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Water Sorption in Polyamide 6/Poly(Amino-ether) Blends. I. Sorption Characteristics and Phase Behavior

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ABSTRACT

New partially miscible polyamide 6 (PA)/poly(amino-ether) (Blox) blends were immersed in water to study the change in both the significant water uptake of PA, and the phase behavior of the blends. Water sorption was partially irreversible and always Fickian, with a much greater decrease in the water uptake and in the diffusion coefficient of the blends than expected. This was attributed to hindering by Blox of water diffusion both at microscopic and molecular levels.

Plasticization of Blox was clearly less important than that of PA, as its T_g decreased 26°C in wet conditions while in the case of PA the decrease was 60°C up to sub-zero temperatures. No intermediate condition between fully dry or fully wet was seen either in PA or Blox. The displacement of the tan δ peak of PA to clearly lower temperatures and its greater intensity in wet conditions, suggest that the presence of a PA phase might be detected by water sorption, as the tan δ peak is difficult to detect in dry conditions.

Key Words: Polyamide; Poly(amino-ether); Water sorption; Phase structure.

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INTRODUCTION

Sorption of liquids by polymers is an attractive research field that has been widely studied. This is true in the case of both chemically active agents[1] and water.[2–6] Besides the sorption characteristics, the study of the possible plasticization of the host polymer, possible chemical attack and the effects of the liquid on the mechanical behavior are interesting areas of study. These kind of sorption studies have been carried out mainly on pure polymers and in the case of multicomponent systems as polymer composites and polymer blends.[7–15]

Polyamides are widely used materials due to their excellent balance of properties. Poly(ε-caprolactam) (PA) is one of the most commercially extended polyamides, as it shows a good combination of mechanical properties (high rigidity, hardness, impact strength and abrasion resistance), and a low price. In common with other polyamides, it also shows good solvent resistance, with the exception of proton-donor liquids such as water, due to the proton-acceptor nature of PA. For this reason, PA absorbs water both in the liquid state and as a vapor, leading to both plasticization of PA and to a decrease in T_g, stiffness, and strength.[16–18]

Polymer blending is a powerful technique to modify the properties of a polymer. Blending PA with other polymers with lower water affinity could give rise to a reduction on the water sorption[19] and a consequent beneficial effect on the mechanical properties of the material. However, the sorption characteristics of polymer blends have not been well studied in general. In the particular case of PA-based blends, the sorption of aromatic hydrocarbons in PA/rubber blends,[20] that of water and dichloromethane in PA/ethylene–vinyl alcohol copolymer blends,[21] and that of water in compatibilized and uncompatibilized PA/PP,[22] PA/ABS[23] and PA/rubber[24] blends have been studied. In these papers, the influence of composition on the sorption or transport behavior,[20,21,23,24] that of compatibilization on sorption,[22,23] and the temperature dependence[20,24] or the sorption characteristics[20,21,24] were some of the aspects studied.

Poly(hydroxy ether of ethanolamine and bisphenol A) (Blox) is a new polymer with three hydroxyl pendant groups in its repeating unit and thus, with proton-donor characteristics, that has not been the subject of many blending studies. PA/Blox blends have been shown[25] to be partially miscible, as they are composed, besides an almost pure PA phase, of a Blox-rich phase containing large amounts of PA. This partial miscibility and the very small dispersed phase size (0.1–0.5 μm) gave a very positive mechanical behavior with synergisms, not only in stress related properties such as modulus of elasticity and yield stress, but also in a strain related break property, ductility.

The chemical structure of Blox is less prone to water sorption than that of PA, due to the different proton-donor/acceptor nature and the proportion of interacting units. For these reasons, the influence of the presence of Blox on the water sorption characteristics of PA was studied, by leaving PA/Blox blends in the presence of water up to very long times (45 days), and measuring the water uptake and desorption of the blends against time. The corresponding diffusion coefficients were calculated, and the sorption/desorption curves of the blends across the whole composition range discussed. DMTA scans of the wet blends after intermediate and long exposure times to water were performed, and the results used to discuss the phase behavior of the blends under different wetting regimes.

EXPERIMENTAL

The polymers used in this work were PA (Durethan B30S from Bayer Hispania S. A., Barcelona, Spain) and an experimental poly(amino-ether) resin kindly supplied by Dow Chemical under the name Blox. The PA has a molecular weight $Mv = 29,000$, determined by viscosimetry at 25°C in aqueous formic acid (85%). Blox has a melt flow index (MFI) of 9.0 g/10 min, determined at 200°C and with a 2.16 kg load. The T_g's of both pure PA and Blox were measured by DMTA as described below. They were 57 and 80°C respectively in the dry state. After long water sorption times, they decreased down to −8 and 54°C, respectively. Both polymers were dried before processing, in order to avoid moisture-induced degradation reactions, PA for 14 h at 80°C *in vacuo* and Blox for 6 h at 65°C in an air circulation oven.

To obtain the blends, pellets of PA and Blox were mixed at the desired weight ratios and tumbled together before injection molding. The polymers were directly melt mixed and injection-molded in a Battenfeld BA 230E reciprocating screw injection molding machine. The barrel temperature was 230°C and the mold temperature 20°C. The choice of the barrel temperature was determined by the melting temperature of PA and the possibility of degradation reactions of Blox at higher temperatures. The screw of the plasticization unit was a standard screw with a diameter of 18 mm, L/D of 17.8, compression ratio of 4, and helix angle of 17.8°. No mixing devices were present. The injection speed and pressure were 6.1 cm^3/s and 1500 bar, respectively. Tensile ASTM D-638, type IV specimens with a thickness of 2 mm were obtained.

The sorption measurements were carried out at 23°C on injection molded tensile specimens. The samples were immersed in distilled water for periods up to 6 weeks. After the immersion period, they were dried with filter paper to remove the surface liquid and weighed. Subsequent desorption experiments were performed at room temperature for up to 12 days. The solvent content in the samples at time t was calculated as:

$$\% \text{ solvent} = (\text{weight at time } t - \text{initial weight}) \times 100/\text{initial weight}$$

The crystallization–melting and the phase behaviors of the blends were studied by DSC and DMTA, respectively. A Perkin–Elmer DSC-7 calorimeter was used at a heating rate of 20°C/min in a nitrogen atmosphere. Two heating scans were carried out between 15 and 260°C. Cooling between both scans was carried out at the maximum rate provided by the calorimeter. Dynamic mechanical tests were carried out in a Polymer Laboratories apparatus, at a frequency of 1 Hz in flexural mode and at a heating rate of 4°C/min from −50 to 150°C. Samples were cut from the central section of the injection molded tensile specimens.

RESULTS AND DISCUSSION

Water Sorption

In Fig. 1, the water uptake of the blends and of the two pure components is plotted against the square root of time. The volume content of the components of the blends should be used in sorption studies, but given the similar density of the two components of the blends

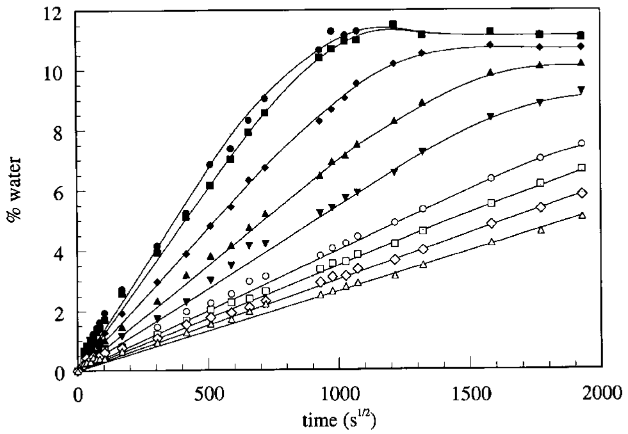

Figure 1. Water uptake curves for PA/Blox blends: (●) pure PA, (■) 90/10, (◆) 80/20, (▲) 65/35, (▼) 50/50, (○) 35/65, (□) 20/80, (◇) 10/90 and (△) pure Blox.

(1.121 and 1.198 g/cm^3 for PA and Blox, respectively), and that the weight composition was used to prepare the blends, the weight composition will be used in the present discussion.

As can be seen in Fig. 1, both in the blends and in the pure components, the water uptake shows initially an overall linear dependence on the square root of time. The intrinsic error of the measurement of data obtained at very short sorption times was comparatively high. Therefore, they were not used to fit the linear rule. This fitting indicates that Eq. (1) holds

$$\frac{M(t)}{M(\infty)} = \mathrm{k}\,\mathrm{t}^{0.5} \tag{1}$$

where M(t) and M(∞) are the water uptake at the time t and at equilibrium, respectively, and k is an empirical constant. Therefore sorption is Fickian as also found in other PA-based blends such as PA/ABS[23] with water, and PA/elastomer with either water[24] or aromatic hydrocarbons.[20] As can also be seen, in the case of both pure PA and the 90/10 blend, the sorption curves show a slight maximum before attaining the equilibrium state. This overshoot effect is well known in polymeric materials,[26–30] and has been also seen in water sorption of PA/elastomer blends[24] at large PA contents. It is attributed to a solvent induced crystallization that decreases the solvent uptake ability of the polymer, as the crystalline phases are impermeable.

As can also be seen in Fig. 1, the sorption speed is maximum for pure PA and decreases as the Blox content of the blends increases. A similar behavior was observed in water sorption of a PA/ABS blend.[23] This behavior is probably related with the high proton-acceptor nature of PA that allows the formation of hydrogen bonds with water, and therefore leads to a high affinity between PA and water, whereas the hydroxyl groups of Blox can behave both as proton-donors and as proton acceptors. However, in an ethylene–vinyl alcohol copolymer (EVOH)/amorphous polyamide blend[14] the hydroxyl groups present in the polymer chain of EVOH showed a stronger affinity with water than that of the amide groups of the polyamide. This was probably due to the number of interacting units per mass unit, that was higher in the component with the higher affinity with water, i.e., EVOH in Ref.[14] and PA in this study. Clustering of water molecules in a sorption process can also take place,[31] increasing the size of the penetrating unit and, thus, decreasing the diffusion coefficient. Moreover, the more hydrophobic the polymer is, the lower is the water concentration for clustering to take place. As a consequence, clustering of water molecules should be more probable in presence of Blox, leading to smaller diffusion coefficients.

To quantify the effects of the presence of Blox on the sorption speed, the diffusion coefficient (D) was calculated by means of Eq. (2):

$$\mathrm{k} = \left(\frac{16D}{\pi L^2}\right)^{0.5} \tag{2}$$

that should be applied to planar specimens,[32] where k is the slope of the sorption curve defined above, and L is half the specimen thickness. The water sorption data at very low sorption times were not used to calculate D, due to the already commented high error of the measurement. Only the D values of the PA-rich blends and medium compositions were calculated, as they are of the greatest practical interest, and because the equilibrium sorption needed to calculate D was not experimentally attained in the case of Blox-rich blends. The values obtained are shown in Table 1.

As can be seen, the sorption speed clearly decreases in the presence of Blox. It should be noted that this decrease is more effective than that could be expected due to the amount of Blox present in the blends. This is because in the case of the 80/20 blend for instance, despite the fact that water is absorbed by Blox, the decrease in D of the blend is a 38% with respect to that of pure PA.

The decrease in water sorption of PA can be calculated from that of the blends and that of pure Blox. This was done for all the blends for a sorption time of 6 days, and the results are plotted in Fig. 2. As can be seen, the calculated water content of PA in the 90/10 blend is the same as that of the pure PA, in agreement with the similarity of equilibrium sorption observed in PA and in the 90/10 blend. With this exception, the ability of the PA for water sorption clearly decreased. This will partially avoid the negative effect of the plastification induced by water uptake on the mechanical properties of PA.

This decrease in the sorption uptake is probably due to the fact that the presence of either Blox-rich particles or Blox matrix hinders the sorption of water by PA. This is because

Table 1. Diffusion coefficients ($D \times 10^{-10}$, cm^2/s) of PA-rich PA/Blox blends in water.

Blend	D
PA	18.6
90/10	15.9
80/20	11.5
65/35	7.5
50/50	6.4

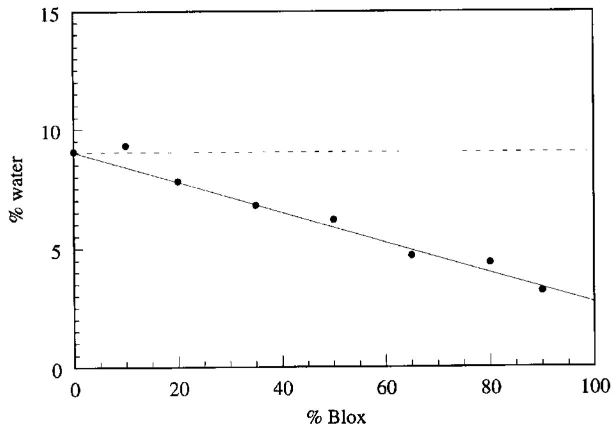

Figure 2. Water content after a sorption time of 6 days per PA gram in PA/Blox blends.

in the case of the almost pure PA phase, the Blox particles imply either a longer (around the Blox particles) or a slower (through the Blox particles) path. In the case of the large amount of PA present in a Blox-rich matrix, Blox will constitute a more efficient molecular scale barrier to the sorption of water by PA. In the case of a PA/elastomer[20] the effect observed was similar, although PA was in this case the impermeable phase and the elastomer the permeable one. Decreases in water uptake were also seen in poly(ethylene oxide)/poly(methyl methacrylate) blends[13] when the hydrophobic polymer content increased.

With respect to the equilibrium sorption, as can be seen in Fig. 1, it also decreases as a consequence of the presence of Blox, and the time for the maximum sorption shows an extraordinary increase as the Blox content increases. For instance, for the 30% Blox blend it is more than six weeks. In a PA/PP blend,[22] the presence of increasing amounts of a compatibilizer gave rise to much longer equilibrium times. This effect does not seem to take place in the blends of this work. This is because, as the PA content increases, the higher PA content in the Blox-rich phase should increase the compatibility of the blend, but the equilibrium time decreased. This is the opposite to that seen in Ref.[22] and is probably due to the increasing Blox content, for which sorption is slow.

With respect to desorption, the water content of both the blends and the pure components is shown in Fig. 3 against the square root of desorption time. As can be seen, the desorption speed decreases with both time and Blox content. The correspondent desorption coefficients (D_d) are shown in Table 2. As can be seen in Fig. 3, in agreement with the hindering of sorption by Blox, desorption is also hindered. The decrease in D_d (32% with respect to that of pure PA) is larger than the Blox composition, but smaller than the decrease observed in sorption (38%). Surprisingly, the higher desorption speed of the Blox-poor blends with large water content, led to the fact that some curves crossed each other after long desorption times.

Finally, as can also be seen, the sorption process was not fully reversible. This was because, although full desorption was not attained, the extrapolation of the desorption curves clearly showed that significant water contents between 2 and 4% roughly, will very probably remain in the blends. The irreversibility of the process was also clearly depicted

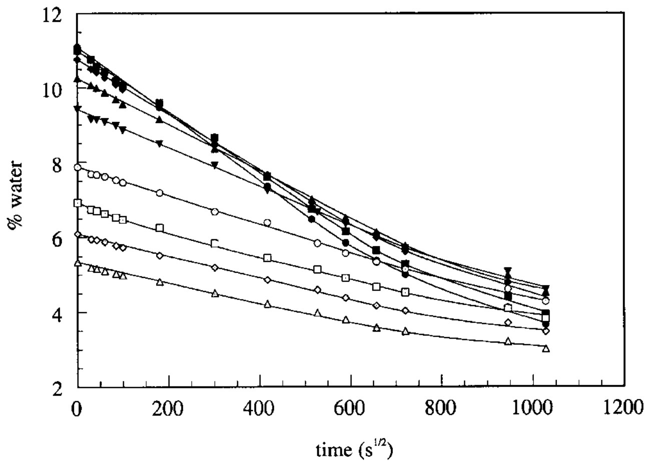

Figure 3. Desorption curves for PA/Blox blends. Symbols as in Fig. 1.

by the presence after desorption in Blox and the 10/90 blends of small cracks (typically between 1 and 5 mm long) in the surface of the specimens which are shown in Fig. 4.

Phase Behavior Upon Sorption

As found previously,[25] PA/Blox blends are constituted by a PA crystalline phase (30% of the PA content whatever the Blox content), an almost pure amorphous PA phase, and a mixed mostly Blox-rich phase with PA contents that range from 8% in the 20/80 blend to 30% and 62%, respectively, in the 50/50 and 80/20 blends. The presence of water in the blends and the consequent plastification can modify the amount and the nature of these three phases. In fact, it is known that water sorption can produce phase separation in miscible blends such as poly(vinyl pyrrolidone) (PVP)/polysulfone (PSF),[15] PVP/phenoxy[8] or poly(ether sulfone)/polyethyloxazoline.[11] These possible modifications will be studied both by DSC and DMTA.

Table 2. Diffusion coefficients ($D_d \times 10^{-10}$, cm^2/s) of PA-rich PA/Blox blends in water during the desorption process.

Blend	D_d
PA	10.4
90/10	8.2
80/20	7.0
65/35	6.4
50/50	4.7

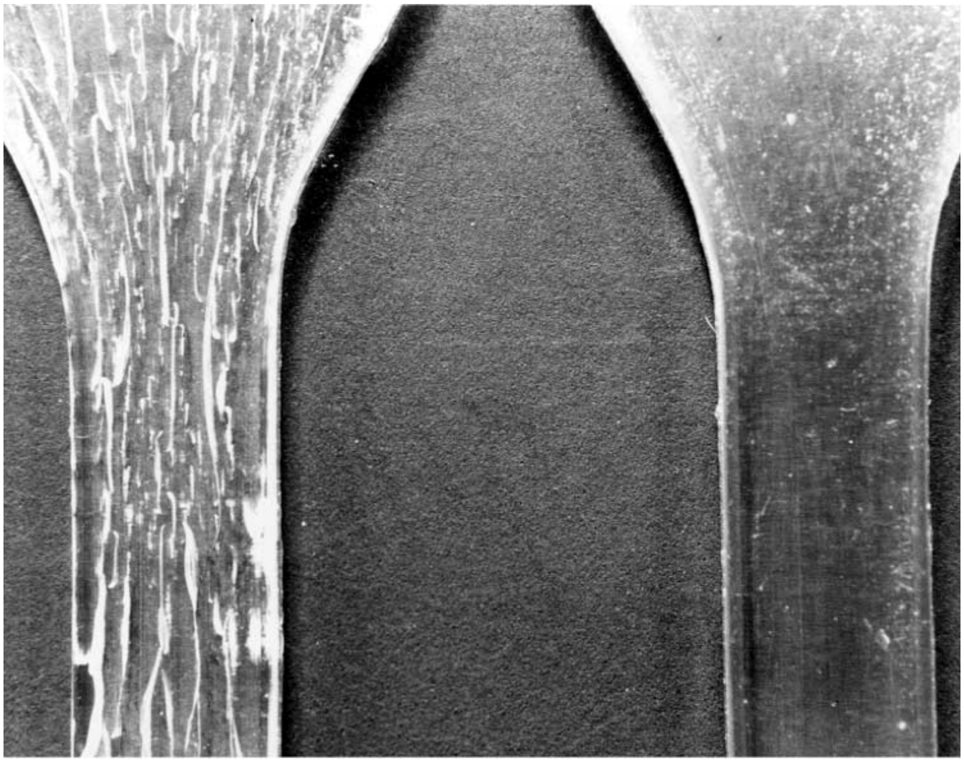

Figure 4. Specimens of pure Blox before sorption (right) and after desorption (left).

A possible change of the crystalline content of the blends could not be measured by DSC due to the irregular shape of the endotherms of the first scan. This was probably due to the presence of a significant amount of water in the samples. A second scan would not be of interest, as the crystalline characteristics would very probably change. However, some information about solvent-induced additional crystallization of PA can be inferred from Fig. 1. As can be seen, after a maximum correspondent to equilibrium, there was a decrease in the water content. This decrease was significant in pure PA and slight in the 90/10 blend, but was not observed in the rest of the blends. The decrease is due[27,33] to the additional crystallization induced by the presence of solvent. Crystallization decreases the amount of free volume available to host water molecules, and therefore, it expels water from PA. This effect can be used to estimate the crystallinity increase on sorption. The decrease in water content was roughly 0.25% in the 90/10 blend and its amorphous phase contains 11.4% water in the overshoot zone. Therefore, a decrease in water content of 0.25% corresponds to a decrease in the amorphous phase content of the order of 2.2%. Thus, the crystallinity of the PA will change from 30% before sorption to roughly 32% after complete sorption. A crystallinity increase of 2.2% is almost insignificant as it is close to the estimated accuracy of the measured crystalline content value.

The melting temperature of PA did not change after sorption. This indicated that the crystalline characteristics of the PA crystalline phase did not change upon sorption, as could be expected due to the well-known impermeability of the crystalline phase to liquids.

The phase behavior of the blends was also studied by DMTA. The different effects of the presence of water in the two phases of the blend were clearly seen in the 80/20 blend, therefore no additional blend was studied. The plots of the tan δ of the wet blend were complex. Therefore, to help discussion, the tan δ scans of the pure PA after different water sorptions will be discussed first, and the data shown in Fig. 5.

As can be seen in Fig. 5 for pure PA in which the tan δ curve of dry PA (curve a) and that of PA after long sorption time (curve d) are compared, the tan δ peak of the dry PA at 57°C (curve a), slips after long sorption time (curve d) to −8°C. This indicates the very strong plasticization effect of water on PA,[17] and shows the position of the peak of the equilibrium soaked PA. As can also be seen, the peak of curve (d) is distinct and similar in width to that of the dry PA of curve (a); this implies that the presence of intermediate humidity states after a long sorption time is not possible.

The scans of PA with intermediate water contents (curves b and c), show two tan δ peaks at temperatures very similar (the high temperature peak is closer) to those observed in dry PA (curve a) and in equilibrium soaked PA (curve d). As can also be seen, as the water content increases, the intensity of the dry PA peak decreases, whereas that of the soaked PA increases. The similarity in temperature of the two peaks with those of dry PA and wet PA, their distinct shape and width similar to that of dry PA, indicate that the type of PA is either dry or an almost equilibrium soaked, and that PA with intermediate water contents is not found even after an intermediate sorption time. This experimental fact agrees with the Fickian nature of sorption, as it assumes sorption to be much slower than polymer relaxation. The fast relaxation of PA is seen in curves (b) and (c) of Fig. 5; where, although PA is still far from equilibrium, the maximum relaxation peak at −3°C is obtained both in curves (b) and (c).

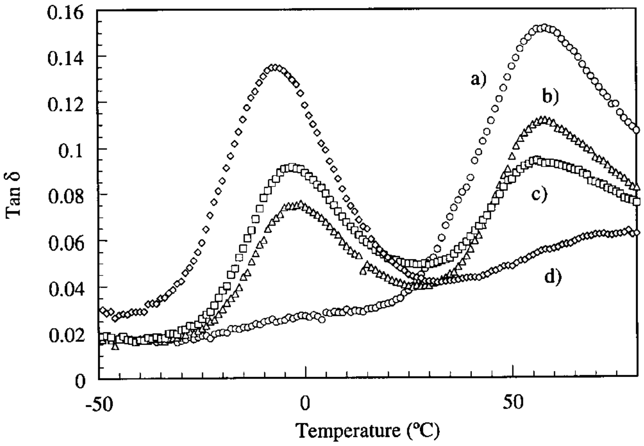

Figure 5. DMTA scans for pure PA before (○), and after 12 (△), 24 h (□) and long sorption times (◇).

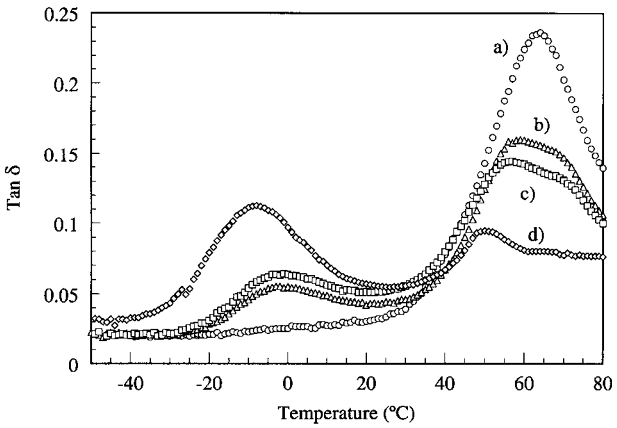

Figure 6. DMTA scans for 80/20 composition PA/Blox blend before (○), and after 12 (△), 24 h (□) and long sorption times (◇).

Figure 6 shows the tan δ scans of the 80/20 blend against temperature in the dry state (curve a), after 12 and 24 h sorption times (curves b and c) and at equilibrium (curve d). The behavior of pure Blox at very long sorption times (1 year) is not shown in Fig. 6 due to the much higher intensity of the peak (0.6), which was centered at 54°C. As can be seen, and despite the biphasic nature of the blend,[3] a single tan δ peak is seen in curve (a) of the dry blend. The blend contains both PA and Blox; therefore, it corresponds to the addition of the peak of the pure PA at 53°C, and that of the much more prominent (ten-fold that of PA) tan δ peak of the Blox-rich phase at 64°C.

As can also be seen, after a long sorption time, (curve d), two tan δ peaks appeared. The location of the low temperature peak at − 8°C corresponds to a fully plasticized PA, because it appeared at the same temperature in pure PA. The peak at high temperature (50°C) must be due to the equilibrium soaked Blox-rich phase as it appears at almost the same temperature as that of soaked Blox (54°C). Its temperature is lower than that in the dry blend, indicating that Blox was also plasticized by water, in agreement with the significant water content of Blox at long sorption times. The position of the peaks of both components in the wet state was similar to that of the pure components in the same conditions. This indicated that the presence of a blend component does not influence the plastification of the other. This is the contrary to that took place in an amorphous PA/EVOH blend.[14]

As can also be seen in curve d) of Fig. 6, the presence in the wet blends of the two tan δ peaks of the 80/20 blend is very clear. This is the opposite of the tan δ plot of the dry blend of curve a) and of Ref.[25] in which the presence of two peaks, as in other case,[14] was really very difficult to detect. This suggests that the sorption of a liquid plasticizer may be an experimental technique to reveal the presence of phases with transitions difficult to observe, either because they are close together or very broad.

In the case of the blend after intermediate sorption times (curves b) and c)), two tan δ peaks also appeared. The low temperature peaks must correspond to the plasticized wet PA. The nature of the phase that gives the wide peak from 55 to 70°C is more difficult to state. This peak could be due to a) remaining unplasticized PA that was seen to exist in Fig. 5 for the neat PA at the same sorption times, b) remaining unplasticized Blox-rich phase as water uptake was slower than in PA and, c) a fully plasticized Blox-rich phase. Taking into account that neither of the 3 possibilities can be neglected, and that the peak appears to comprise several overlapping peaks, the presence of the three phases is proposed.

CONCLUSIONS

Water sorption of PA/Blox blends is Fickian with a slight overshoot in pure PA and in the 90/10 blend that was attributed to a slight solvent-induced crystallization. The presence of Blox clearly hindered sorption, as the decrease in the water uptake and the reduction of the diffusion coefficients upon blending were much larger than that Blox content could indicate (a 38% decrease in the diffusion coefficient of the blend for instance with 20% Blox). This is attributed to the more difficult advance of water due to the presence of the less permeable Blox both at microscopic and molecular scale. The sorption process was partially irreversible as some water probably remains after sorption, and small cracks appeared in the surface of the specimens of both pure Blox and the 10/90 blend.

PA to a large extent (T_g decrease 60°C) and Blox to a minor extent (T_g decrease 26°C) were plasticized by water. This led to the coexistence at intermediate sorption times in pure PA of a completely dry and an equilibrium sorption PA phase, and to the presence in the blends of similar Blox phases. The presence of two clear $\tan\delta$ peaks in the saturated blends was in clear opposition to the single peak observed in the dry state. This suggests that plasticization by a liquid solvent may be a method to reveal transitions when they are either very broad or very close together.

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