Mechanical and Dielectric Breakdown Properties of PBT/ TPE, PBT/PBT/PET, and PBT/Antioxidant Blends

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ABSTRACT: To improve the thermal aging flexibility of poly(butylene terephthalate) (PBT), PBT was melt-blended with three type thermoplastic elastomer [poly ether-ester type (TPE1), polyester-ester type (TPE2), and poly(buthylene 2,6-naphthalate)/poly(tetramethylene glycol) block copolymer type (TPE3)], PBT/poly(ethylene terephthalate), (PET) alloy (Alloy), and phosphate type antioxidant (T1). The content of the three type TPEs and Alloy was fixed at 20 parts per 100 g of PBT. The morphology and thermal behavior of these blends have been investigated with scanning electron microscopy (SEM), differential scanning calorimetry (DSC), and thermogravimetry (TG). In the case of PBT/Alloy-20 and PBT/TPE3-20 blends show clean fractured surface, whereas for PBT/TPE1-20 and PBT/TPE2-20 blends, the elongated pieces or fiber can be seen abundantly which indicates a good compatibility. TG traces show a significant shift of the weight loss toward higher

INTRODUCTION

Poly(buthylene terephthalate) (PBT) is linear aromatic polyester that have been commercially available engineering plastics. It has high mechanical strength, excellent processing characteristics, and electrical properties, and good resistance to chemical attack for a broad range of applications.^{1–3} These characteristics make it highly suitable for uses in films, textiles, bottles, molding components, and fibers because of its fast crystallization rate and high elasticity compared with those of poly(ethylene terephthalate) (PET) while exhibiting desirable physical and mechanical properties.⁴

Recent developments resulted in polymer blends of PBT with PET,^{5–9} nylon 6,¹⁰ polycarbonate (PC),^{11,12} and polytetramethyleneglycol¹³ to improve impact resistance at low temperatures and other mechanical properties. PET/PBT blends have high electrical insulation properties, thermal resistance, and good mechanical properties.^{7,8} They have a lower glass temperature¹⁴ and a lower melting temperature $(T_m)^{5,15}$ compare with PET in spite of the same crystemperature for PBT/Alloy-20, whereas PBT/TPE1–20, PBT/TPE2–20 and PBT/TPE3–20 blend decrease in thermal stability than PBT. To investigate the applicability for insulation material, the prepared blend samples were extruded an electric wire and flexibility and electric breakdown voltage (BDV) of wire after thermal aging were studied. For PBT/TPE1–20 and PBT/TPE2–20 blends did not show any cracks after flexibility test at 130°C for 6 h and 225°C for 30 min. In contrast PBT, PBT/Alloy-20, PBT/TPE3–20, and PBT/T1–1 showed a partial crack in the insulation after flexibility test at 130°C for 6 h although its good flexibility at 225°C for 30 min. © 2009 Wiley Periodicals, Inc. J Appl Polym Sci 114: 3008–3015, 2009

Key words: poly(buthylene terephthalate); polymer blend; miscibility; morphology

tallinity. Escala and Stein⁶ reported that the PET/ PBT blends showed a single, composition-dependent glass transition temperature (T_g) at all compositions suggesting that PET and PBT were miscible in the amorphous state. Similar results were also reported by others.^{7,8} The low content of copolymer in the blend which is formed above T_m provides good deformability without the necking phenomena which is characteristic for PET.

Many custom extruders overcome the extruding problems such like tight-tolerance, small-diameter, and thin-walled product by using high draw-down ratios. This technique significantly improves dimensional tolerances, increases line speed, and makes tooling much easier to fabricate. However, running a high draw-down ratio also imparts molecular orientation, residual stress, and strain in the finished products during high speed extrusion process. Among them, molecular orientation can significantly increase the tensile strength and reduce the elongation of the polymer materials in the machine direction. Finally this made the preferred sites for cracking, crazing, and shrinkage. The crystallization of PBT is much faster other polyesters and changes the crystallinity by annealing, mechanical deformation, and relaxation. These crystallization properties of PBT make a drawback for thin layer extruding. It is well known that the material properties of polyester

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can be modified by blending in the melt with other polycondensates.^{5,6,14–17} Park¹⁶ demonstrated that 20 parts polyester type thermoplastic elastomer (TPE-E) containing PBT/PET/TPE-E blends exhibited good breakdown voltage (BDV) and tensile properties. It is also reported that PBT/TPE-E/styrene-ethylenebutadiene-styrene copolymer (70/20/10 wt %) blends exhibited good processibility, good thermal, and electrical insulation properties.¹⁷ In this study, PBT was melt-blended with three type thermoplastic elastomer (TPE), PBT/PET alloy (Alloy), and antioxidant (T1) to improve the thermal aging flexibility. The prepared specimens were subjected to thermal and mechanical tests, and the fractured surface was observed using scanning electron microscopy (SEM). The prepared blends were also extruded an electric wire and its thermal aging flexibility and dielectric breakdown voltage (BDV) after thermal aging were investigated.

EXPERIMENTAL

Materials

PBT [TRIBIT 1500, Samyang, Jeonju, Korea], PET/ PBT alloy resin [Alloy, KP513 (PBT/PET = 30/70 wt %), Kolon Industries, Kwacheon, Korea], nylon 66 [KN333HS, Kolon Industries, Kwacheon, Korea], polyester-ester type TPE [TPE1, UM551; polyetherester type TPE [TPE2, EM740, DSM Engineering Plastics, Seoul, Korea], poly(buthylene 2,6-naphthalate) (PBN)/poly(tetramethylene glycol) block copolymer (PBN-b-PTMG) type TPE [TPE3, EN5000, Plastech Service, Seoul, Korea], PBN (Teijin Chemicals, Tokyo, Japan), and antioxidant [T1, bis(2,4-dicumylphenyl) pentaerythritol diphosphite, Doverphous S-9228, Plastech Service, Seoul, Korea] were used as received. All resins were predried in a convection oven for at least 12 h at 80°C to remove any moisture from the pellets before processing.

Characteristics of the Polymer Materials

Material	Density (g/cm ³)	Hardness (°C)	T_m (°C)	Melt flow index (g/10 min)
PBT	1.31	112	221	4 (230°C/2.16 kg)
Alloy	1.40	116	251	5 (265°C/2.16 kg)
TPE1	1.26	41	200	16 (230°C/2.16 kg)
TPE2	1.27	71	218	5 (230°C/2.16 kg)
TPE3	1.24	75	226	11 (235°C/2.16 kg)
PBN	1.24	93	242	12 (230°C/2.16 kg)
Nylon 66	1.14	118	255	9 (270°C/2.16 kg)

Instrumentation

Scanning electron microscopy (SEM) observations of the specimens were performed on a Hitachi S-4200 model. The fractured surfaces of the blends were prepared by using cryogenic fracturing in liquid nitrogen (N_2) followed by a coating with gold in an SPI sputter coater. The morphology was determined using an accelerating voltage of 15 kV.

Thermal properties of the blends were determined by DSC (Perkin Elmer DSC 7, Norwalk, CT). Thermal history of the blends was removed by scanning to 300°C with the heating rate of 20°C/min. After cooling down the specimen at the rate of -5° C/min to room temperature, it was reheated at 20°C–300°C with the heating rate of 20°C/min and the DSC curves were obtained.

Cracks after flexibility test of extruded wire were measured a video microscope (Inf-500TM CCD Video Microscope, Moritex Corp., Japan) with a PC that contained video capture software (i-Solution Lite, Image and Microscope Technology, Korea).

Thermal stability of samples was determined by Thermogravimetry (TGA Q50, TA instrument). The TG curves were obtained under an N_2 atmosphere at a flow rate of 4 mL/min and a scanning from 20 to 800°C with the heating rate of 20°C/min.

Hardness of the specimens was measured with a Rockwell hardness tester and the readings (R scale) were averaged. Five locations were measured for each specimen and surface.

Compounding and extruding of blends

PBT/TPE [20 g of TPE1, TPE2, or TPE3/100 g of PBT), PBT/Alloy (20 g of Alloy/100 g of PBT) and PBT/T1 (1 g of T1/100 g of PBT) were melt-blended in a twin-screw extruder (Twin 60ϕ , Theysohn, Germany) using a temperature setting of Rear zone: 235°C, Second zone: 250°C, Forward zone: 250°C, Adapter: 265°C, and Die: 270°C and a mixing rate of 10 kg/h, and then cut into pellet form by a pelletizer. Blend samples were dried in a hopper equipped with a dehumidifying dryer at least 24 h at 100°C to remove any moisture from the pellets before extruding.

The electric wire was composed of conductor and 1st, 2nd, and 3rd insulation layer. The 1st insulation layer was applied to the annealed copper conductor by extrusion with prepared blends (thickness: $30 \ \mu$ m). The 2nd layer was applied to 1st extruded layer by extrusion with same blends and thickness of 1st layer. The 3rd layer was applied to 2nd extruded layer by extrusion with nylon 66 (thickness: $30 \ \mu$ m). All insulation layers were extruded (temperature setting of Rear zone: 235° C, Second zone: 255° C, Forward zone: 260° C, Adapter: 265° C, and Die: 270° C) at a speed of 200 rpm using an extruder (ϕ 20 Extruder, Flontec, Seoul, Korea) equipped with a 1 : 3 compression ratio screw. Extruded samples were quenched by passage through a water-cooling zone at a rate of 200 m/min.

Sample code	DSC thermal properties						
	1st scan	2nd	scan		$-\Delta H_c (J/g)$		
	T_{m1} (°C)	T_{m21} (°C)	$T_{m22} (J/g)$	T_c (°C)			
PBT	221	214	222	195	74.7		
PBT/TPE1-20	221	209	217	192	60.0		
PBT/TPE2-20	221	209	220	187	68.1		
PBT/TPE3-20	222	212	221	196	73.8		
PBT/Alloy-20	222	213	223	195	73.9		
PBT/T1-1	220	212	220	194	72.4		
PBN	242	232	240	215	65.7		
Alloy	251	_	230	148	40.7		
TPEÍ	200	_	198	182	7.1		
TPE2	218	_	219	193	65.7		
TPE3	226	218	226	202	50.3		

 TABLE I

 DSC Thermal Properties of the PBT/TPE, PBT/Alloy and PBT/T1 Blends

Tensile test procedure

The polymer pellets were subsequently hot pressed to sheets of uniform thickness (around 0.3 mm) using a plate press at 265°C for 5 min under ca. 5 atm and quickly immersed into water. The sheet thus formed was free from any distortion problems. Dumb-bell specimens for mechanical tests were prepared in accordance with ASTM D638 specification. The test specimens were preconditioned to 20% relative humidity and $20^\circ C~\pm~1^\circ C$ to standardize test conditions. Tensile properties of samples were determined with a universal test machine (UTM, Model DECMC00, Dawha test machine, Korea) at a cross head speed of 250 mm/min. The mean value of at least five specimens of each sample was taken, although specimens that broke in an unusual manner were disregarded.

Extrusion weight (g/10 min) of blends

Extrusion weight (g) of the blend samples was measured for 10 min using an extruder (ϕ 20 Extruder, Flontec, Korea) equipped with resin bypass (8 mm inner diameter and 10 mm length orifice). The extruder was preheated from room temperature to 265°C, and then the pellets of blend samples were fed in to the extruder and allowed to melt. The polymer stays in the molten form inside the extruder for ~ 10 min and then the molten polymer is vented at a screw rotate speed of 200 rpm out from the exit valve.

Thermal aging and flexibility test

A straight piece of 300 mm wire was wound for 10 continuous carefully and adjacent turns around a polished mandrel of 40 mm diameter. The specimen is placed into an oven with forced air circulation at 130°C for 6 h or at 225°C for 30 min. After removal from the oven, the specimen was allowed to cool to

room temperature. After cooling the thermal aged wire was straightened and was examined for cracks under a video microscope.

Breakdown voltage measurement

The measurements of BDV were carried out at 60 Hz AC voltage by using a high voltage tester (SM-40PT08, Sungmin Instruments, Seoul, Korea) with a rate of 0.5 kV/s in air. The test sample consists of copper conductor (diameter of 0.4 mm) and outer insulator (insulation thickness of 0.9 mm). The voltage was applied between the conductor and aluminum foil wrapped intimate contact with the center 152 mm of the wire sample. Five samples are tested and the average voltage is calculated.

Abrasion test

Abrasion test was performed using an Abrasion Tester (Core Tech, Seoul, Korea). The test pieces were $20 \times 100 \times 0.3 \text{ mm}^3$ in size and were prepared by compression-molding at 265°C. The travel distance of specimen was 10 mm at a speed of 60 cycles/min, and the test load was 5 N. The abrasive that was used was silicon carbide abrasive paper (#120, AA-120; Daesung Abrasive, Seoul, Korea). The percentage of abrasivity was calculated based on the weight loss after the test.

RESULTS AND DISCUSSION

DSC thermal properties of the PBT/TPE, PBT/alloy and PBT/T1 blends

PBT/TPE, PBT/Alloy, and PBT/T1 blends were melt-mixed using twin-screw extruder and the results of DSC thermal properties are summarized in Table I. The abbreviation of the sample code in Table I, PBT/TPE1–20, for example, means that the



Figure 1 DSC thermograms of the PBT/TPE blends.

content of TPE1 in the PBT was 20 parts (20 g of TPE1/100g of PBT). The 1st scan DSC data of samples were collected by a heating at the rate of 20°C/ min from 20 to 300°C. T_m of 1st scan DSC thermogram of all blends show one peak and did not change compare with PBT. When PBT/TPE blends and PBN melt was slowly cooled at a rate of -5° C/ min and remelted, the one melting peak of 1st scan

DSC thermogram was divided two peaks (Fig. 1). It is well known that PBT exists in two crystalline structures, α and β form, and that the transition between two forms takes place reversibly by annealing, mechanical deformation, and relaxation. The structure of PBN is equivalent to that of PBT except that only the benzene ring is replaced by the naphthalene ring. PBN also has two crystal forms, A and B forms, and that four methylene groups in the B form is more extended than that of the A form as in the case of PBT. It can be also founded that T_m^{21} (lower melting peak) and T_m^{22} (higher melting peak) of PBT/TPE1-20 and PBT/TPE2-20 blends shift lower temperature slightly [Fig. 1(b)]. PBT/TPE1-20 and PBT/TPE2–20 showed decreased T_c and heat of crystallization (ΔH_c) than those of neat PBT whereas for PBT/TPE3-20, PBT/Alloy-20, and PBT/T1-1 did not change.

Morphology and mechanical properties of polymer blends

Figure 2 represents a fractured surface of extruded PBT/TPE-20, PBT/Alloy-20 and PBT/T1-1 blends. An immiscible polymer blend system generally has a microstructure with the phase separation between components.²⁰ In case of TPE and Alloy, antioxidant containing blends did not show any phase separation between components. It is noted that SEM image of the PBT/Alloy-20 and PBT/TPE3–20 shows clean fractured surface. In the micrographs taken on the surface of TPE1 and TPE2 containing specimen shows ripped structure which indicates a good compatibility and toughening effect. On the entire fractured surface, elongated pieces, or fiber can be seen abundantly during cryogenic fracturing in liquid N₂.

Table II summarized tensile properties, hardness, and abrasion weight loss of prepared blend samples. Tensile strength of PBT/TPE1-20 and PBT/TPE2-20 blends decreased whereas for elongation at break increased significantly compare with PBT. It is observed that an \sim 17 and 11% overall increase in the elongation at break such addition. Polymeric materials such as polyesters and polyethylene copolymers can be used as plasticizers.21,22 TPE1 and TPE2 behaved as a polymeric plasticizer to PBT and improved the flexibility and ductility of the blends. In contrast, elongation at break of PBT/ TPE3-20 and PBT/Alloy-20 was significantly decreased compare than PBT. Mechanical properties of the polymer blends are affected by several main factors, besides compatibility also by morphology resulting from blending conditions, change of molecular weight of components, super-molecular structure of components, conditions of preparation of sample for measurement, and others. In general, the addition of a rigid material to a polymer causes



(e) PBT-Alloy-20

(f) PBT-T1-1

Figure 2 SEM micrographs of the extruded PBT/TPE, PBT/Alloy and PBT/T1 blends.

	Tensile pr	operties			
Sample code	Tensile strength (MPa)	Elongation at break (%)	Hardness (R scale)	Abrasion weight loss (mg/cm ²)	
PBT	68 ± 3	497 ± 50	112	0.3	
PBT/TPE1-20	46 ± 8	583 ± 35	83	1.7	
PBT/TPE2-20	31 ± 10	550 ± 41	82	1.7	
PBT/TPE3-20	38 ± 8	17 ± 5	85	1.7	
PBT/Alloy-20	49 ± 9	288 ± 20	115	0.2	
PBT/T1-1	60 ± 5	447 ± 50	110	0.6	
PBN	61 ± 11	141 ± 22	93	0.1	
Alloy	32 ± 12	400 ± 45	116	0.3	
TPEÍ	24 ± 5	360 ± 35	45	1.0	
TPE2	37 ± 7	318 ± 25	71	0.3	
TPE3	54 + 9	685 ± 35	75	1.3	

 TABLE II

 Mechanical Properties of the PBT/TPE, PBT/Alloy and PBT/T1 Blends

TABLE III Extrusion Weight of PBT/TPE, PBT/Alloy and PBT/T1 Blends			
Sample code	Extrusion weight (g/10 min)		
PBT	100		
PBT/TPE1-20	104		
PBT/TPE2-20	105		
PBT/TPE3-20	106		
PBT/Alloy-20	95		
PBT/T1-1	100		
PBN	90		
Alloy	95		
TPEI	135		
TPE2	110		
TPE3	98		

considerable lowering of elongation at break. When the rigid PET and PBN unit was mixed with PBT, mobility of PBT is decreased and finally it is possible to reduce the elongation at break of blends. It is noted that TPE1, TPE2, or TPE3 was mixed with PBT, there was an increasing trend in abrasion weight loss with decreasing hardness compare with PBT. As abrasion resistance basically depends on the hardness of a material, most conventional abrasionresistant of polymer materials achieve their hardness by adding mineral filler.¹⁶ The hardness of TPE is smaller than that of PBT matrix so it has a significant reduce on abrasion resistance.

Extrusion weight and thermal stability of polymer blends

Polymer materials are most commonly processed in a viscous molten state, which allows them to be shaped and molded flow. For the thin film extrusion process, melt viscosity is an important property since it affects the flow of the molten polymer. Extrusion weight of prepared blend samples was measured at 265°C for 10 min, and the results are summarized in Table III. The extrusion weight of PBT/TPE blends increased minimum 4% than PBT due to the melt viscosity of the TPE is lower than that of PBT at the same temperature. Extrusion



Figure 3 TGA thermograms of the PBT/TPE and PBT/Alloy blends.

weight decreases when the Alloy blended with PBT because the mobility of the polymer molecules is more restricted as the melt is mixed with rigid PET moieties.

The investigation on the thermal stability was carried out using a TGA under N2 atmosphere. Some typical weight-loss curves as a function of temperature are shown in Figure 3. The addition of TPE resulted in a decrease in thermal stability than PBT and similarity thermal stability although it's different T_m . TGA curve of PBT is characterized by a very little char residue (7%), whereas that of PBT/TPE1-20 shows the highest char residue among all the samples under investigation (21%). As far as the blended TPE is concerned, the char residue appears to depend on T_m , regularly increasing as the T_m of TPE is increased. For PBT/Alloy-20 blend, the TG traces show a significant shift of the weight loss toward higher temperature, with stabilization about 10°C higher than PBT.

Flexibility and dielectric breakdown properties of polymer blends

Table III shows flexibility test and BDV results of extruded samples before and after thermal aging.

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Sample code	After extruded		After thermal aging at 130°C for 6 h		After thermal aging at 225°C for 30 min	
	Flexibility	BDV (kV)	Flexibility	BDV (kV)	Flexibility	BDV (kV)
PBT	No crack	13.3	Crack	0.0	No crack	9.9
PBT/TPE1-20	No crack	12.8	No crack	9.5	No crack	10.2
PBT/TPE2-20	No crack	13.0	No crack	9.2	No crack	10.4
PBT/TPE3-20	No crack	12.4	Crack	0.0	No crack	11.4
PBT/Alloy-20	No crack	13.2	Crack	0.0	No crack	9.8
PBT/T1	No crack	13.2	Crack	8.7	No crack	10.2
PBN	No crack	9.8	Crack	0.0	Crack	0.0

 TABLE IV

 Insulation Properties of the Extruded Wire Before and After Flexibility Test



(a) PBT



(b) PBT/Alloy-20



(c)PBT/TPE1-20

Figure 4 Crack phenomenon of the extruded wire samples after the flexibility test at 130°C for 6 h.

Dielectric breakdown causes a high-temperature arc to propagate through a dielectric medium, and this can lead to ignition of the medium or of surrounding objects. The BDV of extruded samples show similarity value. After the flexible test at 225°C for 30

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min, the BDV of extruded wire decreased significantly compare with unaged ones (Table IV). An approximately 8–32% overall decrease in the BDV from 9.9 to 11.4 kV was observed. The thermal aging is manifested in two ways by causing formation of polymer radicals²³ and as an increase in the number and size of micro-voids in the material.²⁴ In solid insulating materials, they usually create a weakened path within the material by creating permanent molecular or physical changes.

After flexibility test at 130°C for 6 h, PBT, PBT/ TPE3-20, PBT/T1-1, and PBN shows insulation crack phenomenon (Fig. 4). Thermal aging plays a major role due to the degradation of the polymers. T_1 is high molecular weight, low volatile, and high phosphorous content antioxidant and used for outstanding processing protection against discoloration and thermal degradation. Thus, it is suggests that the crack of PBT/T1-1 after flexibility test did not originate from thermal degradation of PBT. In contrast PBN is higher tensile strength and elongation at break than PBT/TPE3-20, the crack of insulation was happened after flexibility test at 225°C for 30 min. PBN has a higher $T_{m\nu}$ T_c (Fig. 1) and a more rapid rate of crystallization compared with PBT. PBN and PBT can undergo crystallization during cooling from the melt, as well as by isothermal treat-ment below the melting point.²⁵⁻²⁷ Extruded insulation layer was quenched by passing through a water-cooling zone at a rate of 200 m/min and was not fully crystallized. During the thermal aging for 6 h at 130°C, PBT and PEN with more amorphous phases oriented undergo larger internal rearrangement of microstructures. Thermally-induced crystallization of polymer chains promotes intermolecular forces, thus increasing the rigidity and brittleness of the polymer. Finally, this effectively reduces the flexibility of thin extruded layer causing the crack phenomenon. In sharp contrast, the PBT/TPE1-20 and PBT/TPE2-20 blends did not show any cracks after flexibility test at 130°C for 6 h. This could be attributed to a dilution effect with the TPE1 and TPE2 acting as polymeric plasticizers.

CONCLUSIONS

PBT was melt-blended with polyether-ester type TPE (TPE1), polyester-ester type TPE (TPE2), PBN-*b*-PTMG type TPE (TPE3), PBT/PET (Alloy), and (2,4-dicumylphenyl) pentaerythritol diphosphite (T1) as antioxidant using a twin-screw extruder. The content of the three type TPEs and Alloy was fixed at 20 parts per 100 g of PBT. The prepared blends were subjected to thermal and mechanical tests. The prepared blends melt was slowly cooled at a rate of -5° C/min and remelted, the one melting peak of 1st scan DSC thermogram was divided two peaks due

to change of crystalline structures of PBT. TGA measurement shows that the addition of 20 parts TPE1, TPE2, or TPE3 resulted in a decrease in thermal stability than PBT and similarity thermal stability.

To investigate the insulation flexibility and electric breakdown properties of blends, PBT/TPE1-20, PBT/TPE2–20, PBT/TPE3–20, PBT/Alloy-20, and PBT/T1-1 were extruded on the conductor and quenched at a rate of 200 m/min. After the flexible test at 225°C for 30 min, the BDV of all wires decreased significantly while the insulation crack of wires was not happened. After flexible test at 130°C for 6 h, PBT, PBT/TPE3-20, PBT/Alloy-20, and PBT/T1-1 show insulation crack phenomenon. Thermally induced molecular orientation reduces the flexibility of PBT phase and causes the cracks in the insulation. In contrast PBT/TPE1-20 and PBT/ TPE2–20 blends did not show any cracks after flexibility test at 130°C for 6 h. In addition, tensile strength and hardness of PBT/TPE1-20 and PBT/ TPE2-20 blends decreased, whereas elongation at break, extrusion weight, and abrasion weight loss increased compare with PBT. This implies that incorporation of TPE1 and TPE2 acts like polymeric plasticizer, preventing the resin from becoming brittle and losing flexibility. Since these blends have good dielectric breakdown property, insulation flexibility, and extrusion processibility, they can be used for industrial applications such like thin film extrusion and coating process.

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