Phenoxy/Hytrel Blends. II. Dynamic and Tensile Properties of Unreacted Miscible Blends

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ABSTRACT: The dynamic and tensile properties of Brabender mixed, compressionmolded miscible, and unreacted polyhydroxy ether of bisphenol A (Ph)/Hytrel blends were studied. Blending mainly produced a decrease in the specific volume, and in the strength of the β transition of Ph. The β transition strength decrease was attributed to both specific interactions and specific volume decrease. The measured modulus of elasticity, yield stress, and ductility of the blends were discussed as a result of the combined effect of the β transition strength decrease, crystallinity, and free volume content changes, and the position of the T_g s of the blends with respect to the testing temperature. © 1999 John Wiley & Sons, Inc. J Appl Polym Sci 72: 85–93, 1999

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INTRODUCTION

In the first article,¹ devoted to polyhydroxy ether of bisphenol A, Phenoxy, (Ph)/Hytrel (Hy) blends, their phase behavior and chemical nature were studied. Interchange reactions took place during processing in the melt state at long mixing times, but were negligible at times shorter than 15 min. The reactions produced first branched copolymers and then crosslinked products. They were the fastest and the most general when at the intermediate compositions (30-70% Ph).

Only one T_g was seen by DSC, whatever the composition. The miscibility of the blends was seen by DMTA, and was mainly due to specific interactions. This was to the detriment of copolymers produced by interchange reactions that may miscibilize the blend. The development of interchange reactions did not significantly affect the

phase behavior of the blends because of their inherently miscible nature.

The mechanical properties of these blends before reaction are described in this article. Besides the structure of the components and the crystallinity level, there are two solid-state characteristics that usually mainly influence the mechanical properties of isotropic miscible polymers blends. These are the possible blending-induced decrease in free volume content, and the possible partial secondary transition strength decrease. The free volume content has overall influence on most of the properties important from a practical point of view,² while the secondary transition is closely related with the toughness and ductile behavior of high T_g amorphous polymers such as Ph or polycarbonate (PC).^{3,4}

The mechanical properties of Hy are of interest, seemingly because of the two more or less cocontinuous phases made up of a network of crystalline poly(butylene terephthalate) (PBT) domains in a cocontinuous polyether terephthalate matrix.⁵ Those of Ph blends have been the subject of much attention. However, those of fully miscible blends have not been widely studied. This is despite the

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fact that compatibility is assured, and that a wide range of properties, not only mechanical, may be obtained by changing composition. This is probably due to the relatively few miscible blends of commercial polymers. Among the miscible blends whose mechanical properties have been studied, besides the fairly well-known polystyrene/poly(phenylene oxide)⁶⁻⁸ and PC/Kodar,⁹ others such as Ph/ PBT^{10,11} fairly similar to that of this article, poly-(ether ether ketone)/poly(ether imide),¹² Ph/poly(methyl methacrylate)^{13,14} have also been the subject of attention. In most of the cases the mechanical properties were related to the specific volume,^{6-8,14} morphology,¹³ crystallinity level,¹⁵ and in some cases, to the strength of secondary transitions.⁸

The miscible nature of the Ph/Hy blends, and the undesired foreseeable change¹¹ of properties towards more rigid but clearly less ductile and tough materials when reaction occurs, make the study of the unreacted blends more attractive than that of reacted ones. In a tentative initial study on reacted blends, larger reaction times did not provide any change in the specific volume, and only slightly decreased the β transition strength in the intermediate compositions. Finally, an increase in the modulus of elasticity and in the yield stress and a ductility decrease similar to those of other Ph-based blends^{11,16,17} were observed.

For these reasons, the dynamic and tensile properties of miscible and unreacted Ph/Hy blends have been studied here. The dynamic properties have been discussed with regard to blending induced secondary transition strength and free volume changes, and these parameters and the crystallinity level and T_g values have been related to the most relevant mechanical properties obtained from the tensile test.

EXPERIMENTAL

The polymers used and the mixing and processing methods were described in Part I. To avoid a possible influence of different storage times on the nature and properties of the blends, they were maintained at room temperature for 2 weeks. The calorimetric scans were performed in a Perkin-Elmer DSC-2 calorimeter at a scan rate of 20°C/ min. The different transitions and enthalpies were determined in the usual way. The dynamicmechanical analysis was performed in a DMTA (Polymer Laboratories) in single cantilever mode at 1 Hz. The scans were carried out at a constant heating rate of 4° C/min from -150° C until the sample became too soft to be tested.

The density of the blends was measured at 23 \pm 2°C in a density gradient column. The specific volume of the amorphous phase was determined taking into account the crystallinity measured by DSC, the density of crystalline PBT 1.396 g/cm³,¹⁸ and that the volume was additive. The self-densification of Hy was not taken into account due to its small value.¹⁹

Tensile specimens (ASTM D-638 Type IV) were punched out from the compression molded sheets by a pneumatic die. Tensile tests were performed at 23 \pm 2°C and at a crosshead speed of 0.5 mm/min for modulus measurements and then at 50 mm/min up to fracture. The modulus of elasticity (*E*), nominal yield stress (σ_y) and ductility (ε_b) were measured. A minimum of 10 specimens was tested for each reported value. When the standard deviations of the mechanical properties do not appear, it is because they were smaller than the symbol size.

RESULTS AND DISCUSSION

Dynamic Mechanical

In Figure 1, the E' values of the pure polymers and those of the blends against temperature are shown. As can be seen, two E' decreases were present in the E' plot of Ph: the fist one corresponds to the T_g at roughly 100°C, and the broad second, which extends from -100 to -25°C approximately, and corresponds to the secondary transition.¹ In the case of Hy, only the decrease that corresponds to the T_g was seen from approximately -60 to 10° C, with a slope smaller than that of Ph. No E' decrease corresponding to the β transition of Hy was seen near -100°C.¹ In the case of the blends, both the E' decrease, that corresponds to the T_g and that of the β transition of Ph, were seen. The width and localization of the E^\prime decrease that corresponded to the $T_g {\rm s}$ were intermediate between those of the pure polymers.

The tan δ against temperature plots of the blends and of the pure components in the secondary transitions range are shown in Figure 2. The T_g behavior of the blends was discussed in part I of this work. The Ph shows two secondary transitions: a β transition at -68° C and a γ transition at -110° C that appears as a shoulder of the β transition. This γ transition is not taken into account due to its weak nature. As can be seen, the



Figure 1 DMTA $\log E'$ plot of the blends and of the pure polymers against temperature. To aid clarity, the curves are shifted on the vertical axis.

 β transition of Hy only appears in the 10/90 blend due to its weak nature. That of Ph, however, appears in all the blends with the exception of that same composition. It is known that mixing, particularly when combined with miscibility,^{20–22} may lead to a change in the strength of a secondary transition and in the temperature at which it takes place.

As can be seen in Figure 2, both the β transition temperature (T_{β}) and the β transition strength change as a result of blending. A decrease in the T_{β} produces a larger range below room temperature, at which the toughening effect of the transition is seen. With respect to the change of the transition strength, it may clearly modify the mechanical response of polymers. This is mainly true in the case of high T_g polymers, such as Ph with strong low temperature secondary transitions. This is because their ductile and tough behavior, which is due to the segmental movements characteristic of the secondary transitions, might develop towards a brittle and weak behavior if these secondary transitions were to weaken or disappear. This would not be relevant in the case of Hy, due to its below room temperature T_g , but would be in the case of Ph. Thus, the modifications of the β transition of Ph are more important than those of Hy. For these reasons, the possibility both of a T_{β} change and of a transition suppression must be determined and quantified.

The T_{β} s of Ph and Hy are plotted in Figure 3 against blend composition. As can be seen, the T_{β} of Ph moves to at lower temperatures when the blend becomes richer in Hy. The T_{β} of Hy increases with the Ph presence in the only composition at which it is detected. However, given the error of the T_{β} measurement, which was $\pm 2^{\circ}$ C in three different scans, and the presence of only two values, little information is available about its variation with the composition of the blend. The slip of the T_{β} of Ph towards lower temperature is not detrimental for properties, provided that its strength is maintained. This is because the lower



Figure 2 DMTA tan δ plot of the blends and of the pure polymers against temperature in the secondary transition range. To aid clarity, the curves are shifted on the vertical axis.



Figure 3 β Transition temperatures of Ph (\bigcirc) and Hy (\bigcirc) against composition.

the T_{β} , the larger will be the range below room temperature at which it is active. This variation of the T_{β} of Ph is in the same sense and similar in importance to that observed in Ph/polycaprolactone,²³ and is intermediate between that observed in acetylated Ph due to simple dilution²⁴ and those observed both in Ph/4'-[(2-hydroxy-3-Phenoxypropil)oxy] acetalinide²⁵ and in Ph/poly(ethylene oxide).²⁶

With respect to the strength of the β transitions, there are several ways to measure it. The most evident way is to measure the area under the peak. However, sometimes the height of the peak,⁴ and also the area under the peak but of tan δ vs. $1/T^{19,22,23,27}$ not vs. *T* are used. In this work all three methods were used and compared. The strength of the β transitions was first measured as in former works^{19,22,23,27} by

$$R \sim \int_{\text{peak}} \tan \delta d\left(rac{1}{T}
ight)$$
 (1)

where the baseline is taken to be the straight line linking the borders of the peak. This way of measuring relaxation strength appears to be misleading when asymmetric peaks and significant T_{β} shifts are produced. However, when the areas under the tan $\delta - T$ and tan $\delta - 1/T$ peaks were measured and plotted against composition, despite a shift in T_{β} of 12°C, the two plots were almost equal in shape. When the transition strength was measured by means of the height of the peak with respect to the same baseline, the values were slightly different. For this reason, only the peak height and the peak areas in the tan $\delta - 1/T$ plot were used as a measure of the relaxation strength, and are plotted in Figure 4 against blend composition. The values of the relaxation strength are relative to the strength of the secondary transition of Ph. The strength of the secondary transition of Hy is also plotted as a reference.

As can be seen in Figure 4, the decrease in strength of the β relaxation of Ph with respect to additivity is important in all the blend compositions, with the exception of the 30/70 blend in which it is additive. The lack of partial relaxation strength suppression in the 30/70 blend is surprising, compared with the other compositions. This is because the relaxation is clearly stronger than that of the 50/50 blends, despite its composition. One possibility is that the slight β transition of Hy could be overlapped with that of Ph and, despite its weak strength, could modify the observed β transition of Ph. If this were true, the transition would have had to be wider. This widening of the β transition of Ph in the 30/70 composition, rather clearly seen in the left side of the transition in Figure 2, did not take place in the 50/50 blend, and explains the β transition behavior of the 30/70 blend.

The overall β transition strength [measured as in eq. (1)] decrease is important (mean relative value of 0.28 with respect to the additive rule in the Ph-rich and intermediate compositions) and should influence such mechanical properties of the blends as ductility or impact strength. This partial β relaxation suppression effect has been



Figure 4 Strength of the secondary transition of Ph (\bigcirc, \bigoplus) and Hy (\square, \blacksquare) in the blends relative to that of the pure Ph against composition of the blends. Black symbols (\bigcirc, \blacksquare) strength measured as in eq. (1). Empty symbols (\bigcirc, \square) strength measured by the height of the maximum of the peak to the baseline.

Table I T_{g} , Melting, and Crystallization
Enthalpies and Crystallinities from the First
DSC Scan of Hy in Ph/Hy Blends Melt Mixed for
15 min

Composition of the Blend	T_{g} (°C)	ΔH_m (cal/g)	ΔH_c (cal/g)	Crystallinity (%)
100/0	85	_	_	
90/10	67			_
70/30	56	1.90	1.88	_
50/50	35	4.09	0.32	22
30/70	3	4.91	0.33	19
10/90		6.88		22
0/100	-52^{a}	9.03	_	27

^a From ref. 34.

observed in other Ph-based miscible blends involving hydrogen-bonding interactions between the components. 21,23

This suppression may be the result of the interactions in the local motions of the hydroxy ether groups of Ph that have been seen to exist^{1,23} but it could also be due to a blending-induced decrease in free volume.^{22,28-31} Both parameters are often related because a free volume decrease may appear as a consequence of specific interactions, but might not be related when miscibility is not due to specific interactions. Moreover, it is known that the free volume content of the amorphous phase is an important parameter when the mechanical properties of polymers are discussed. This is because deformation becomes easier in large free volume surroundings. Thus, both to relate the specific volume with the mechanical properties, and to elucidate at what extent the decrease in free volume is effective in the β transition strength decrease, the specific volume of the blends was measured against composition, and is discussed in the following section.

Specific Volume Measurements

The crystalline content of the blends influences the specific volume and the mechanical properties of the blends. For this reason, the crystalline content of Hy in the blends, and the melting (ΔH_m) and crystallization (ΔH_c) enthalpies were measured by DSC and are collected in Table I. Crystallinity was calculated taking a value of 32 cal/ g^{32} for the ΔH_m of the pure PBT. As can be seen, the Hy crystallinity of all the crystalline blends before the scan is rather similar. These results indicate that Hy, Hy-rich blends, and blends of

intermediate composition, almost fully crystallized in the cooling before the scan. Only the 70/30 blend crystallized in the calorimeter. This was probably helped by the ability of the Hy-rich and 50/50 blends, which have their calorimetric T_g s below and slightly above room temperature, respectively, to continue crystallizing at room temperature after molding.

When the specific volumes of the whole blends were plotted against blend composition, densification of the blends was seen at all compositions, mainly in crystalline blends. However, the usual plot of specific volume against composition is not suitable in the case of semicrystalline blends when the free volume of the amorphous phase is discussed. This is because the crystalline content of the blends usually changes with composition, and this change has a direct and concomitant influence on density. For this reason, the specific volume of the amorphous phase of the blends was calculated as stated in the experimental part, and is plotted against composition in Figure 5. The error bars correspond to the experimental error of the DSC determinations of the crystallinity, that was estimated to be $\pm 2\%$. When they do not appear, they are smaller than the symbols.

As can be seen, densification of the amorphous phase takes place at all compositions. On preliminary inspection of Figure 5, it seems that a single continuous concave curve could be drawn. However, taking into account the accuracy of the values, there are two kinds of specific values in the plot; those corresponding to Ph-rich blends, with $T_{\rm g}$ s clearly above room temperature, and those



Figure 5 Specific volume of the amorphous phase of the blends against composition. The mean typical deviation of the plotted values was ± 0.001 cm³ g⁻¹. The specific volume of Hy if it were in the glassy state is collected as an empty circle.



Figure 6 Schematic calculation of the specific volume of Hy in the glassy state.

corresponding to Hy-rich blends, with T_g below room temperature¹ (see Table I). Thus, in this plot (and in the plots of the mechanical properties), besides the change of specific volume, the change of T_{σ} from below to above testing temperature is also present throughout the composition. This leads to an additional specific volume change because the free volume dependence on temperature is different below and above T_g . This additional change is concomitant with that induced by blending. As a consequence, when the specific volume behavior of the glassy Ph-rich blends is studied, the deviations from additivity of specific volume cannot be properly discussed by taking as a reference the experimental values of Hy in the rubber state. The specific volume of Hy at room temperature if it followed the change proper to the glassy state should be more suitable. It may be estimated from the experimental value, and the difference in change of specific volume with temperature in polymers³³ between the glassy state $(2.5 \times 10^{-4} \text{ cm}^3 \text{ g}^{-1} \text{ K}^{-1})$ and the rubber state $(5 \times 10^{-4} \text{ cm}^3 \text{ g}^{-1} \text{ K}^{-1})$ as in Figure 6. Using the calorimetric T_g of Hy of -52°C ,³⁴ there is a temperature difference with room temperature of 75°C, that gives rise to a value of the specific volume of Hy at room temperature if it were in the glassy state of approximately $0.863 \text{ cm}^3 \text{ g}^{-1}$. This calculation appears accurate enough to assure a negative deviation of the specific volume of Ph-rich blends. However, a similar estimation in the case of the Hy-rich blends and the correspondent discussion of the plot in the Hy-rich side are not made because the accuracy of the calculation is estimated to be not good enough.

As can be seen in Figure 5, in the Ph-rich side the decrease in specific volume agrees with the β transition strength decrease, but the overall behavior of the specific volume of the amorphous phase in Figure 5 is difficult to correspond with that of the β transition suppression of Figure 4. This is because the abrupt curvature change in Figure 5 when Hy content increases in the glassy 50/50 composition does not appear in Figure 4, although its position may be slightly affected by its proximity to T_g . Moreover, there is no negative volume of mixing in the 50/50 composition, but a clear β transition strength decrease takes place. Thus, it appears that there is not a full correspondence between specific volume decrease and β transition strength suppression. This points to both the specific interactions and probably also specific volume decrease as being responsible for the observed β transition strength decrease.

Tensile Properties

The moduli of elasticity of the blends against composition are shown in Figure 7. As can be seen, the moduli of the Ph-rich blends are only slightly smaller than that of Ph. The moduli of Hy-rich blends are similar to those of Hy, despite the Ph presence, and that of the 50/50 blend is intermediate between those of the two components. As in the case of the specific volume, modulus values of blends with T_{σ} s above and below the testing temperature cannot be discussed together. This is because in semicrystalline polymers also, the temperature dependence of modulus is different below and above T_g , and the modulus changes through the T_g . These effects are superimposed on that of the blending-induced change of modulus with composition, and gives rise to modulus



Figure 7 Modulus of elasticity of the blends against composition. The modulus of Hy if it were in the glassy state is represented by an empty circle.

values in the Hy-rich side lower than those that would be found if all the blends were in the glassy state.

This leads to the fact that when deviation of the modulus from additivity in glassy Ph-rich blends is discussed with respect to structural parameters, the appropriate reference in this case, too, is not the experimental modulus of Hy, but the modulus of its amorphous phase if its change with temperature would have taken place in the glassy state. This change of the modulus of elasticity is not experimentally available. However, that of the storage modulus in a dynamic test may be estimated in a similar way as in the case of the specific volume of Figure 6, thus, extrapolating the straight line of its plot against temperature in the glassy state to room temperature. Supposing that the change in modulus of elasticity with temperature in these rubbery materials is similar to that of E' in a dynamic mechanical test, a value of the modulus of elasticity in the glass state of 1330 MPa was calculated. This value is plotted in Figure 5, and will be used as a reference to discuss the moduli of the glassy Ph-rich blends. Its value is different to that of PC/Hy blends²² due to the different E' and processing conditions. The calculation of the modulus of pure Hy appears accurate enough to assure a positive deviation of the modulus in the Ph-rich side, but the same calculation in the case of the Hy-rich blends would be less accurate. Moreover, in the case of Hy-rich blends the behaviors of modulus and that of specific volume cannot be related because their relation above T_{σ} is not as certain as in the glassy state. As a consequence, only the Ph-rich blends will be discussed.

The synergistic behavior of the moduli of elasticity of Ph-rich blends may be due to three factors: (1) crystalline content, (2) secondary transition strength decrease, and (3) specific volume decrease. With respect to the crystalline content, the Ph-rich compositions are amorphous. Moreover, the crystalline nature of pure Hy leads to a high reference value. So that crystallinity, although obviously the reason why the Hy-rich blends are in the solid state, is not the reason for the positive deviation from additivity of the modulus values of Ph-rich blends.

With respect to the second possibility, it is known that the presence of a secondary transition affects the modulus of elasticity.^{25,35,36} With respect to the third possibility, the behavior of modulus and that of density have been often related.^{6,9,37,38} A model that relates the positive



Figure 8 Yield stress of the blends against composition.

deviations from additivity of the modulus of elasticity for miscible amorphous blends with those of the density has been proposed.⁶ In the glassy Ph-rich blends, the decrease in specific volume with respect to the direct additivity rule is in agreement with the positive deviations in modulus from the same rule. As a consequence, although nothing can be said in the case of Hy-rich blends, the effect of the negative specific volume of mixing appears clear in Ph-rich blends.

Thus, besides the T_g crossing, which is the responsible for the observed strong modulus decrease at intermediate compositions, and crystallinity, which is the reason for the Hy-rich blends to be in the solid state at room temperature, the specific volume and the β transition suppression are the reasons for the whole modulus behavior of the Ph-rich blends.

The behavior of the yield stress against composition is shown in Figure 8. As can be seen, the plot is similar to that of the modulus. This similarity between the results of both these small deformation properties is often seen in polymers³⁹ and polymer blends,^{22,40,41} and as it is seen, it takes place not only in the rigid state, but also in the Hy-rich side. However, at Ph-rich compositions, the yield stress of the blends is even higher than that of pure Ph. Thus, in this small strain, but not elastic, property, the decrease in deformation ability of Ph-rich blends due to the β transition strength and specific volume decrease, overcomes the effect of the T_g decrease and leads to an increase in the yield stress that is not seen in the modulus. This stronger influence of the decrease in specific volume on the yield stress was also seen in another partially miscible blend.⁴²



Figure 9 Ductility of the blends against composition. The values of Ph and the 90/10 and 70/30 blends were $10.8 \pm 2.6, 4.3 \pm 0.3$, and $4.6 \pm 0.3\%$, respectively.

In Figure 9 the ductility, measured as the elongation at break, is represented against composition. As can be seen, the blends show an important ductility decrease related to that predicted by the additivity rule. There are two clear zones, i.e., the Ph-rich, and the Hy-rich blends. The ductility of the 50/50 blend approaches that of the Hy-rich blends because its T_g is only slightly above room temperature. The two zones correspond to the blends in the glassy and rubber-like states, respectively.

The decrease in ductility of Ph-rich blends has to be related to the observed β transition strength decrease and to the specific volume decrease. As can be seen, these effects even counteract the much more ductile Hy presence, giving rise to a ductility decrease of the blends related to that of Ph. In the 50/50 and Hy-rich blends, when the testing temperature is close to or above the T_{σ} , the high ductility values are a consequence of the definitive effect of the low T_g values that clearly overcome the effect of the specific volume and β transition strength decrease. This ductility behavior is the opposite to that which was found to take place in Ph/PBT blends,43 where linear and also synergistic values on the Ph-poor side were seen. Moreover, additivity in miscible polymer blends when ductility is measured is not unusual.¹⁴ These differences with respect to other blends and this overall behavior will be also probably due, besides to the facts stated above, to the rubber-like nature of Hy and to the nonmonotonous dependence of elongation at break on the T_{g} change.

CONCLUSIONS

Both the temperature and the strength of the β transitions of both Ph and Hy change as a result of blending. The unexpected high strength of the β transition of the 70/30 blend is due to the overlapping of the β transitions of both components. The β transition strength suppression of Ph is important, and is probably due to both specific interactions and to the specific volume decrease.

The mechanical properties of the Hy-rich blends are a consequence of the combined effect of crystallinity and T_g . The abrupt change observed in mechanical properties in intermediate compositions is mainly due to T_g crossing. Finally, in Ph-rich blends, the effect of the decrease in the β transition strength and of the specific volume decrease on mechanical properties is a function of the scale at which deformation takes place. Thus, it takes place at the very low and elastic deformation characteristics of the modulus of elasticity, is more important in the yield stress, and in the case of ductility produces an important deviation with composition that even overcomes that expected from the change of composition.

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