS/LDPE blends although the increase of elasticity in these blends has been obtained (see also **Figure 6**). The plateau can be detected if the dispersed droplets are deformable and the times required for the process of shape recovery (relaxation) are not relatively long. In other words, the plateau at low frequency does not exist if there is no deformation of droplets. This is likely to occur in an immiscible polymer blend having very high or very low interfacial tension [15,37]. As the interfacial tension between the PS and LDPE is relatively high (about 5.8 mN/m) [34,38], the LDPE droplets would behave like rigid particles under small amplitude oscillatory shear. Thus, in this work, if the deformation of the droplets did take place under low frequency of oscillation, it is expected that the droplet relaxation time of the dispersed particles (λ_D) would be relatively long and could not be detected within the frequency range of the rheological measurements [36]. The values of λ_D and the secondary plateau modulus (G_P) for the blend samples estimated using Eqs. (3) and (4) are shown in **Table 2**. As seen in this table, the estimated droplet relaxation time and the secondary plateau modulus are well outside the experimentally accessible ranges. This long relaxation time is likely to be a result of the relatively large dispersed droplets (**Figure 3**). According to the model, the effects of interfacial tension are seen only in the terminal zone (low frequency) of both phases, where the blend components (PS and LDPE) behave as Newtonian fluids. Varying values of the interfacial tension is found not to provide any better fit for these blends as the terminal zone of the blend component is far below the frequency range used. Therefore, it is not possible to infer the interfacial tension between the blend components (PS and LDPE) using small amplitude rheological measurements. From a practical point of view, extending the measuring frequency downward to 0.0001 rad/s is not possible as this would take more than 3 - 4 h for the first data point to be obtained and the test sample would already be largely degraded. Moreover, the transducer of the rheometer may not be able to cope with the extremely low value of G' predicted at relatively low frequency. The results shown in this work are in general agreement with the work of Bousmina [15,37] and Shi *et al*. [36] and indicate that a slight increase in elasticity would be obtained from immiscible blends with high interfacial tension and relatively large dispersed droplet size.

Conclusions

The linear viscoelastic properties of the immiscible PS/LDPE blends have been investigated. The complex viscosity, storage and loss moduli, and tanδ of the blends lay between the dispersed phase (LDPE) and the matrix phase (PS). The plots of tanδ as a function of frequency clearly reveal that at high frequency (1 - 100 rad/s) the elastic properties of these blends are dominated by the contribution of the PS and this reduced with an increase of the concentration of LDPE. By contrast, at low frequency (0.01 - 0.1 rad/s) the increase of the elasticity of all the blends is attributed to the shape relaxation process of the LDPE droplets. The enhancement of elasticity of the PS/LDPE blends with low concentration of dispersed phase compared to the blend components is also confirmed by a higher storage modulus $G'(\omega)$ curve at low frequency range. The Palierne model provided very good agreement with the experimental data for the blends with 10 and 20 wt% LDPE while slightly worse agreement with the experimental data is found for the case of the blend having 30 wt% LDPE. This is explained by the increased contribution of LDPE phase and the instability of the morphology arising from coalescence. Within the frequency range of 0.01 - 100 rad/s, the second plateau at low frequency is not observed in the storage modulus $G'(\omega)$ curve of these blends. It is expected to be due to the long relaxation times of the relatively large LDPE droplets of these blends.

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