

Structural features and relaxation properties of PET/PC blends containing impact strength modifier and chain extender

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Abstract. It has been investigated how methylene diphenyl diisocyanate (MDI) influences the morphology, rheological, mechanical and relaxation properties, as well as PET crystallizability, of PET/PC/(PP/EPDM) ternary blends produced by the reactive extrusion process. It appears that irrespective of phase structure of the blends, MDI causes a rise in melt viscosity (decreased MFI-values) of the material which is the result of an increased molecular weight of the macromolecules; PET crystallization becomes retarded. MDI improves compatibility between PET and PC in PET/PC/(PP/EPDM) ternary blends. Addition of MDI leads to higher values of the dynamic shear modulus for PET high elastic state (in the temperature range between T_g PET and cool crystallization temperature of PET); the PET cool crystallization and melt crystallization processes become retarded; the PET and PC glass transition temperatures approach one another. MDI has been shown not to influence significantly the blend morphology or the character of interaction between the PP/EPDM disperse phase and PET/PC blend matrix.

Keywords: polymer blends, poly(ethylene terephthalate), polycarbonate, impact modifier, chain extender

1. Introduction

The blends of polycarbonate (PC) with poly(alkylene terephthalates), particularly poly(ethylene terephthalate) (PET), have been the subject of much attention from researchers as well as scientific schools [1–4]. This can mainly be explained by growing possibilities for developing PET/PC-based engineering materials; their properties are far more advantageous than those of the two homopolymers. Besides, when making blends, it seems possible to use – at least as one of the blend components – industrial or/and household wastes, for example, used (post-consumer) PET bottles. As a result, the commercial production of similar materials becomes quite profitable.

One of the disadvantages of PET/PC binary blends is their low impact strength measured on notched samples [2]. PET/PC blend materials show relatively high values of impact strength only at temperatures between the component glass transition temperatures (T_g PET and T_g PC) [2]. For practical applications, therefore, such PET/PC blends are most suitable that contain special additives, i.e. impact strength modifiers (ISM) [5] which can increase the impact strength.

Another important problem that arises during compounding and processing of PET/PC molten blends is to prevent hydrolytic and thermal degradation of macromolecules of polyesters being blended. This problem comes, first of all, from the fact that

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molten blends are usually processed at a temperature of 270°C or higher, because PET has a high melting temperature; the reaction rate turns out to be high and causes degradation of the macromolecular chains [6]. The reactions of macromolecular breakdown lead to reduced molecular weights, lower values of mechanical properties and lower thermal stability, which unfavorably influences the possibility of repeated processing or recycling of polyester materials [7].

Complete polycondensation initiated in the solid phase is one of the ways to prevent a negative influence of macromolecular breakdown on the properties of polycondensation thermoplastics, among them saturated polyesters such as PET and PC [8, 9]. High technological expenses required for the solid-state additional polycondensation, however, make reprocessing of polyester raw materials unprofitable.

During the last 10–15 years especially, alternative technologies have been developed for the modification of polyesters. These technologies show essential advantages compared with the solid-state polycondensation. These alternative technologies are mostly based on chemical transformations of macromolecules in molten polyesters; such chemical transformations occur with the help of additional bi- or multi-functional chemicals capable of extending the chain (so-called chain extenders) [10–15]. The chain extenders (CE) interact in melt mostly with terminal functional groups of polyester macromolecules and link them with one another; so the molecular weight does decrease, it rather increases to some extent. It was established that reactions of interaction between macromolecules and CE proceed most favourably in melt in the reactor-extruder equipped with one or two screws [16–18]. Usually, a vacuum treatment and special catalysts are used to promote these reactions [18].

This technology based on extrusion equipment becomes economically advantageous for processing polyester materials. Its abilities have been mainly investigated only for certain types of polyesters. It hasn't been understood clearly what role is played by CE in polyester blends of a more complex composition. An example of such blends is PET/PC containing ISM. The purpose of this work is to consider structural details and relaxation properties of these blends.

2. Experimental

2.1. Materials

The material was PET produced commercially by Mogilevhimvolokno Co. (Grade PET-8200 intended for making bottles and other types of packages; intrinsic viscosity, 0.882 dl/g; concentration of terminal carboxyl groups, 22.8 mmole/kg; glass transition temperature, T_g PET = 80°C). The second basic component for the blend was PC produced by Zarya Co, Russia (molecular weight \approx 35 000; T_g PC = 144.5°C). The ISM was elastomer PP/EPDM based on a mixture of polypropylene and ethylene-propylene-diene rubber (melting temperature of the polypropylene component T_m PP = 165.5°C; crystallization temperature T_{cr} PP = 110.5°C; MFI = 2.8 g/10 min (at $T = 210^\circ\text{C}$; $P = 21.6$ N; $D_c = 2.095$ mm); high flow limit of strain 10 MPa; relative elongation at rupture 595%; glass transition temperature $T_g = -40^\circ\text{C}$). The PP/EPDM concentration was constant, 5 wt%, in all of the compositions tested (Table 1). The chain extender, as in work [19], was methylene diphenyl diisocyanate (MDI); its concentration was 1 wt%.

Table 1. Compositions of test materials

Composition No	Components and their concentration [wt%]
1	PET – 100 wt%
2	PC – 100 wt%
3	PET/PC – 25 wt%/(PP/EPDM) – 5 wt%
4	PET/PC – 50 wt%/(PP/EPDM) – 5 wt%
5	PET/PC – 75 wt%/(PP/EPDM) – 5 wt%
6	PET/PC – 25 wt%/(PP/EPDM) – 5 wt%/MDI – 1 wt%
7	PET/PC – 50 wt%/(PP/EPDM) – 5 wt%/MDI – 1 wt%
8	PET/PC – 75 wt%/(PP/EPDM) – 5 wt%/MDI – 1 wt%

2.2. Preparation of blend compositions and test samples

The blend compositions have been compounded by the reactive extrusion method on extrusion-granulating line based on the twin-screw extruder TSK-35/40 (China); the screws diameters were 35 mm each; $L/D = 40$. The melt temperature in the extrusion zone was 265°C. MDI was introduced into the blend material as follows. First PET and PC granules were dried. Then the mixture of these granules was treated by MDI immediately before loading it into the material cylinder of the extruder.

The test samples for measuring values of the mechanical properties of the materials were injection moulded on machine EN-30 (Taiwan); screw diameter, 35 mm; shot volume, 30 cm³. The mould temperature was set equal to 50°C.

The relaxation spectrometry was performed on plates of dimensions: 50×5×1 mm. The plates were made on laboratory-type moulding plunger machine with a shot volume of 2.5 cm³.

Before compounding and making test samples the materials have been dried to a residual moisture content below 0.02%.

2.3. Methods of analysis

The mechanical properties of the materials in tension were determined on a universal testing machine Instron 5657 (Great Britain). The Charpy impact viscosity was determined on Charpy notched samples using the pendulum hammer PSW-1.5 (Germany). The test samples were blades with a neck measuring 50×5×3 mm and bars: 80×10×4 mm. The rheological properties were judged by MFI-values found at $T = 265^{\circ}\text{C}$, $P = 21.6\text{ N}$ and $D_c = 2.095\text{ mm}$ (instrument IIRT-AM, Russia).

The analysis of temperature dependences of mechanical loss tangent ($\tan\delta$) and dynamic shear modulus (G') of the samples gave information on the dynamic-mechanical (relaxation) properties of the materials. The tests were run as in other works [2, 20], using the reverse torsion pendulum of design developed at MPRI NAS, Belarus. The test frequency was 1 Hz. The measurement accuracy was: 0.1°C for temperature; ±3% for $\tan\delta$; ±1% for G' . The test samples – plates measuring

50×5×1 mm – were injection moulded on the laboratory moulding machine of plunger type; the shot volume was 5 cm³.

The structural morphology of the materials was studied by SEM-images taken of sections of bars (from the central portion of bars) after they had been exposed to liquid nitrogen for 30 min. The scanning electron microscope was VEGA II LSH (Czech Republic). The differential scanning analysis was performed using an instrument DSM-10M (Russia) at a heating/cooling rate of 16°C/min with weighed samples of 8 mg each. The samples were used for the analysis taken from the middle part of the specimens used for the relaxation spectrometry.

3. Results and discussion

3.1. Effect of MDI on mechanical and rheological properties of PET/PC/(PP/EPDM)

It can be seen in Table 2 that MDI – 1 wt% added to PET/PC/(PP/EPDM) blends regardless of their phase structure changes their properties. It is worth mentioning that in PET/PC blends containing PC-25% the dispersed medium was formed by PET whereas with PC-50% there were two continuous media – PET and PC. With PC-75%, the dispersed phase was PET [21]. The blends modified by MDI showed a somewhat higher level of mechanical properties such as σ_{LF} , σ_{HF} , ϵ_r and a (Table 2). An addition of MDI distinctly increased K_s . The MFI-values were noticed to decrease for all of the compositions. The latter fact is indicative of efficiency of MDI as a chain extender in the blends under investigation [19]. It can be understood, therefore, that PP/EPDM present in PET/PC blends is not

Table 2. Values of mechanical and rheological properties of materials

Composition No	MFI [g/10 min]	σ_{HF} [MPa]	σ_{LF} [MPa]	K_s [%]	ϵ_r [%]	a [kJ/m ²]
1	23.0	55	30	100	232	7.4
2	6.1	68	53	98	126	16.6
3	22.0	53	34	87	180	17.7
4	17.0	55	38	84	140	19.6
5	13.0	56	42	86	146	48.3
6	18.0	54	35	95	214	19.0
7	14.0	56	39	94	142	21.4
8	11.0	54	43	90	150	49.5

Here and henceforth, the compositions are marked as in Table 1. σ_{HF} , σ_{LF} , ϵ_r are, respectively, high and low flow limits and relative elongation at rupture; K_s is strength factor for melt weld flow in injection moulding determined from the expression: $\sigma_{HF1}/\sigma_{HF2} \cdot 100\%$, where σ_{HF1} and σ_{HF2} – are, respectively, values of the upper flow limit for blade-samples moulded by melt injection in the face plane of one end and of two ends; a is Charpy impact strength measured on sharply notched samples at $T = 23^{\circ}\text{C}$.

harmful towards MDI used as a chain extender for polyester materials. However, despite a decrease in MFI (a higher melt viscosity), the blends modified by MDI showed an increase in K_s . As this factor much depends on both the melt viscosity and on adhesion strength interaction between the phases in blends of thermodynamically incompatible or partly compatible polymers, it can be assumed that incorporated MDI causes an intensification of interphase adhesion in PET/PC blends along with an enhancement in compatibility of the components.

Table 2 shows that Charpy impact strength values determined for notched samples of compositions No.5 and No.8 are much higher than for compositions No.3–4 and 6–7. An obvious reason for this is the fact that in compositions No.3–4 and 6–7, the non-impact-resistant PET forms a continuous (disperse) medium. In compositions No.5 and No.8 the disperse medium is PC, whose impact strength much exceeds α -values for PET (Table 2, compositions No.1 and No.2). Thus the reason for a sharp rise in α values for compositions No.5 and No.8 is

that the impact strength level for them is supported mainly by PC phase; PC is highly resistant to impact breakdown that follows chiefly the mechanism of shear flow [2]. In the blends studied, the effect of PP/EPDM is evidently reduced to increasing the degree of heterogeneity of the blend in general and of PC phase in particular. MDI acting as a chain extender encouraged an increase in shear flow resistance of the material at impact loading. That is why all of the compositions containing MDI show somewhat higher α -values.

3.2. Dynamic-mechanical properties of the blends

Relaxation spectrometry allows estimating variations in dynamic mechanical properties of blends within a wide temperature range and yields experimental data which can be indicative of specific interactions between phases in blends [2, 20, 21]. The temperature dependences of $\tan\delta$ and G' for initial PET and PC, as well as blend compositions based on them, are shown in Figure 1. Numerical

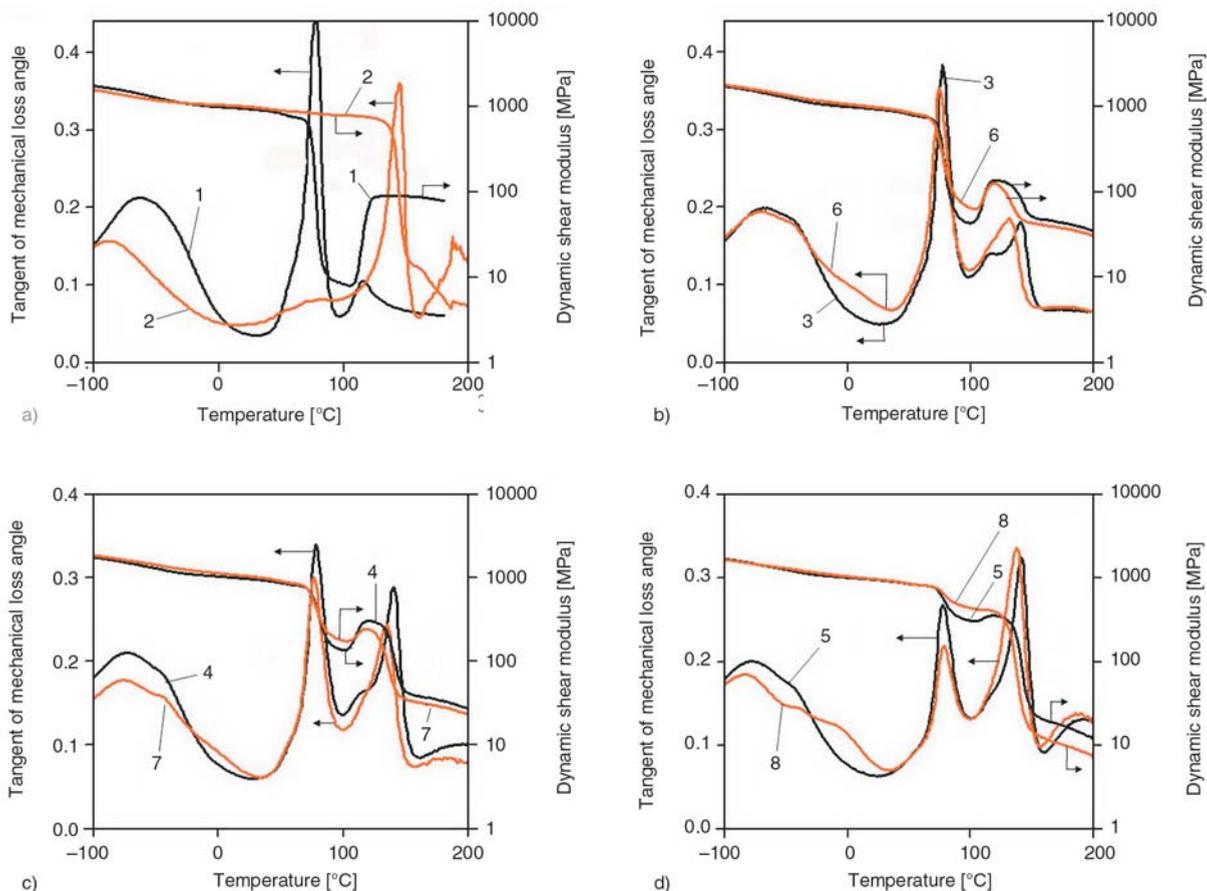


Figure 1. Mechanical loss tangent and dynamic shear modulus for polyester materials: here and henceforth the numbers of compositions are designated by digits on curves in accordance with Table 1

Table 3. Values of parameters characterizing relaxation properties of polyester materials

Composition No	$T_{g\text{ PET}}$ [°C]	$T_{g\text{ PC}}$ [°C]	ΔT_g [°C]	T_β [°C]	G'_{100} [MPa]	$\Delta G'_{cc}$ [MPa]
1	76.5	–	–	–61.3	8	82
2	–	144.5	–	–87.3	795	–
3	76.8	141.0	64.2	–66.8	43	93
4	78.0	141.2	63.2	–71.7	136	166
5	77.6	141.6	64.0	–76.9	308	84
6	74.3	132.0	57.7	–70.4	65	60
7	78.0	135.0	57.0	–74.2	158	59
8	78.2	138.0	59.8	–82.3	437	0

$\Delta T_g = T_{g\text{ PC}} - T_{g\text{ PET}}$; T_β is temperature of main maximum within the β -relaxation temperature region; G'_{100} is dynamic shear modulus at 100°C; $\Delta G'_{cc}$ is a rise in G' values with respect to G'_{100} caused by cool crystallization of PET

values of the parameters that characterize relaxation properties of the materials are listed in Table 3. An analysis of the available information led to several important conclusions about the effect of MDI on relaxation behaviour and interphase interactions in PET/PC/(PP/EPDM) ternary blends.

It can be understood from Table 3 that after MDI was added the values of T_g for PET and PC approach one another. The value of ΔT_g decreases mainly at the expense of decreased $T_{g\text{ PC}}$, not at the expense of increased $T_{g\text{ PET}}$. This can be explained by an improved compatibility of the components under the effect of MDI, as well as by plastification of PC by the amorphous phase of PET the T_g values of which are lower than $T_{g\text{ PC}}$. The addition of MDI makes the values of major peak of β -relaxation shift appreciably, by 3.5–5.4°C, to the lower temperature region. The kinks present in the peak maximum of β -relaxation at $T = -41^\circ\text{C}$ are a result of simultaneous glass transition process of PP/EPDM elastomeric phase and β -relaxation process of basic components of blend (PET and PK). T_g of pure PP/EPDM is -40°C . The scanning spectroscopy data show that not only at the segmental level, but also at the level of PET and PC monomer units, effective intermolecular interactions take place; MDI plays an essential role in them.

After MDI was added, T_β values of the blends shifted (by $\approx 5.4^\circ\text{C}$) to the lower temperature region. The lower T_β values predetermine an opportunity of developing more frost-resistant (lower brittle temperature) materials based on MDI-modified blends.

The most important consequence of MDI added to the PET/PC/(PP/EPDM) ternary system is a con-

siderable rise in dynamic shear modulus values within the region of high elastic state of PET (at temperatures between $T_{g\text{ PET}}$ and cool crystallization of PET in blend, being $\approx 120^\circ\text{C}$ as determined by the scanning spectroscopy technique). For a quantitative estimation of this effect, Table 3 gives $\Delta G'_{100}$ values which approximately correspond to G' minimum values of the materials when PET in blends undergoes devitrification.

After PET was devitrified and the blend was heated to $\approx 120^\circ\text{C}$, the amorphous portion of this polymer undergoes cool crystallization, and G' values of the material rise (Figure 1, Table 3). The presence of MDI in the blend material causes PET cool crystallization to proceed at a slower rate up to complete stopping in PET/PC – 75%/(PP/EPDM) – 5%/MDI – 1% blends (for these blends $\Delta G'_{cc} = 0$, Table 3). It can be seen that a rise in $\Delta G'_{100}$ has been caused by a more active interphase (segmental) interaction between the amorphous PC and amorphous phase of the devitrified PET. It is owing to stronger adhesion between the phases – and, probably, to partial mutual dissolution of the components that restricts segmental mobility – that PET cool crystallization is either retarded or suppressed in blends containing MDI. Some earlier works had described the determinative effect of segmental mobility on crystallizability of partly crystalline thermoplastics [21–23].

3.3. Results obtained by differential scanning calorimetry technique

It was established earlier that mechanical properties of PET-based blend materials much depend on the tendency of this polymer to crystallize. An increase

Table 4. DSC-results obtained for polyester materials

Composition No	Cool crystallization at heating		Melting		Crystallization at melt cooling		a ^a [%]
	T _{cr} [°C]	ΔH _{cr} [J/g]	T _m [°C]	ΔH _m [J/g]	T _{cr} [°C]	ΔH _{cr} [J/g]	
1	130.4	28.2	253.3	44.1	194.4	27.7	11.0
3	139.5	15.5	251.0	25.2	177.8	25.9	9.5
4	143.6	13.1	252.8	18.8	182.3	25.8	8.2
5	139.0	7.4	249.5	9.2	179.9	8.6	5.5
6	145.3	12.0	244.0	21.1	165.5	29.3	8.9
7	146.6	10.8	248.9	13.4	169.0	14.5	3.9
8	148.0	4.5	252.5	5.7	160.7	8.5	3.4

^aCrystallinity $a = (\Delta H_m - \Delta H_{cr}) \cdot 100 / (k \cdot \Delta H_m^*)$, where ΔH_m is latent melting heat, ΔH_{cr} is latent heat of cool crystallization, $\Delta H_m^* = 144.664$ J/g is latent melting heat of PET with 100%-crystallinity [19], k is PET concentration (in wt parts) in the material

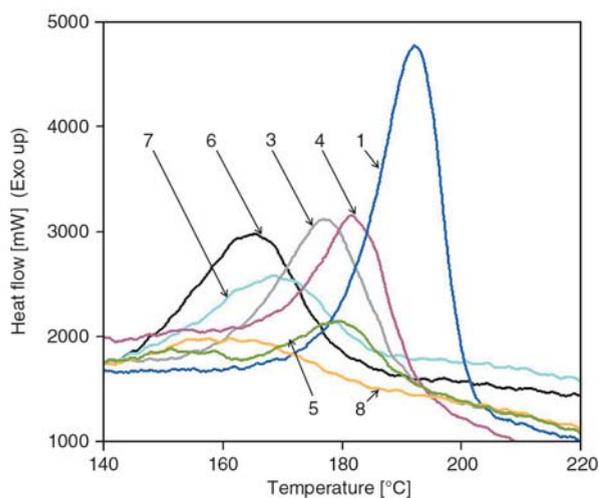


Figure 2. DSC-curves for cooling polyester blends

in the rate of crystallization and in crystallinity of the PET-component can result in a decrease in the blends plasticity and impact strength [24].

The results of analysis of PET crystallizability for tested blend materials are compared with initial polyester in Figure 2 and Table 4. The most important result of DSC-analysis is that the presence of MDI leads to rate retardation of crystallization and a lower crystallinity in a blend. The severity of MDI influence upon PET crystallizability was found to depend on the blend composition: the higher the PC concentration in the blend, the stronger the influence of MDI. This can be explained by the fact that in PET/PC/(PP/EPDM) ternary blends, PET undergoes crystallization at a slower rate, in comparison with pure polyester, because of interaction with PC-component [2].

The influence of MDI on PET crystallization in melt is especially easily visualized when analyzing DSC-curves of cooling. It can be seen in Figure 2 and Table 4 that in comparison with pure PET and a binary blend, the extent of melt overcooling reaches 20°C in order to ensure PET crystallization.

Thus in MDI – modified blends, diisocyanate acts as a chain extender and increases the PET molecular weight. It also interacts with terminal groups of PET and PC macromolecules, and obviously is helpful in forming copolymers. As a result the PET molecular mobility is restricted causing PET crystallization to proceed at a slower rate. The introduction of MDI in a PET/PC/(PP/EPDM) system may be one of the effective ways to make PET amorphous in the material.

Cuts of blends were made in liquid nitrogen and SEM-images (Figure 3) were analyzed to reveal that all of the blends morphologically look identical. Within a PET/PC quasi-homogeneous matrix, PP/EPDM forms sphere-like particles of a maximum size between some fractions of a micrometer up to 3 to 4 μm. Larger particles are relatively few. In these micrographs it is impossible to distinguish and identify phases of the composites independently of the phase structure of the materials because the interaction between PET and PC is intensive and they partly compatible in the blends investigated. No traces of a foreign phase are present on PP/EPDM particles which implies that there are no strong specific interactions between the impact strength modifier used and PET/PC blend matrix irrespective of the type and phase structure of the blends. It appears, therefore, that MDI modifies predominantly the basic components of the PET/PC blend matrix and not PP/EPDM and its interaction with the polyester matrix.

4. Conclusions

The reactive blending of PET, PC, 5 wt% of impact strength modifier – PP/EPDM – and MDI – 1wt% has been observed to cause great variations in the molecular structure and values of main properties

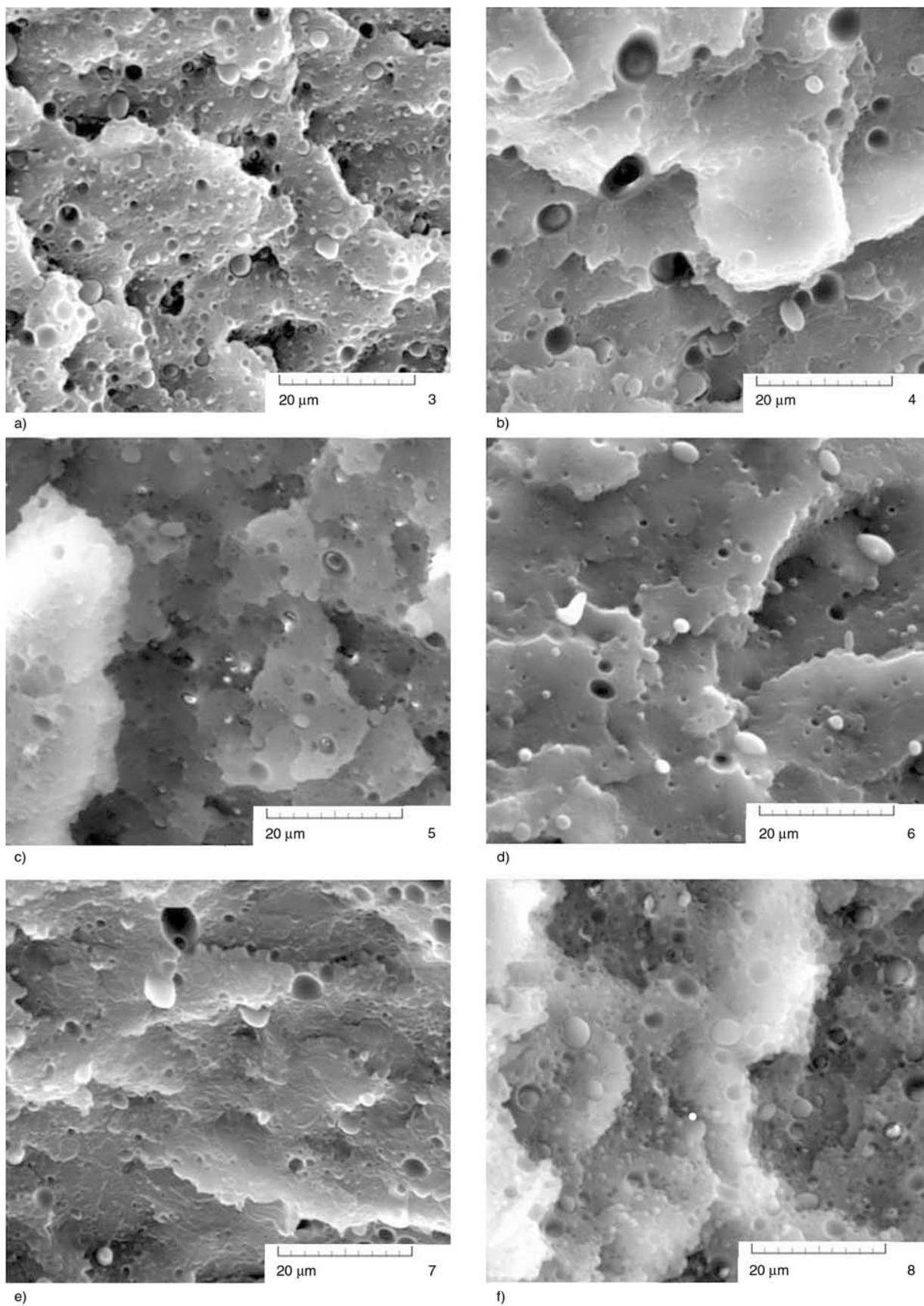


Figure 3. SEM-images (a–f) of polyester blend sections (3–8) cut under liquid nitrogen

of the materials under discussion. The addition of MDI leads to increased melt viscosity of the material (decreased MFI values) which can be explained by increased molecular weights of the components; PET crystallization proceeds at a lower rate. MDI improves compatibility of PET and PC in PET/PC/(PP/EPDM) ternary blends. This modifier causes a substantial rise in the dynamic shear modulus within the region of PET high elastic state (over the temperature range between T_g PET and temperature of PET cool crystallization). It also promotes approach of PET and PC glass transition temperatures; the processes of PET cool crystallization and crystallization from melt become retarded. MDI does not cause a noticeable influence on the blend morphology and on the character of interaction between the PP/EPDM disperse phase and PET/PC blend matrix.

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