Interfacial Tension of PBT/SAN Blends by the Drop Retraction Method

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The aim of this work was to evaluate the interfacial tension from the poly(butylene terephtalate) and poly(styrene-co-acrylonitrile) (PBT/SAN) interface region using the drop retraction method. SAN filaments were sandwiched between two PBT films; the whole system was heated up to 240 °C, in a hot stage coupled to an optical microscope. The rheological parameters of the PBT/SAN system were obtained by parallel plates rheometry. An increase of the interfacial tension with the PBT molecular weight was observed with values between 0.57 and 1.06 mN/m, depending on the molecular weight. Theoretical values were calculated using the geometric-mean and harmonic-mean equations and were found to be similar to the experimental results. Viscosity measurements showed that the higher the SAN/PBT viscosity ratio, the lower the interfacial tension of these blends.

1. Introduction

The development of new polymeric blends is an important and creative technological way of obtaining new applications for polymeric materials, decreasing costs and optimizing properties of the final products. Most of the polymers are immiscible during their mixture with other polymers and form multiphase systems with a variety of morphologies, like: disperse droplets, fibrils, lamellar and co-continuous structures[1]. The type of blend morphology affects strongly its final properties. Therefore the morphology control is a critical factor in the optimization of the polymer blend performance. This morphology depends on the interfacial tension, among other parameters[1]. Favis and Willis[2] have pointed out that the morphology of immiscible polymer blends seems to be controlled by the following parameters, in order of importance: interfacial tension > viscosity ratio > shear stress.

Studies on toughening of PBT with other rubber modified plastics containing high amount of elastomers, like ABS and AES, have concentrated efforts on simpler systems, for a better understanding of the blend morphology establishment, due to the complexity of these multiphase systems. The adequate dispersion of both styrenic terpolymers in the PBT matrix depends strongly on the interfacial tension of the system. Usually the poly(styrene-co-acrylonitrile), SAN, matrix phase from the ABS or AES terpolymer generates the interfacial region with PBT in the PBT/ABS and PBT/AES blends. In addition, those blends have another well established interface region between the rubbery phase and the SAN phase generated by the terpolymers synthesis. Paul and his collaborators[3,4] have done many studies to understand those blends performance. However, the interfacial tension for PBT/SAN has not yet been evaluated to contribute to those studies.

Thus, the main objective of this work was to measure the interfacial tension between PBT and SAN, using the drop retraction method, developed by Mo et al.[5] and to compare the results with theoretical results.

2. Interfacial Tension Theoretical Background

The surface tension is a direct manifestation of intermolecular forces at any surface. The surface molecules of a liquid or solid material are influenced by the unpaired molecular forces due to the surface discontinuity, which leads to surface molecules having additional energy. When those molecules are in contact with surrounded air the surface tension increases as the intermolecular forces among the surface molecules increase. On the other hand, the contact of a material surface with other material different than air generates an additional energy in the interface region. The additional free energy at the interface between two phases is known as interfacial tension[6]. The magnitude of the interfacial tension depends on the surface tension of each contact surface.

Many authors[7-17] have used theoretical predictions from empirical equations that correlate interfacial tension with surface tension. From these empirical equations, the interfacial tension (γ) between two materials can be calculated through the harmonic mean equation (Equation 1) or through the geometric mean equation (Equation 2):

$$\gamma = \gamma_1 + \gamma_2 - \frac{4\gamma_1^d\gamma_2^d}{\gamma_1^d + \gamma_2^d} - \frac{4\gamma_1^p\gamma_2^p}{\gamma_1^p + \gamma_2^p} \tag{1}$$

$$\gamma = \gamma_1 + \gamma_2 - 2\left(\gamma_1^d\gamma_2^d\right)^{1/2} - 2\left(\gamma_1^p\gamma_2^p\right)^{1/2} \tag{2}$$

where γ_1 is the surface tension of material 1, that is, $\gamma_1 = (\gamma_1^d + \gamma_1^p)$, γ_2 is the surface tension of material 2, or $\gamma_2 = (\gamma_2^d + \gamma_2^p)$, where the superscript "d" refers to the non-polar contributions (dispersion contribution) and the superscript "p" to the polar contributions to the surface tension of each material.

To estimate the interfacial tension (γ) through Equations 1 and 2 it is necessary to know the polar(γ_i^p) and the non-polar (γ_i^d) contributions to the surface tensions γ_i. Both surface tension contributions may be represented as fractions of γ_i, as can be observed in the Equation 3. The polar contribution fraction may be referred as the surface polarity χ_i^p, while the non-polar contribution fraction is added to one. Therefore, if both surface tension and the surface polarity are known for each material, it is possible to find γ_i^p and γ_i^d, respectively.

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$$\chi_i^p = \frac{\gamma_i^p}{\gamma_i} \qquad (3)$$

The surface polarity has been considered independent of temperature, i. e., $\frac{d\chi_i^p}{dT} = 0$

The surface tension can be easily obtained for any material through experimental measurements or can be found in the literature through specific handbooks. On the other hand, the surface polarity χ^p can be obtained as a function of the cohesive energy density ΔE or as function of the solubility parameter, as shown in Equation 4:

$$\chi^p = \left(\frac{\Delta E^p}{\Delta E} \right) = \left(\frac{\delta_p}{\delta} \right)^2 \qquad (4)$$

where, ΔE^p is the polar contribution to the ΔE and δ_p is the polar contribution to the overall solubility parameter δ. The surface polarity values obtained from surface tension have been found to agree with those obtained from cohesive energy densities calculations for a high number of polymers[18].

Hansen and Skaarup[19] assumed that the cohesive energy density, ΔE, which is proportional to δ, arises from dispersive interactions (δ_d), permanent dipole-dipole interactions (δ_p) and hydrogen bonding forces (δ_h), and is obtained as shown in Equation 5:

$$\delta^2 = \delta_d^2 + \delta_p^2 + \delta_h^2 \qquad (5)$$

Those parameters (δ_i) are referred as Hansen parameters; however, cannot predict the overall δ with accuracy for all the materials systems. The specific interactions between groups and molecules may not be given by a simple addition rule. Even so, the estimation of the Hansen parameters can be quite useful. The Hoftyzer and van Krevelen methods[19] allow estimations of those parameters by the following expressions:

$$\delta_d = \frac{\sum_i F_{di}}{V_{molar}} \qquad (6)$$

$$\delta_p = \frac{\sqrt{\sum_i F_{pi}^2}}{V_{molar}} \qquad (7)$$

$$\delta_h = \sqrt{\frac{\sum_i E_{hi}}{V_{molar}}} \qquad (8)$$

where F_{di} is the dispersive force contribution, F_{pi} is the polar force contribution, E_{hi} is the hydrogen bonds energy, for each chemical group of the polymer and V_{molar} is the molar volume. Any of the Hansen parameters can be easily calculated for any polymer if its molecular repeat unit is known. The respective values for F_{di}, F_{pi} and E_{hi} are found tabulated in specific handbooks[19]. The surface polarity χ^p values for polymers determined through the use of interfacial properties have been observed to have good correlation with the ones determined using the cohesive densities[19]. It should be cautioned, however, that Equation 3 defines the surface polarity, while Equation 4 defines the bulk polarity. These two values may not be the same, if the polymer has sufficiently long surface-active segments, which tend to preferentially adsorb on the surfaces.

Therefore, from the above calculation procedures it is possible to estimate the the interfacial tension (γ) between two materials of the Equations 1 and 2.

In the molten state it is very difficult to measure the interfacial tension between polymers due to their high viscosities. Therefore, numerous methods have been developed to measure this parameter, which can be classified as: thermodynamics (static), dynamics and rheological[20].

The thermodynamics methods require long measurement times, due to the polymers high viscosities; therefore, there is a risk of polymer thermal degradation. The main thermodynamic methods are: the pendent droplet, the sessile droplet and the rotating droplet[20].

The dynamic methods have the advantage of taking less time, thus compensating problems due to thermal perturbation. The main dynamic methods are: the breaking thread and the droplet retraction. The "breaking thread" is more complex from the experimental point of view and especially for systems were the viscosity ratio is higher than one[20].

As a matter of fact there is not a preferential method for measuring the interfacial tension, because each one has its advantages and disadvantages.

In the three-dimensional deformation model, the drop shape is subsequently described by a symmetric, positive-definite, second rank tensor S; its eigenvalues represent the square semi-axis of an ellipsoid. The evolution of this tensor results from the action of the interfacial tension and from the hydrodynamic drag exerted by the movement and can be described by an evolution model proposed by Maffettone et al.[21], given by:

$$\frac{dS}{dt} - \Omega \cdot S + S \cdot \Omega = -\frac{f_1}{\tau} \left[S - g(S)I \right] + f_2 \left(E \cdot S + S \cdot E \right) \qquad (9)$$

where Ω is the vorticity tensor, f_1 and f_2 are coefficients which are functions of the viscosity ratio, τ is a characteristic time, I is a second order unit tensor and E is the deformation rate tensor.

Assuming constant droplet volume, Mo et al.[5] developed an equation for the evolution of S in a flow field. If E and Ω are equal to zero, Equation 9 can be simplified to:

$$(\lambda_1 - \lambda_2) = (\lambda_1 - \lambda_2)_0 \exp\left(-f_1 \frac{t}{\tau} \right) =$$

$$(\lambda_1 - \lambda_2)_0 \times \exp\left[-\frac{\gamma}{\eta_m R_0} \frac{40(p+1)}{(2p+3)(19p+16)} t \right] \qquad (10)$$

where : $\lambda_1 = L^2$, $\lambda_2 = B^2$, L being the larger semi-axes and B the smaller semi-axes, η_m is the matrix viscosity, p is the viscosity ratio $= \eta_d/\eta_m$, where η_d is the viscosity of the disperse phase, R_0 is the equilibrium droplet radius and t is the measurement time in seconds.

3. Experimental

3.1. Materials

Three different molecular weights PBT's were used in this work, named Valox 195, 325 and 315, from GE South America Plastics. The SAN, grade CN77E, was from Bayer Polymers S.A.

3.2. Melt flow index

Melf flow index was measured following ASTM D1238-00 standard procedure using a load of 1.20 kg and a temperature at 250 °C.

3.3. Molecular weight measurements

The PBT number average molecular weight, $\overline{M}_n$ was determined by measuring the intrinsic viscosity [η] using an Ubbelhode viscosim-

eter and a solvent mixture of 60/40 (weight %) of phenol (Synth) and 1,1,2,2-tetrachloroethane (J.T. Baker).

The PBT samples were previously dried during 16 hours at 60 °C under vacuum. PBT solutions were prepared using a concentration of 0.3 g/dL at room temperature.

The intrinsic viscosity [η] was measured at 30 ± 0.1 °C using the one point method[22,23] and the relationships given by Solomon and Ciuta (Equation 11), Rao and Yaseen (Equation 12) and Deb and Chatterjee (Equation 13).

$$[\eta] = \left(\sqrt{2} / c\right)\left(\eta_{sp} - 1n\eta_r\right)^{1/2} \tag{11}$$

$$[\eta] = \left(1 / 2c\right)\left(\eta_{sp} + 1n\eta_r\right) \tag{12}$$

$$[\eta] = \left(1 / c\right)\left(31n\eta_r + 1,5\eta_{sp}^2 - 3\eta_{sp}\right)^{1/3} \tag{13}$$

where c = solution concentration, η_{sp} = specific viscosity = $(\eta_r - 1)$ and η_r = relative viscosity = η/η_o, where η is the measured viscosity and η_o is the solvent viscosity. The intrinsic viscosity [η] was averaged out from values obtained through each one of the above equations. The correlation between $\overline{M}_n$ and [η] given by Borman[24] was used, as shown in Equation 14:

$$[\eta] = 1.166 \ 10^{-4} \ \overline{M}_n \tag{14}$$

The average molecular weight of the SAN copolymer samples was determined by gel permeation chromatography, GPC. The SAN solutions were prepared in tetrahydrofurane solvent (Vetec). The solution was filtered and injected at 40 °C at 1 mL/min in a Waters 410 chromatograph, with a refraction index detector and Ultrastyragel columns. The calibration curves were constructed with 12 PS monodisperse standard samples from Shodex Standard.

3.4. Determination of the amount of acrylonitrile in the SAN copolymer

A Fisons Instruments equipment model EA 1108 CHNS-O was used to determine the concentration of nitrogen of the SAN samples.

The amount of nitrogen (% N) measured was therefore used to calculate the % of acrylonitrile in the SAN copolymer, by using the following relationship:

$$\%Acrylonitrile_{SAN} = \frac{\%N \times 100}{26.42} \tag{15}$$

3.5. Rheological measurements

The zero shear viscosity η_0 was measured in a strain controlled rheometer, ARES, from Rheometrics, at 240 °C, under nitrogen atmosphere, using parallel plates with 25 mm diameter and gap of 1 mm. The time for measurement of η_0 was similar to the time for the droplet retraction.

3.6. Interfacial tension measurement

Films of PBT were produced by compression molding, at 240 °C, after drying the PBT at 60 °C, during 12 hours, under vacuum. SAN filaments were prepared after heating the polymer at 220 °C; the SAN was therefore drawn to produce filaments with 80~120 μm of diameter. These filaments were set between the PBT films, forming a "sandwich" structure. This "sandwich" structure was fixed between two glass slides and the whole set was heated up to 240 °C using a hot stage THMS 600 from Linkan. To avoid thermal degradation, a nitrogen saturated atmosphere was used. The thread rupture and droplet retraction was monitored using a transmitted light optical microscope, Leica Model DM RXP and image acquisition software Image-Pro Plus. The droplet aspect ratio was thus determined.

4. Results and Discussion

Table 1 shows the melt flow index of the polymers and the values of number average molecular weights, $\overline{M}_n$, of the PBT and SAN samples obtained as received and after the interfacial tension measurements.

Table 2 shows the values of η_0 of the polymers, at different times. The viscosity ratio p = η_{SAN}/η_{PBT} was calculated from those results (Table 3).

The PBT surface tension at 240 °C was calculated using the values of surface tension at 20 °C and (-dγ/dT) of the literature[16] for

Table 1. Melt flow index and number average molecular weights of the polymers.

Polymer	MFI (g/10 min) 250 °C / 1.20 kg	$\overline{M}_n$ (g/mole)	$\overline{M}_n$ (g/mole), after submitted to 240 °C for different times
PBT 20 (Valox 195)	71.3[*1]	21,300 ± 2,400[*2]	24,300 ± 2,700[*2] (15 min)
PBT 30 (Valox 325)	10.0[*1]	33,600 ± 3,800[*2]	30,100 ± 3,400[*2] (30 min)
PBT 40 (Valox 315)	6.2[*1]	41,700 ± 4,600[*2]	40,900 ± 4,600[*2] (50 min)
SAN (CN77E)	10.0[*1]	46,900[*3]	41,500[*3] (15 min)
(75% S / 25% AN[*4])			41,000[*3] (30 min)

[*1]Following ASTM D1238-00 standard procedure; [*2] By solution viscosimetry; [*3] By GPC; [*4] From equation 15.

Table 2. Zero shear viscosity, η_0, of the PBT and SAN samples, measured at 240 °C for different times.

	η_0 (Pa.s)			
Polymers	t = 0 min	t = 15 min	t = 30 min	t = 50 min
SAN	566	528	526	504
PBT 20	79	105	-	-
PBT 30	489	-	447	-
PBT 40	1,022	-	-	670

Table 3. The viscosity ratio, p, of the PBT and SAN samples, measured at 240 °C for different times.

p = ηSAN/ηPBT	t = 0 min	t = 15 min	t = 30 min	t = 50 min
SAN/PBT20	7.16	5.03	-	-
SAN/PBT30	1.16	-	1.18	-
SAN/PBT40	0.55	-	-	0.75

the poly(butylene isophthalate), due to their chemical similarity as shown in Figure 1.

The surface tension of SAN with 25% of acrylonitrile at 20 °C was calculated by interpolation in a surface tension versus % acrylonitrile curve obtained from the literature[16].

The values of density ρ for both SAN and PBT, shown in Table 4, were obtained from the data file of the Moldflow MPI 4.1 software.

It was verified that the majority of the (-dγ/dT) values[18] for polymers is in the range between 0.06 and 0.08; therefore due to the difficulty of calculating the SAN surface tension at 240 °C, those values were used to estimate its surface tension, which is also shown in Table 5.

The value of χ^p for the PBT was obtained from the literature[16] while the value of χ^p for the SAN copolymer was calculated from the cohesive energy density, Equation 4. The values of χ^p, γ^d and γ^p for the PBT and the SAN are given in Table 4.

Figure 2 shows the SAN filament breaking and drop retraction in the PBT matrix. These pictures were taken after 45 minutes at 240 °C, with 30 seconds of time interval between each picture.

The change in the SAN droplet retraction dimensions, given by the difference $\lambda_1 - \lambda_2$, where $\lambda_1 = L^2$, $\lambda_2 = B^2$, as in Equation 10, was measured as a function of time and it is shown in Figure 3. The average interfacial tension (γ) was obtained from the slope of the curves at different R_0, and it is shown in Table 5.

It can be observed that the results obtained from the drop retraction experiments reasonably agree with the calculated values from the harmonic mean and the geometric mean equations.

The empirical equations are useful for calculating the interfacial tension although there is a difference between the experimental and

the calculated data because the calculation procedure has referred to the polymer as mer units and the experimental data make use of the polymer bulk to obtain the interfacial tension. However, there is no other data for PBT/SAN systems to compare with the experimental results and its can give an indication of the efficiency of the method. In addition, the experimental values stay between the harmonic
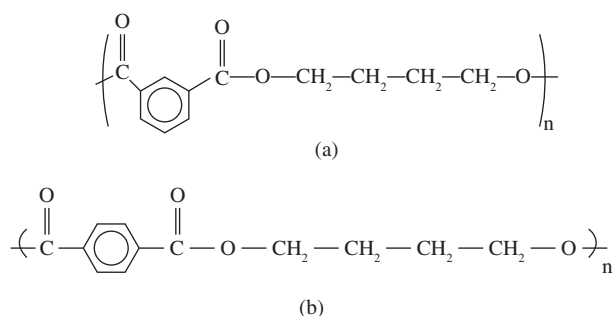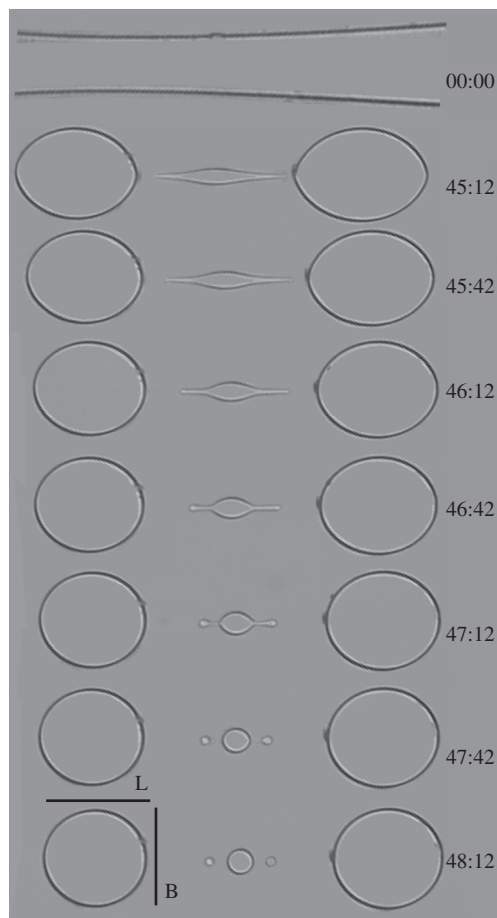

Figure 2. SAN filament break and droplet retraction in a PBT matrix after 45 minutes at 240 °C, where L and B are the semi-axis of the ellipsoid drop, respectively.

(a)

(b)

Figure 1. Chemical structure of: a) Poly(butylene isophthalate) and b) Poly(butylene terephthalate) (PBT).

Table 4. Density (ρ), surface tension (γ), (-dγ/dT), surface tension components (γ^d, γ^p) and polarity (χ^p) for PBT and SAN, at different temperatures.

	ρ (25 °C) (g/cm³)	ρ (240 °C) (g/cm³)	γ (20 °C) (mN/m)	−dγ/dT (mN/mK)	γ (240 °C) (mN/m)	χ^p	γ^d (240 °C) (mN/m)	γ^p (240 °C) (mN/m)
PBT	1.31	1.05	47.8	0.080	30.2	0.270	22.05	8.15
SAN	1.06	0.96	41.5	0.06	28.3	0.150	4.25	24.05
				0.07	26.1		3.92	22.18
				0.08	23.9		3.59	20.31

Table 5. Experimental and calculated values of the interfacial tension (γ) of the PBT/SAN system.

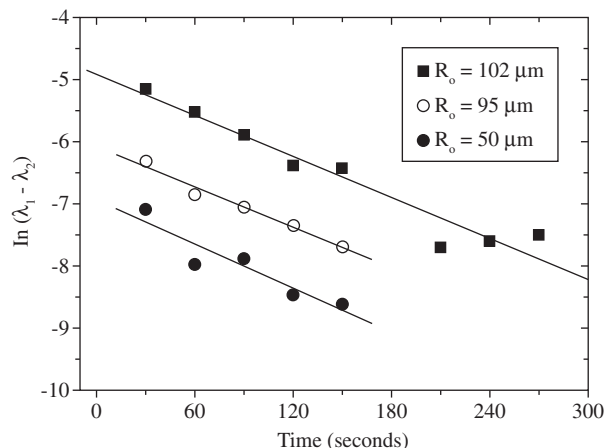| γ (mN/m)	Experimental	Calculated values from harmonic mean equation (−dγ/dTSAN)			Calculated values from geometric mean equation (−dγ/dTSAN)		
		0.06	0.07	0.08	0.06	0.07	0.08
PBT20/SAN	0.57 ± 0.02	-	-	-	-	-	-
PBT30/SAN	0.87 ± 0.28	1.32	1.49	1.85	0.67	0.77	0.96
PBT40/SAN	1.06 ± 0.01	-	-	-	-	-	-

Figure 3. The change in the SAN droplet rectraction dimensions as function of the time.

and geometric means calculated values, as already shown by Wu[16] in several polymer pair systems (PEO/PDMS, PVAc/PDMS, PCP/PDMS, PCP/PnBMA, PCP/PS, PMMA/PNBMA, PMMA/PtBMA, PMA/PEHA, etc). It is also observed from Table 5 that the change in $(-d\gamma/dT)$ from 0.06 to 0.08 does not change significantly the calculated interfacial tension values. For the PBT/SAN system, it was observed an increase in the interfacial tension as a function of the increase in the PBT molecular weight, as already observed by Kamal[25] for PP/PS systems. For that system the increase was observed up to a certain molecular weight then it leveled off for further increase.

The calculated values of the interfacial tension for PBT/PS blends were obtained by Bu and He[18] at 250 °C. They used the harmonic mean equation to obtain those values and found the interfacial tension to be equal to 1.29 mN/m, which is in the same range of results for the PBT/SAN system found here.

5. Conclusions

The interfacial tension of the PBT/SAN system was measured at 240 °C using a modified droplet retraction method, developed by Mo et al. The experimental values have shown a reasonable agreement with the calculated ones using the harmonic and geometric averages equations. The increase in the PBT molecular weight promoted an increase in the interfacial tension for the PBT/SAN system.

6. Acknowledgments

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