Mechanical properties of ternary blends of ABS + HIPS + PETG

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Abstract

The effect of a commercial styrene/butadiene/styrene-based compatibilizer (Styroflex) on the tensile and impact properties of ternary blends of poly(acrylonitrile-*co*-butadiene-*co*-styrene) (ABS), high impact poly(styrene) (HIPS) and poly(ethylene terephthalate-*co*-cyclohexanedimethanol terephthalate) (PETG) was investigated. The tensile yield strengths and the moduli of the blends were of similar magnitude as the parent polymers. However, notched Charpy impact properties showed significant deviations with high synergy in ABS/PETG blends and strong antagonism in HIPS/PETG blends. Addition of Styroflex improved the impact properties of all blends containing HIPS and ABS. Dynamic mechanical analysis studies confirm the phase separated nature of ABS/PETG binary blends.

Key words: ABS; HIPS; PETG; polymer blends; Charpy impact strength

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Introduction

Poly(ethylene-*co*-cyclohexane 1,4-dimethanol terephthalate) (PETG) is an amorphous polyester widely used in plastic sheet and other applications. It combines good melt strength and toughness with excellent sheet clarity [1]. In developing countries plastic sheet extrusion production runs are often short and the same equipment is used to produce several different products. The changeover from one polymer to another produces co-mingled industrial waste. In South Africa large quantities of (PETG) contaminated with other polymers is generated for which there is currently little or no market demand. Thus the purpose of this study is to investigate strategies that could facilitate the recycling of PETG containing factory scrap.

Similar mixed plastic waste forms result during the production of laminated sheet products [2] and the recycling of waste computer equipment [3]. The reprocessing of such factory scrap as a blend of the component polymers can be considered provided the mixture exhibits acceptable physical properties [2, 4]. Unfortunately immiscibility is the rule for most polymer pairs and this inevitably leads to phase separation, poor interfacial adhesion and brittle materials [5, 6]. Blends of ABS with poly(ethy1ene terephthalate) (PET) are an exception showing enhanced impact properties compared to the parent polymers [7, 8].

The mechanical properties of multiphase blends are dependent upon the properties of the constituent polymers, the relative proportion of the blend components, and the spatial organization of the phase domain structures including the interfacial region or "interphase" and the extent of adhesion at the interface [3, 5, 9, 10]. Polymer compounding involves complex flow fields and final polymer shaping involves rapid cooling of viscous polymer melts. Consequently the blends usually do not achieve thermodynamic equilibrium but

become kinetically trapped in non-equilibrium morphologies [Scott 1995]. Miscible blends usually show property values that are intermediate to those of the parent polymers. However, nonlinear property improvements are possible in "compatible" phase-separated blends [6, 7, 9]. The term "compatibility" is used in technological literature when the blend has acceptable mechanical properties [4]. Compatibilization can be effected by the addition of small amounts of a third polymer, e.g. a graft or block copolymer of the two constituent polymers [4, 11, 7, 8, 9, 12]. This improves interfacial adhesion and allows the achievement of a finer dispersion of phases [4, 9]. For example, poly(ethy1ene terephthalate) (PET) blends with poly(acrylonitrile-butadiene-styrene) (ABS) are stabilized by adding maleic anhydride grafted ABS as compatibilizer [8] and ABS/ poly(butylene terephthalate) blends are further toughened by poly(styrene-*co*-glycidyl methacrylate) [12].

The use of elastomeric compatibilizers in phase-separated blends tends to shift mixture properties from hard and brittle towards soft and tough by forming separate rubbery phases [12]. This is illustrated by the continuous improvement in impact strength of a 50/50 PBT/HIPS blend on adding increasing amounts of styrene-butadiene-styrene triblock copolymer [12]. This is advantageous as impact toughness usually is the mechanical property that suffers most from blend incompatibility. However, the concomitant loss in flexural modulus (stiffness) is an undesirable consequence.

Lindsey et al. [2] and Brennan et al. [3] reported that blends of ABS and HIPS show near additivity of modulus, tensile strength and elongation at break. However, Brennan et al. [3] reported reduced impact strength and suggested the use of a suitable impact modifier. Cook et al. [7] observed that ABS/PET blends show a distinct maximum in the notched Izod impact strength near the 50/50 composition. ABS is also widely employed as impact modifier for polycarbonate (PC) [8]. The toughening mechanism has been attributed to extensive craze

3

formation (initiated at the rubbery component) in the plastic matrix leading to large energy absorption.

Mantovani et al. [12] previously observed that high additions of triblock styrene-butadiene-styrene copolymers improve the impact properties of PET/ABS blends. BASF studies [13] have shown that Styroflex 2G66 is an effective compatibilizer for ABS/HIPS/PP blends over a wide range of compositions. It is a modified SBS styrene-butadiene triblock copolymer in which the inner block is a randomized poly(styrene-*co*-butadiene). In this study we investigated the efficiency of this commercial product in the system ABS/HIPS/PETG. The ultimate purpose was to determine compositions that would give acceptable properties when recycling predominantly PETG-based commingled factory scrap.

Experimental

The polymers used in this study are listed in Table 1. The mixtures considered in the ternary system HIPS/ABS/PETG are indicated in Tables 1 to 4 and also schematically in Figures 1 to 5. The effect of Styroflex on blend properties was studied at the 7.5% add-on level. Each polymer and compound was dried at 80°C for at least 12 hours before processing. The test specimens for mechanical property testing were made on an injection-molding machine. Well-mixed blends were prepared by compounding on a Berstorff ZE 25 laboratory-scale, co-rotating twin-screw compounder or simply by direct injection molding of premixed polymer granules on a Demag Ergotech 100. Tensile properties were determined on a Zwick 1456 tensile tester using the ISO 527-2/1A/50 protocol, i.e. at a draw speed of 50 mm/min. Charpy impact tests were conducted according to ISO 179/1eA.

ABS/PETG binary blend samples were characterized by dynamic mechanical thermal analysis (DMTA) using a TA Instrument DMA 2980 under nitrogen atmosphere in the temperature range of −120 to 150°C. The measurements were made in the bending mode (double cantilever configuration) at a frequency of 1 Hz. Specimen dimensions were 35 mm x 10 mm x 4 mm. The temperature was scanned at a rate of 3 K/min. The measured data are presented as plots of the storage modulus E' and tan δ at 1 Hz.

Results and discussion

Mechanical properties. Tensile properties of blends prepared using the compounding route are shown in Figures 1 to 3 and listed in Table 2. Perusal of the data shows that the tensile yield strength, Young's modulus and elongation-at-break for Styroflex-free blends take on values that are intermediate to lower than those of the pure polymers. The tensile moduli of the blends agreed to within 1% with the values expected from the linear blending rule:

$$E(x_1, x_2, x_3) \; = \; E_1 x_1 + \; E_2 x_2 + \; E_3 x_3 \qquad\qquad (1)$$

Here $E(x_1, x_2, x_3)$ is the Young's modulus of the blend; E_i component i and x_i is the mass fraction of component i. Adding Styroflex caused a slight lowering of the modulus values.

The tensile strengths at yield (σ_Y) and the elongations-at-break (ε_B) of the blends are lower than expected from the linear blending rule by as much as 9% and 115% (for the 1:1 HIPS/ABS blend) respectively. Adding 7% Styroflex causes a further lowering of tensile strengths but leads to a significant increase in ε_B except for the 1:1:1 blend. The elongation-at break shows a complicated variation with composition. However, it was possible to correlate the tensile strength data using the second order Scheffé model:

$$\sigma_Y(x_1, x_2, x_3) = \sigma_{11} x_1^2 + \sigma_{22} x_2^2 \; + \; \sigma_{33} x_3^2 + 2\sigma_{12} x_1 x_2 + 2\sigma_{13} x_1 x_3 + 2\sigma_{23} x_2 x_3 \qquad (2)$$

Here $\sigma_Y(x_1,x_2,x_3)$ is the yield stress. It was equivalent to the maximum tensile stress for all the samples. The σ_{ii} represent the corresponding values for the neat polymer i and the σ_{ij} are adjustable model parameters. Relative least squares data reduction yielded $\sigma_{12} = 27.54$; $\sigma_{13} = 30.83$ and $\sigma_{23} = 41.18$ MPa.

Notched Charpy impact data is presented in Tables 3 and 4 as well as in Figures 4 to 6. Table 3 shows that, compared to the directly molded samples, compounding of mixtures actually leads to a small decrease of about 10% in notched impact strength. However, this is about the same magnitude as the standard deviation of the impact values. Table 4 reveals that, except for HIPS/PETG combination, all binary blends have good impact properties. In fact, ABS and PETG mixtures show much improved impact properties (See Figure 4 & 5) [7]. Note that Styroflex addition has little effect on the poor properties of the compounded HIPS/PETG binary mixture.

It is clear from Figure 5 that the notched Charpy data for the ABS/PETG blends shows highly nonlinear behavior. The results for compounded Styroflex-free blends were fitted using a novel mixture model [14]. The response surface is shown in Figure 6. Note that even low levels of HIPS contamination cause a precipitous loss of impact properties in ABS/PETG blends. Figure 4 shows that this deterioration is somewhat ameliorated by the presence of Styroflex.

Another feature of the data in Figure 4 is the higher variability of the notched Charpy results obtained when Styroflex is present. The data were subjected to a detailed statistical analysis [14]. It revealed that the positive effect of Styroflex observed in this study for blends containing ABS is statistically significant. The key conclusion of the mechanical property

evaluations is that the excellent notched impact properties shown by the ABS/PETG blend can only be maintained when contamination with HIPS is precluded.

Dynamic mechanical analysis (DMA). The ABS/PETG binary blends were subjected to DMA testing owing to the interesting notched impact results that were obtained. Fig. 7 presents the variation of the storage modulus (G′) with temperature and blend composition at a frequency of 1 Hz. In the neat polymers three distinct regions can be observed for G': (i) glassy high modulus region where the segmental mobility is restricted (ii) the transition zone where a substantial decrease in the G′ values with increase of temperature and (iii) the flow region where a drastic decrease in modulus with temperature is observed. In the latter region the molecular mobility sets in and the molecules slip past each other.

At very low temperature the polymers are in the glassy state and the effect of temperature is minor. In the region of a glass transition temperature the modulus shows a sudden drop to a lower plateau value. Below Tg, both virgin polymers have comparable E' values. The E' of neat PETG gradually decreases with increase of temperature, and drops rapidly above 74°C where it undergoes the transition from the glassy to the rubbery state.

The three-step nature of the G' curves behavior is typical for immiscible blends and it thus indicates incompatibility of the unmodified ABS/PETG blends. The dynamic modulus curves present three zones of abrupt modulus drops, which correspond to the respective relaxations in the polymer matrix. It is interesting to note that the storage modulus value is slightly lower for all the blends compared to the neat PETG, at temperature range up to 60°C. However, at temperatures above 74°C, while the dynamic modulus values of the neat polyester sample is found to decrease considerably, the modulus values of the blends are found to remain a good

deal higher. At the transition zone around (69°C), there is a sudden decrease in modulus with temperature. The decrease in value of E′ with increase in the concentration of PETG is more pronounced at high temperatures. Thus addition of ABS to PETG enhances the high temperature modulus of the blends. The modulus 'hump' beyond 130°C is assigned to recrystallization of PETG as reported by Cook et al. [7].

The dynamic mechanical spectra of the blends show transitions in the modulus curves and maxima in the tan δ curves which correspond to the glass transitions for the SAN and PETG phases of the parent polymers and therefore have a three-phase morphology. The Tg of the polymers was taken as the temperature corresponding to the maxima in tan δ. The values, obtained for the blends, are fairly similar or close to those of the original polymers. This indicates that the phase compositions were not much affected by the blending process. The relaxation temperature of PETG is indicated at about 84°C and for the SAN component of ABS around 114°C. A low temperature relaxation centered around -84°C, due to the polybutadiene (PBD) segments of ABS, is also observed. The ABS used in this work is a graft copolymer containing 38 mass % nitrile rubber particles with a butadiene/acrylonitrile ratio of 94:6 mass percent. The styrene acrylonitrile copolymer has a styrene/acrylonitrile ratio of 73:27. The difference in Tg is due to the grafting level [21]. Pure ABS shows a secondary relaxation that appears as a shoulder in the low temperature side of the main alpha relaxation at 111°C. A low temperature relaxation (β) around -74°C is also detected for PETG. The ABS/PETG blend is complicated by the close proximity of secondary relaxations of PBD segments of ABS around -84°C and the low temperature relaxation (β) around -74°C for PETG.

As the fraction of PETG is increased in the blend, the tan δ_{max} due to the PETG glass transition increases while the damping due to the ABS component is reduced in magnitude. The position of the PBD relaxation peak is constant, but for PETG rich blends, the amplitude of this peak is smaller, and finally it almost disappears for the systems with the highest PETG ratio. The blends do not show a regular trend in transition temperature values with change in composition. However, the DMA curves clearly reveal that there are no favorable interactions between ABS and PETG, as their transition peaks (Tg of PETG nearly 84°C, Tg of ABS around 114°C) show no shift in position with changes in the blend polymer ratio.

Phase morphology. A separate communication [15] presents scanning electron microscopy results obtained for neat and etched fracture surfaces of different blend samples. This investigation confirmed the essential multi-phase morphology of the ABS/PETG blends. Blend compositions close to the composition limits possess the typical droplets-in-matrix morphology in which the minor blend component exists as dispersed domains in the matrix of the major blend component. Blend of ABS dispersed in PETG, showed much larger particle sizes than the blends of PETG dispersed in ABS. This asymmetric behavior can be explained by a lower melt viscosity of the PETG phase as compared to the ABS matrix phase. As a result, the equilibrium between domain break-up and coalescence is shifted in the direction of coalescence when ABS is dispersed in PETG. Similar findings on increased particle size for ABS dispersed phase have been reported for ABS/PC blends [16]. The compatibilization of ABS/PETG blends by the triblock copolymer based on styrene and butadiene (SBS) was also confirmed by the SEM studies [15]. It was found that adding as little as 1 % SBS significantly reduced the domain size of the dispersed phase in the 70/30 ABS/PETG blends.

As mentioned, the best impact properties were obtained for a blend with a composition of ca. 50/50 ABS/PETG. Blend compositions near this value exhibit interpenetrating phase continuity the two major domains, SAN and amorphous PETG [15]. These observations are in contrast with polycarbonate/ABS blends which show a maximum in notched Izod impact strength at a weight ratio of ca. 80/20 [16]. However, they do mirror those made for corresponding ABS/PET blends [7]. Noteworthy is that impact properties are best when the PET phase is in an amorphous state (obtained by rapid quenching of melts) and that they deteriorated on ageing as the PET phase crystallizes [8]. The intrinsic amorphous nature of PETG precludes crystallization [1]. Examination of the fracture surfaces revealed considerable plastic deformation of both of the interpenetrating phases in the ABS/PET [7] and also the ABS/PETG blends [Joseph 2008]. High impact properties are usually associated with morphologies able to dissipate mechanical energy by large-scale plastic deformation, e.g. by shear yielding or crazing [Kinloch 1983]. Unfortunately just as is the case for ABS/PET blends [7], the actual mechanisms responsible for the improved notched impact behavior shown by the present blend are not yet understood and must await more detailed investigations.

Conclusion

The tensile properties of ABS/HIPS/PETG blends are of similar magnitude as the parent polymers. ABS/HIPS blends exhibit only mechanical compatibility. Impact properties of HIPS/PETG blends are lower than the parent polymers. ABS/PETG blends show significant synergy with respect to notched Charpy impact properties. Addition of a modified SBS compatibilizer (Styroflex 2G66) to such compounded blends only provides a marginal improvement in impact properties. However, it provides statistically significant improvements in the other binary blends as well as the ternary blends. Nevertheless, even small additions of

HIPS to ABS/PETG cause a precipitous drop in notched impact properties. Thus, property improvements can be envisaged when recycling mixed ABS and PETG factory scrap but care should be taken to avoid contamination with HIPS. This implies that, whenever possible, PETG extrusion runs should be sandwiched between ABS production runs.

Dynamic mechanical analysis and scanning electron microscopy (SEM) reveal the essentially phase separated nature of binary ABS/PETG blends. However, SEM provides evidence for a co-continuous phase structure for the two main glassy phases. While the reasons for the improved impact properties of the ABS/PETG blends are not yet understood, it is likely that the nature of the phase morphologies plays an important role.

Acknowledgement

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List of Tables

Table 1: Polymers and their suppliers

Item	Description / Grade	Density kg/m^3	MFR dg/min	Supplier
ABS	Terluran HI 10	1030	5,5 [a]	BASF
HIPS	486	1028	4 [b]	BASF
PETG	Spectar 14471	1270	-	Eastman
Styroflex	2G66	998	13 [b]	BASF

[a] 220°C/10 kg; [b] 200°C/5 kg

Table 2: Tensile properties of ABS/HIPS/PETG blends prepared by compounding

Composition, mass fraction			E, GPa		σ_M, MPa		ε_B, %	
HIPS	ABS	PETG	Neat	Sty*	Neat	Sty*	Neat	Sty*
1,000	0,000	0,000	1,88	-	23,0	-	59	-
0,000	1,000	0,000	1,81	-	35,2	-	13	-
0,000	0,000	1,000	2,02	-	50,4	-	73	-
0,500	0,500	0,000	1,85	1,67	28,3	27,4	45	76
0,500	0,000	0,500	1,96	1,80	33,8	31,9	35	38
0,000	0,500	0,500	1,93	1,73	42,0	37,0	20	26
0,333	0,333	0,333	1,92	1,73	34,1	32,5	38	34
		Styroflex	0,06	-	4,2	-	421	-

* With 7% Styroflex

Table 3: Effect of compounding on Charpy impact properties

	Composition			A kJ/m^2	B kJ/m^2	Δ, %
	HIPS	ABS	PETG			
Without Styroflex	1.000	0.000	0.000	-	13.9	-
	0.000	1.000	0.000	-	37.3	-
	0.000	0.000	1.000	-	8.88	-
	0.500	0.500	0.000	15.1	16.5	-9.2
	0.500	0.000	0.500	5.60	5.35	4.5
	0.000	0.500	0.500	67.9	69.4	-2.2
	0.333	0.333	0.333	9.47	9.60	-1.4
With 7% Styroflex	0.500	0.500	0.000	21.3	23.2	-9.0
	0.500	0.000	0.500	6.06	5.68	6.3
	0.000	0.500	0.500	69.9	82,0	-17.2
	0.333	0.333	0.333	25.6	23.3	9.2

A: Compounded samples; B: Directly molded mixtures; D: 100 (A - B)/A

Table 4: Notched Charpy impact properties of compounded ABS/HIPS/PETG blends

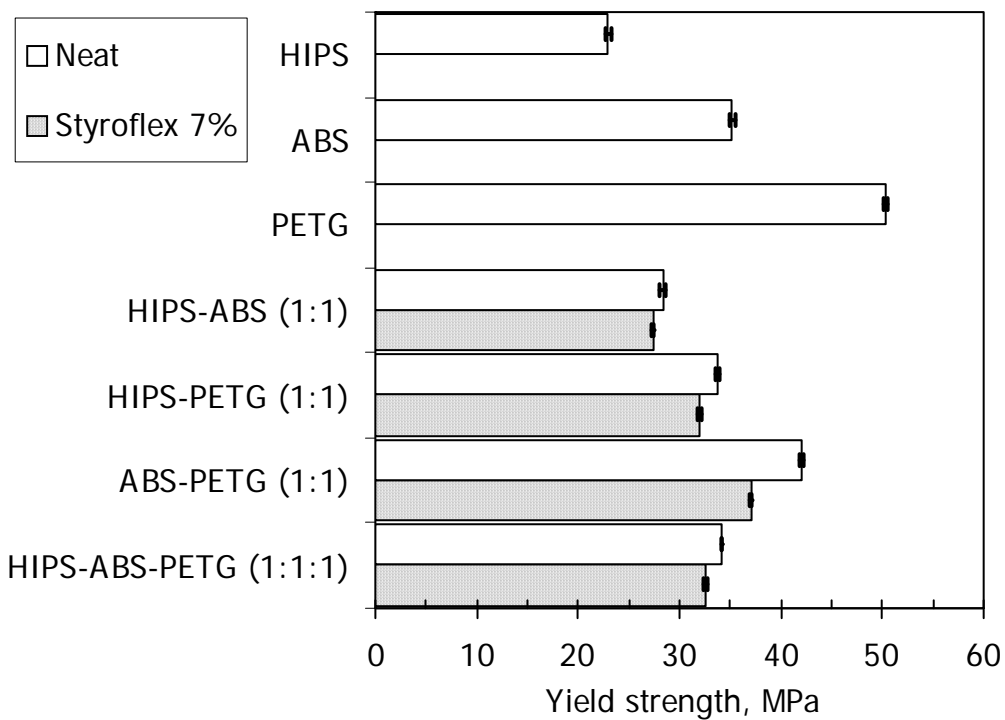| Composition			Notched Charpy	Std Dev
HIPS	ABS	PETG	kJ/m^2	kJ/m^2
1.000	0.000	0.000	13.9	1.1
0.000	1.000	0.000	37.3	0.6
0.000	0.000	1.000	8.9	0.7
0.500	0.500	0.000	15.1	0.6
0.500	0.000	0.500	5.6	0.3
0.000	0.500	0.500	67.9	1.3
0.333	0.333	0.333	9.5	0.9
0.750	0.250	0.000	15.3	0.6
0.250	0.750	0.000	17.8	0.5
0.750	0.000	0.250	8.4	0.4
0.250	0.000	0.750	4.0	0.7
0.000	0.750	0.250	46.3	1.9
0.000	0.250	0.750	16.3	1.7
0.667	0.167	0.167	11.1	1.3
0.167	0.667	0.167	17.0	2.1
0.167	0.167	0.667	7.2	1.0
0.000	0.900	0.100	33.3	1.0

List of Figures

Figure 1: Tensile yield strength of compounded samples. Error bars indicate one standard deviation in measured values.
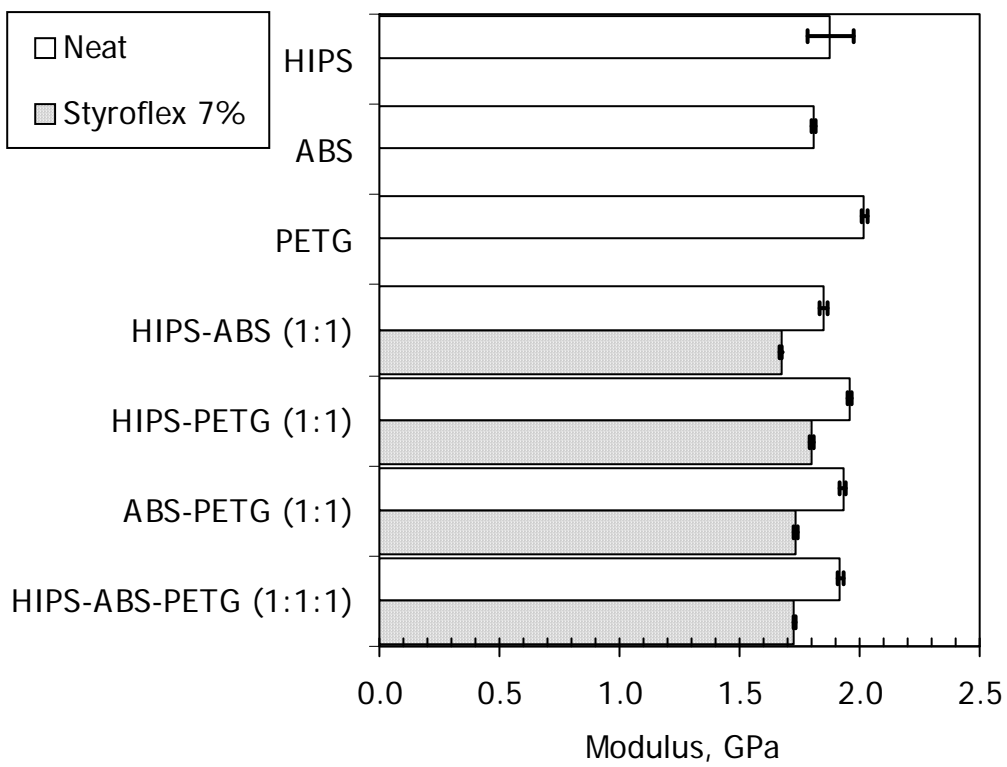
Figure 2: Tensile moduli of compounded samples. Error bars indicate one standard deviation in measured values.
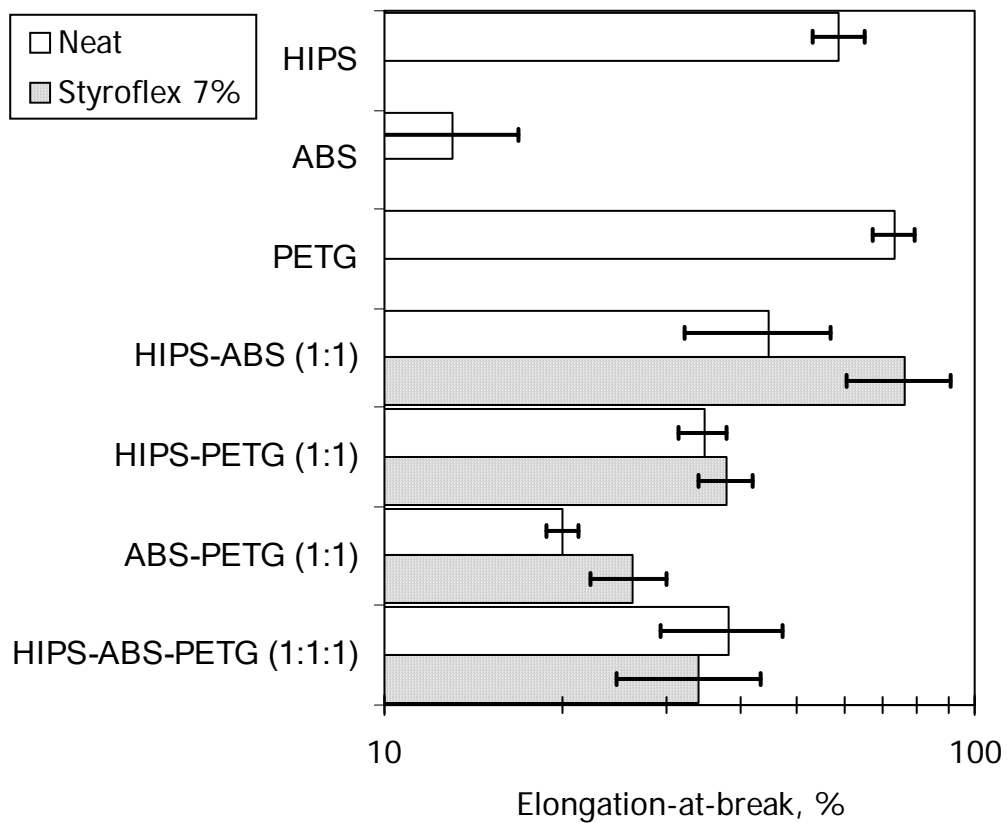
Figure 3: Elongation at break for compounded samples. Error bars indicate one standard deviation in measured values.
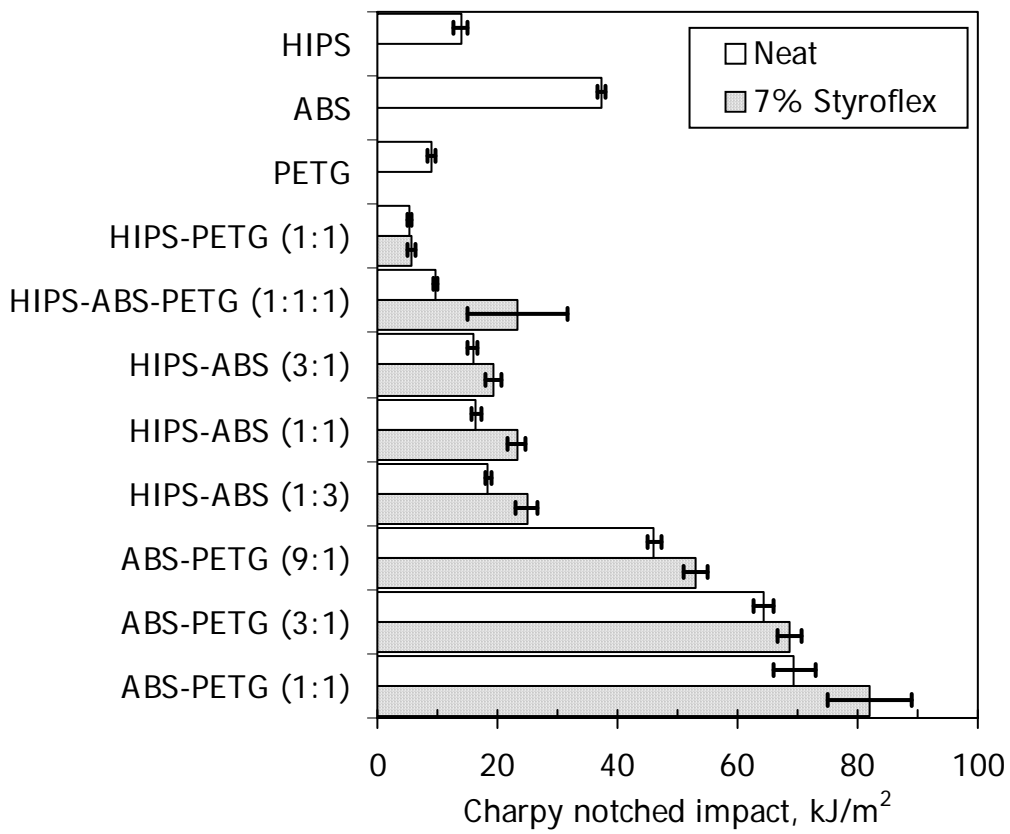
Figure 4: Notched Charpy values for directly molded samples. Error bars indicate one standard deviation in measured values.
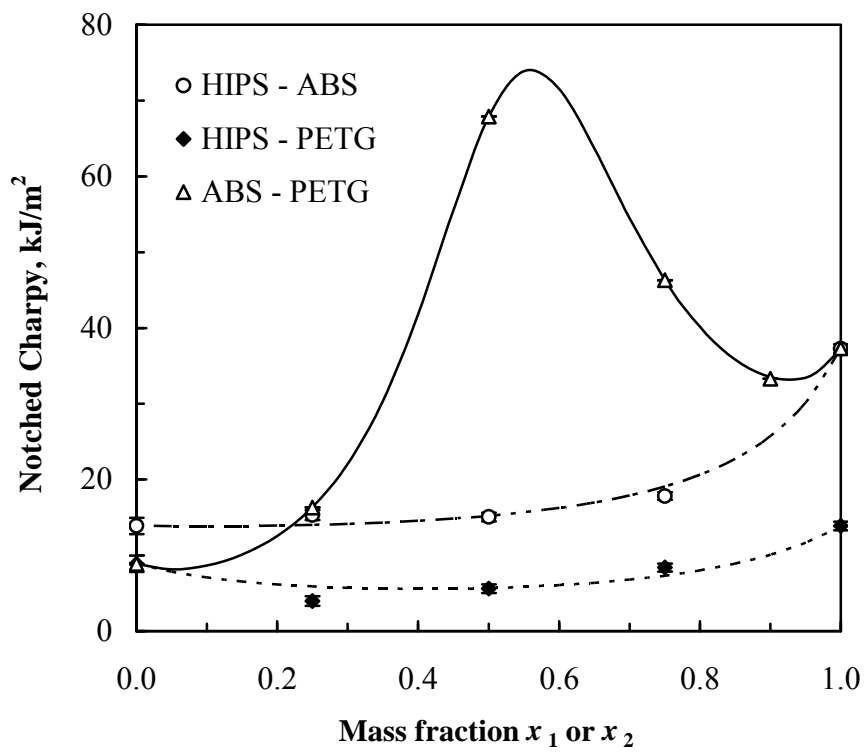
Figure 5: Notched impact strength of binary blends.

Figure 6: Response surface for notched impact strengths of compounded, Styroflex-free samples.

Figure 7: Storage modulus and tan δ for ABS/PETG blends.

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Figure 2: Tensile Moduli of compounded samples. Error bars indicate one standard deviation in measured values.

Figure 3: Elongation at break for compounded samples. Error bars indicate one standard deviation in measured values.

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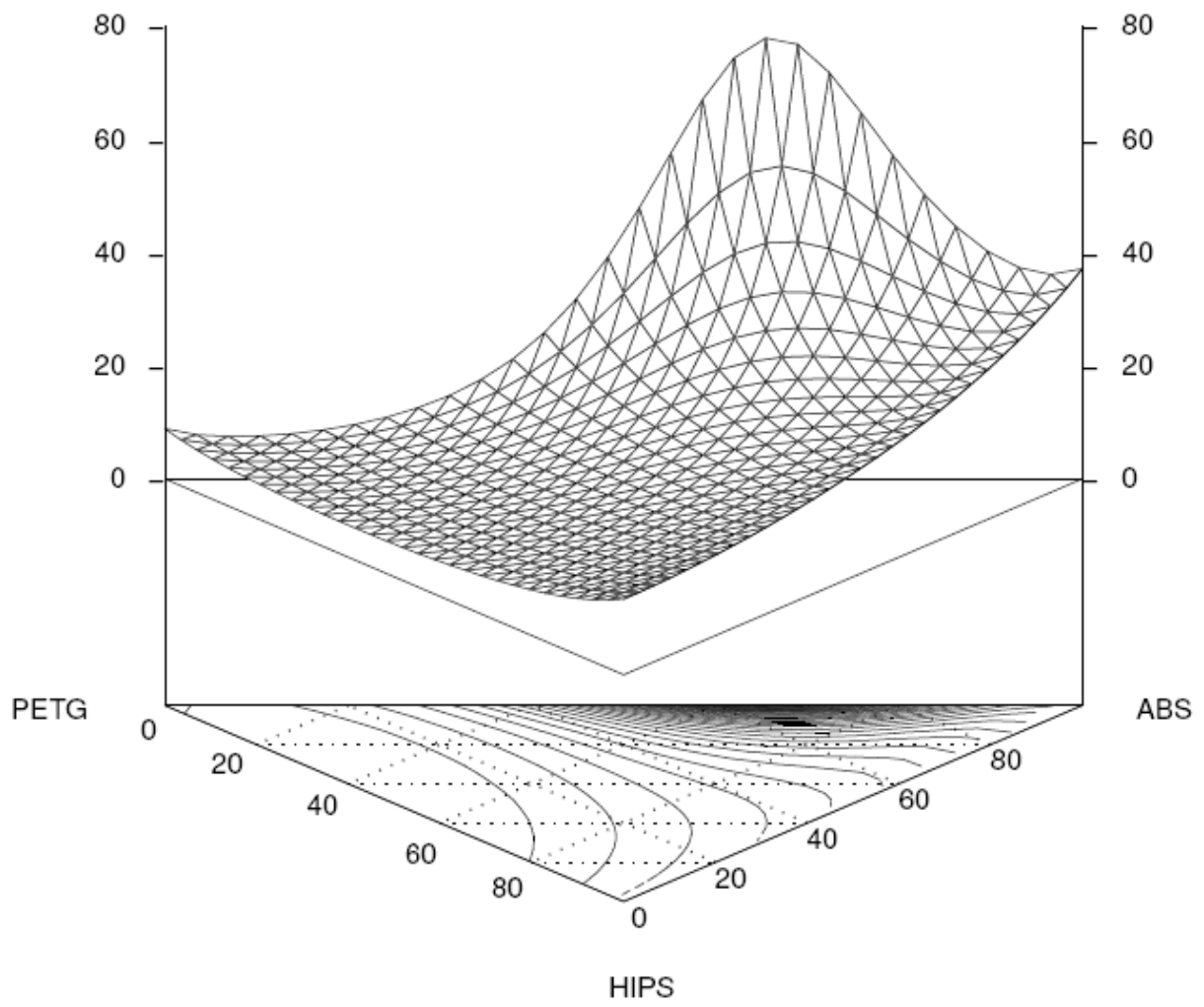
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Figure 6: Response surface for notched impact strengths of compounded, Styroflex-free samples.
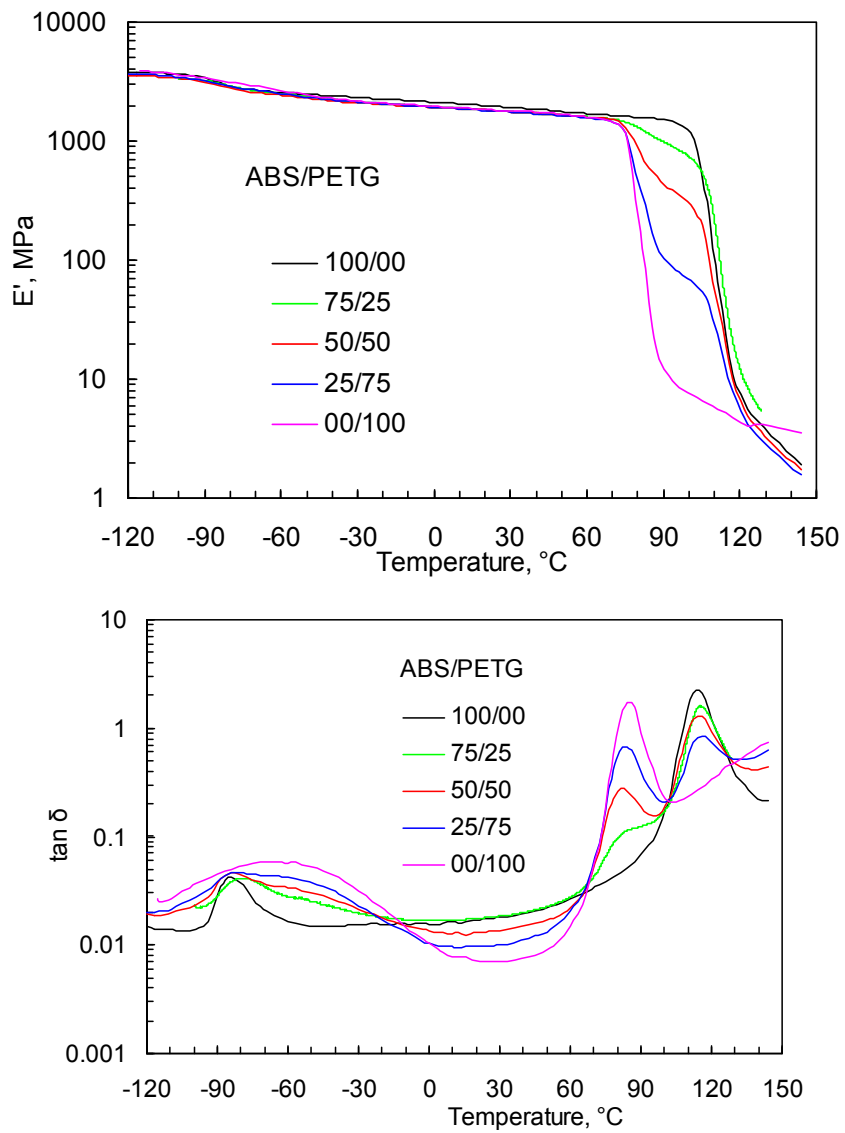
Figure 7: Storage modulus E' and tan δ for ABS/PETG blends measured at 1 Hz.