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# Blended Polypropylene/Polyethylene Terephthalate Fibres: Crystallisation Behaviour of Polypropylene and Mechanical Properties

## Abstract

Polyethylene terephthalate (PET) and the compatibilisers ethylenedistearamide (EDSA) and ethylenevinylacetate (EVAc) are used for the modification of PP fibres that are aimed at improving the dyeability by dyeing fibres in a bath. This causes changes in the melting and crystallisation process that affect the creation of the morphological structure in blended PP/PET fibres as well as the content of the crystalline phase of the PP matrix. The thermal properties of modified PP fibres were evaluated by the DSC method. Some thermal characteristics (Avrami exponent  $n$ , crystallisation kinetic constant  $K$  and free energy of crystal creation  $\sigma_e$ ) of the blended PP/PET fibres at crystallisation were determined as well as their physical-mechanical properties. The various compatibilisers have a different influence on the crystallisation of PP in the PP/PET fibre blend. EDSA behaves as a nucleating agent and increases the crystallisation of PP. The compatibiliser EVAc decreases the crystallisation of PP in the PP/PET fibre blend. For the crystallisation of PP, heterogeneous nucleation with a tridimensional spherulitic growth of crystallites is characterised. The aforementioned additives emphasise this heterogeneous nucleation of crystallites in the crystallisation of PP in the blended PP/PET fibres. The tensile strength and elongation of the blended PP/PET fibres were changed by the influence of PET and compatibilisers.

**Key words:** polypropylene, polyethylene terephthalate, crystallisation, mechanical properties, blend fibres.

## Introduction

Semi-crystalline polymers can exhibit a wide range of different morphological features depending on the precise composition of the system and its processing history. Of particular importance are molecular mass distribution [1], co-monomer content [2], the presence of additives [3] and the thermal history [4, 5]. Many of these factors are exploited as a general means of controlling structure and the fibres' properties.

The preparation of blended PP/PET fibres with variable properties is a new production method [6 - 12, 21]. The blended PP/PET fibres were prepared to improve the dyeability of PP fibres by the exhaust process [6, 7]. Polyester dispersed in a PP matrix of the blended PP/PET fibres influences the supermolecular structure of these fibres, the PP-PET interphase, the crystalline and amorphous portions, and the dyeability of the blended fibres [8 - 10, 21, 22]. PP and PET in the blended PP/PET fibres behave as individual and immiscible components [9, 13]. The technological miscibility of the PP and PET components can be increased with the addition of compatibilisers. The compatibilisers assure homogenous dispersion of the minor component and provide sufficient mechanical properties of blended PP/PET fibres. Reactive or non-reactive compatibilisers for PP

and PET blends can be used to improve mutual interaction at the interphase. The reactive graft copolymer - polypropylene-maleic anhydride (PP-g-MAN), was used but with a minimal effect. Much better results in the processing of PP/PET fibres were achieved by reactive compatibilisers on the basis of 2-4(alkoxyphenyl) and oxazoline [8]. Reactive compatibilisers decrease the interphase stress between components; they also regulate the molecular weight and flow of dispersed PET properties, as well as increase the colour strength of dyed fibres [9, 10].

The simultaneous influence of PET and one of two compatibilisers on thermal properties and the crystallisation of PP in the blended PP/PET fibres presented in this work is evaluated by a differential scanning calorimetry (DSC).

## Experimental

### Materials

The following polymers were used for the preparation of the blended polypropylene/polyethylene terephthalate (PP/PET) fibres:

1. Tatren TG-920 polypropylene (PP), Slovnaft, a.s., Bratislava, MFI = 11 g/10 min (230 °C/2.16 kg).
2. PET LFK polyethylene terephthalate (PET), Slovensky Hodvab, a.s., Senica, IV = 0.5 l.g<sup>-1</sup>.

Ethylenedistearamide (EDSA) and ethylenevinylacetate (EVAc) copolymer were used as interphase agents – compatibilisers.

The blended PP/PET fibres were prepared in two steps - preparation of PP/PET concentrates and PP/PET blended fibres. The composition of the PP/PET concentrates and the blended PP/PET fibres is given in Table 1.

**Table 1.** Composition of concentrates and blend PP/PET fibres.

Samples	Components	Concentrates, wt. %	Fibres, wt. %
1	PP	–	100
2	PP	–	92
	PET	80	6.4
	EVAc	20	1.6
3	PP	95	99.6
	EDSA	5	0.4
4	PP	–	92
	PET	80	6.4
	EVAc	19	1.52
	EDSA	1	0.08
5	PP	8.5	92.68
	PET	90	7.2
	EDSA	1.5	0.12
6	PP	10	92.8
	PET	80	6.4
	EVAc	9	0.72
	EDSA	1	0.08

### Thermal analysis

The Thermal properties of the blended PP/PET fibres and crystallisation kinetics of PP and PP/PET blended fibres were studied by DSC 7 PERKIN ELMER. In these measurements, a nitrogen atmosphere was applied.

Isothermal measurements of the blended fibres were performed in the following procedure: samples of about 4 mg were heated at a rate of 10 °C/min. up to 280 °C. They were then kept at this temperature for 5 min to eliminate any previous thermal history. After that they were rapidly cooled to 135 °C at a rate of 75 °C/min. The isothermal crystallisation behaviour of the PP/PET fibres was observed at this temperature.

Nonisothermal measurements of the blended fibres were performed in the following procedure: 1) heating – cooling, and 2) heating. The samples were melted and cooled with a temperatures range of between 70 °C to 280 °C, with a constant heating and cooling rate of 5, 10, 15, 20 a 30 °C/min.

Melting ( $T_m$ ) and crystallisation ( $T_c$ ) temperatures were obtained from the maximum of endothermic or minimum of exothermic peaks. Melting ( $\Delta H_m$ ) and crystallisation ( $\Delta H_c$ ) enthalpies were determined from the surface of endothermic or exothermic peaks in the nonisothermal conditions measured. The crystallisation degree  $X_c = 100(\Delta H_m/\Delta H_m^0)$ , where  $\Delta H_m$  is the experimental enthalpy of PP in the PP/PET fibres and  $\Delta H_m^0$  is the enthalpy of the 100% crystalline polypropylene (209 J/g).

The kinetics of isothermal crystallisation in the samples was evaluated by the Avrami equation. The kinetic parameters  $n$  - Avrami coefficient and  $K$  - rate constant of the crystallisation were calculated from the equation:

$$X_c = 1 - \exp(-K \cdot t^n) \quad (1)$$

The kinetics and thermodynamics in crystallisation were analysed on the basis of the Hoffman and Lauritzen secondary nucleation theory [14] expressed by the following equation:

$$\frac{1}{n} \cdot \log K_n + \frac{\Delta F}{2.3 \cdot R \cdot T_c} = A_0 - \frac{4 \cdot b_0 \cdot \sigma \cdot \sigma_e \cdot T_m}{2.3 \cdot k_B \cdot \Delta H_m \cdot T_c \cdot \Delta T} \quad (2)$$

where:

$\Delta T = T_m^0 - T_c$  is undercooling,

$T_m^0$  - equilibrium melting temperatures obtained by extrapolation of the

melting and crystallisation temperature (Hoffman-Weeks plots) [14],

$\Delta H_m$  - the enthalpy of melting,

$b_0$  - the distance between two adjacent fold planes,

$k_B$  - the Boltzmann constant,

$\sigma, \sigma_e$  - the free energies per unit area of the surfaces in the lamella parallel and perpendicular to the axis of a high-molecular chain,

$\Delta F$  - the activation energy of the transport process at the liquid-solid interphase and can be calculated from the Williams-Landel-Ferry equation given in the formula:

$$\Delta F = F_{WLF} = (C_1 \times T_c) / (C_2 + T_c - T_g) \quad (3)$$

where:

$C_1$  and  $C_2$  are constants with values

$C_1 = 4.12$  kcal/mol (17.24 kJ/mol)

and  $C_2 = 51.6$  K,

$T_g$  is the glass transition temperature.

The free energy  $\sigma_e$  is obtained from equation (2) while using the following values provided by the literature:

$b_0 = 5.24 \times 10^{-8}$  m (5.24 Å),

$k_B = 1.38 \times 10^{-38}$  J/K,

$\Delta H_m = 209.10^3$  J/kg,

$\sigma = 11$  mJ/m<sup>2</sup> [14].

### Mechanical properties

Mechanical properties were measured with 1112 Instron tensile tester. Clamping

length was 0.1 - 0.3 m and deformation rate was 0.0083 m·s<sup>-1</sup>.

## Result and discussion

Thermal properties of the blended PP/PET fibres show that additives (PET, EDSA, EVAc) influence the crystallisation process of PP in the blended PP/PET fibres (Table 2 and 3).

The melting temperature of the PP component in the blended PP/PET fibres obtained from the 1<sup>st</sup> heating, reflects on the supermolecular structure formed during the preparation of the PP/PET fibre blend. The measurement conditions (various heating rates) also affect the melting temperature of the PP fibres. The melting temperatures of the PP component in the PP fibres and in the PP/PET blend decrease when the heating rate is increased (Table 2).

The melting enthalpy of PP in the blended PP/PET fibres is lower than the melting enthalpy of pure unmodified PP. This relates to lower PP content in the PP/PET fibre blend as well as with the presence of additives (PET, EDSA and EVAc). This can be argued on the basis of the comparison in experimentally measured and theoretically calculated melting enthalpies of the PP component (Table 3 - page 26). The different influence of the

**Table 2.** Melting ( $T_m$ ) and crystallisation ( $T_c$ ) temperatures of PP in the blend PP/PET fibres.

Procedure	Samples	Heating and cooling rate, °C/min				
		5	10	15	20	30
$T_m$ °C 1 <sup>st</sup> heating	1	164.6	157.0 164.0	155.5 163.4	155.6 162.9	163.7
	2	164.1	160.0 163.9	157.8 162.4	162.2	161.7
	3	164.6	163.9	162.9	156.2 163.9	161.7
	4	164.2	163.1	162.6	157.6 162.2	161.2
	5	164.5	163.3	163.3	162.2	161.4
	6	164.2	163.1	163.6	161.9	161.7
$T_c$ °C Cooling	1	117.2	114.9	113.5	112.4	110.5
	2	118.9	116.5	115.2	114.3	112.9
	3	117.2	114.7	113.5	112.7	111.0
	4	117.8	115.1	113.9	113.0	111.0
	5	117.6	114.9	113.9	114.0	110.9
	6	116.9	114.9	113.2	112.1	110.0
$T_m$ °C 2 <sup>nd</sup> heating	1	159.9 166.5	159.1	158.5	158.7	158.3
	2	160.2 165.8	159.4	159.0	158.9	158.3
	3	159.9 166.4	159.0	158.9	158.3	158.8
	4	157.4 166.2	158.9 163.7	158.5	158.3	157.8
	5	160.0 166.4	160.4 162.2	158.7	158.7	158.3
	6	159.6 166.7	158.8 163.9	158.5 161.9	158.0	158.3

**Table 3.** Experimental (exp) and calculated (calc) melting ( $\Delta H_m$ ) and crystallisation ( $\Delta H_c$ ) enthalpies of PP in the blend PP/PES fibres.

Procedure	Samples	Heating and cooling rate, °C/min									
		5		10		15		20		30	
		exp	calc	exp	calc	exp	calc	exp	calc	exp	calc
$\Delta H_m$ , J/g 1 <sup>st</sup> heating	1	90.1	90.1	90.7	90.7	86.8	86.8	85.6	85.6	91.1	91.1
	2	83.4	84.3	83.8	84.9	78.6	81.3	79.9	80.1	80.5	85.3
	3	87.6	90.1	92.1	90.7	89.7	86.8	89.3	85.6	87.8	91.1
	4	80.1	84.3	82.9	84.9	81.6	81.3	80.9	80.1	81.2	85.3
	5	83.5	83.6	82.8	84.2	80.4	80.6	79.1	79.4	77.8	84.5
	6	80.9	84.3	80.0	84.9	82.5	81.3	78.7	80.1	79.8	85.3
$\Delta H_c$ , J/g Cooling	1	91.7	91.7	94.2	94.2	91.5	91.5	89.9	89.9	89.2	89.2
	2	91.2	84.8	85.9	86.7	80.6	84.2	83.9	81.7	82.9	82.1
	3	95.4	91.3	92.6	93.8	92.9	91.1	88.9	89.5	88.2	88.8
	4	88.9	84.4	96.2	86.7	85.2	84.2	85.1	82.7	84.9	82.1
	5	88.4	84.9	85.6	87.3	85.2	84.8	84.2	83.3	79.9	82.7
	6	87.9	85.1	85.9	87.4	84.3	84.9	81.8	83.4	84.1	82.8
$\Delta H_m$ , J/g 2 <sup>nd</sup> heating	1	84.7	84.7	90.6	90.6	85.8	85.8	85.6	85.6	84.3	84.3
	2	79.8	79.3	76.8	84.8	76.9	80.3	79.7	80.1	75.1	78.9
	3	87.6	84.7	89.1	90.6	87.9	85.8	82.2	85.6	81.7	84.3
	4	79.7	79.3	81.5	84.8	82.0	80.3	77.4	80.1	80.7	78.9
	5	77.5	78.6	84.4	84.1	82.7	79.6	75.2	79.4	73.9	78.2
	6	77.8	79.3	80.8	84.8	82.3	80.3	78.1	80.1	76.9	78.9

compatibilizers on the crystallisation of the blended PP/PET fibres was observed both in the PP/PET/EVAc system, as well as in the PP/PET/EDSA system.

The melting enthalpy of the PP component in the blended PP/PET fibres with EDSA does not obtain the calculated values in the melting enthalpy of the PP fibres, however the difference between the experimental and calculated enthalpy is considerably lower than in the sample containing an EVAc copolymer. The same influence of EDSA is characterised by the melting enthalpy of the PP component at all the heating rates measured (5, 10, 15, 20 and 30 °C/min). Different concentrations of the EVAc copolymer cause a decrease in the melting enthalpy of the PP component in all samples of blended PP/PET fibres, with the same amount of PET and EDSA at all heating rates. This decrease depends on the measured conditions of the thermal properties (heating rate). The highest difference between the experimental and calculated melting enthalpies of the PP matrix in the blended PP/PET fibres appears at the lowest (5 °C/min.) and the highest (30 °C/min.) heating rates (Table 3). The nucleating effect of EDSA decreases with increased concentration of EVAc copolymer, which is confirmed by the comparison of the melting enthalpy of PP in the PP/PET fibre blend, with one compatibilizer (EVAc or EDSA) and with both compatibilizers in various ratios. Higher experimental melting enthalpies of the PP fibres with EDSA in comparison with that of calcu-

lated ones confirm the nucleating effect in the additive (Table 3).

The different cooling rates of the blended PP/PET fibres show their different behaviour during crystallisation. The thermal properties in observed fibres obtained in the 1<sup>st</sup> heating reflect on the former supermolecular structure created during the fibre preparation. The thermal properties obtained in the 2<sup>nd</sup> heating reflect on the different crystallisation rate (Tables 2 and 3). The lower cooling rate causes lower crystallisation temperatures (crystallisation forms at a lower temperature) of the PP component in the blended fibres therefore the crystallisation of the PP component in the blended PP/PET fibres is much better. The experimental melting enthalpies of the PP component in the blended fibres, measured at the lowest crystallisation rate (5 °C/min), are higher than the calculated ones. The blended fibres have a splitting peak of the PP component, which corresponds with two PP crystalline modifications. There is  $\alpha$ - $\beta$ -modification with melting temperatures within the range of 154 °C to 159 °C and  $\alpha$ -modification with melting temperatures within the range of 162 °C to 166 °C (Table 2) [15, 16]. An increased cooling rate decreases the experimental melting enthalpy of the PP component and gives  $\alpha$ - $\beta$ -modification form with melting temperatures within the range of 157 °C to 158 °C (Table 2). Lower temperatures and longer crystallisation time are needed to form a stable  $\alpha$ -modification. The influence

of compatibilisers EVAc, EDSA and their blend EVAc/EDSA in the PP/PET blended fibres on the PP crystallisation at the 2<sup>nd</sup> heating is comparable with results obtained from the 1<sup>st</sup> heating.

The thermal parameters - Avrami index  $n$  and crystallisation kinetic constant  $K$  (which characterise the quality of crystallisation) were calculated for pure unmodified PP fibres, PP fibres with EDSA and PP fibres with EDSA and PET (Table 4).

It is well known that the Avrami index  $n$  depends on the nucleation process and on the geometry of a growing crystal. If the Avrami index  $n$  is about 3, then heterogeneous nucleation with tridimensional spherulitic growth in a spherical form is characteristic of polymer crystallisation [17]. Small variations of the Avrami index  $n$  can occur by experimental and specific errors of the Avrami model [18], as well as by the existence of both primary and secondary crystallisation in the material [19]. Primary crystallisation is characterised by the prevalence of nuclei and a relatively faster growth of lamellar crystals, while a secondary crystallisation process is the result of the crystallisation of a component with different modification and/or by an increment in the perfection of existing crystallites [17, 20]. If the value of  $n$  is about 2, then it is possible to indicate the growth of bidimensional crystallites. Polymer crystallisation with a bidimensional crystal growth in the form of a disk [17] is predicted. The kinetic constant of crystallisation  $K$  characterises the rate of PP crystallisation.

From the values of Avrami index  $n$  ( $n = 3.1 - 3.8$ ) we can conclude that during the PP crystallisation in the PP fibres, fibres from PP with an addition of EDSA and in the blended PP/PET fibres with an addition of EDSA, heterogeneous nucleation with a tridimensional spherulitic growth of PP crystallites is observed. The PP fibres with an addition of EDSA have the highest value of Avrami exponent ( $n = 3.8$ ), as well as the highest crystallisation kinetic constant  $K$  ( $K = 2.29$ ). These two values confirm that EDSA is

**Table 4.** Kinetics parameters of PP crystallisation in the blend PP/PET fibres.

Samples	$n$	$K \cdot 10^5$	$\sigma_e \cdot 10^3$ , J/m <sup>2</sup>
1	3,1	2,16	231
3	3,8	2,29	192
5	3,4	2,19	193

**Table 5.** Mechanical properties of blend PP/PET fibres.

Samples	Clamping length, m					
	0,10		0,20		0,30	
	$\sigma_e$ , cN.dtex <sup>-1</sup>	$\epsilon_e$ , %	$\sigma_e$ , cN.dtex <sup>-1</sup>	$\epsilon_e$ , %	$\sigma_e$ , cN.dtex <sup>-1</sup>	$\epsilon_e$ , %
1	1,94	124	1,99	116	2,00	105
3	1,33	94	1,44	105	1,42	90
5	1,85	139	1,69	128	1,71	126

a good nucleating agent for PP. In the PP/PET/EDSA blend the nucleating effect of EDSA on the crystallisation ability of the PP component is suppressed ( $n = 3.4$ ;  $K = 2.1$ ). A decreased nucleating effect of EDSA in this system could be caused by the following; a decrease in the amount of PP in this blend, by the dispersion of PET in a PP matrix, by the creation of a PP/PET interphase and dispersion of EDSA in PP, and by PET components of this system (Table 4).

The secondary nucleation Lauritzen-Hoffman theory (6) was used for the calculation of the free energy ( $\sigma_e$ ) per unit area in the surfaces of the lamellae perpendicular to the chain direction of PP macromolecules. The free energy was calculated by three samples i.e. unmodified PP fibres (sample 1), PP fibres with EDSA (sample 3) and blended PP/PET fibres with EDSA (sample 5). The addition of EDSA and a blend of PET/EDSA to the PP, causes the decrease of free energy in crystal creation as opposed to that in unmodified PP. This means that to create crystallites a lower energy is necessary, and therefore their creation is faster and more frequent in comparison with unmodified PP (Table 4).

Physical-mechanical properties (tenacity, elongation at break) at the various measured conditions (e.g. clamping length) of unmodified PP fibre, PP fibre with EDSA, and blended PP/PET fibre with EDSA were evaluated as well. The results of these measurements are displayed in Table 5.

Additives (EDSA and PET) decrease the tenacity of fibres at all clamping lengths. A significant decrease in tenacity was observed in the PP fibre with EDSA as well as in the PP/PET fibre with EDSA as opposed to just the PP fibre. These results correspond with the thermal characteristics of the evaluated fibres. A PP fibre with EDSA, in which the crystallisation conditions for tridimensional spherulitic growth are the most optimal (Avrami index  $n$  is higher and the free energy  $\sigma_e$  is the lowest – Table 4), has the lowest tenacity. The results obtained

confirm the dependence between thermal characteristics (Avrami index  $n$ , crystallisation kinetic constant  $K$  and the free energy  $\sigma_e$ ) and tenacity of an unmodified PP fibre, PP fibre with EDSA and a blend of PP/PET fibre with EDSA. The Theory of fibre tenacity is based on the assumption that fibre tenacity decreases with the increase of spherulitic size in a fibre.

Additives (EDSA and PET) do not uniformly influence the elongation of the PP fibre with EDSA and the blended PP/PET fibre with EDSA. With an addition of EDSA the elongation of the fibre decreased very fast (approx. 30%) in comparison with the elongation of the non-modified PP fibre (Table 5). Larger tridimensional crystallites affect the behaviour of fibres in loading and prevent higher deformation. With an addition of PET into the system PP/EDSA, the elongation value of the fibre increased over the elongation of the unmodified PP fibers. Better deformability of PET in comparison with PP and its effect on the creation of smaller tridimensional crystallites caused an increase in the elongation of PP/PET fibres with EDSA.

## Conclusions

From the presented thermal properties of the PP and the blended PP/PET fibres with compatibilisers EVAc and EDSA we can show that:

1. The heating and cooling rates influence the thermal properties of the observed PP/PET fibres
2. EDSA and EVAc copolymer influence the crystallisation of the PP component in the blended PP/PET fibres. EDSA increases and EVAc copolymer decreases the PP crystallisation ability in the blended PP/PET fibres.
3. Crystallisation in PP fibres, in PP fibres with EDSA and in the blend of PP/PET/EDSA is characterised by heterogeneous nucleation with a tridimensional spherulitic growth of crystallites. The Avrami exponent  $n$  is in the range 3.3 – 3.8.
4. The addition of EDSA and PET to PP causes a decrease in free energy, which is needed for the creation of crystals.

5. The tensile strength and elongation at break of the blended PP/PET fibres were changed under the influence of PET and compatibilizers.

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