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Enhancing properties of PC/PA blends via compatibilization of olefin-maleic-anhydride copolymer based additives in masterbatch form

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Abstract

Polymer blending has been a simple and efficient way for designing and controlling the performance of polymeric materials using easily available types. Both polycarbonate and polyamide have excellent mechanical properties and thermal stability but their disadvantages such as limited chemical or water resistance can be eliminate by tailoring them. Main difficulties in processing of PC/PA blends are the poor compatibility and high moisture adsorption capacity of the two raw materials complicating processing and also deteriorating mechanical properties of the products. Compatibilizing additives such as olefinmaleic-anhydride copolymer based compounds used in the experimental work can help to overcome the abovementioned difficulties. To determine the processing conditions of the raw materials several drying temperatures have been tested and thermal degradation has been examined by FT-IR spectroscopy. Experimental compatibilizing additives based on an olefinmaleic-anhydride copolymer have been investigated to enhance mechanical properties of the blends prepared by extrusion moulding. Mechanical, rheological, SEM and FT-IR measurements have been performed and at least one additive has been found to be efficient in improving selected properties.

Keywords Material testing · Mechanical properties · Polymer blends and alloys · Rheology

Introduction

Nowadays the permanently increasing demand for engineering plastics has been an incentive to explore the opportunities of producing newer, high-performance polymer blends. The variation of the components and additives of the new materials offers numerous possibilities, but it is also necessary to meet the often good chemical resistance requirements as well as the outstanding mechanical and thermal properties of modern engineering plastics.

Both polycarbonate (PC) and polyamide (PA) are a type of engineering plastics with excellent mechanical properties and good thermal stability, and their disadvantageous properties are symmetrical to each other [1–3]. PC has good water resistance, but it is easily attacked by organic solvents,

T. Kovács kovacs.tamas@mk.uni-pannon.hu whereas PA is well resistant to organic solvents, however, it is characterized by greater hydrolytic degradation depending on the length of the carbon chain between the amide bonds [4–6]. The combination of two polymers are advantageous if certain properties are enhanced, while other properties do not deteriorate substantially compared to the raw materials. Since PC and PA are incompatible polymers, in that case those requirements are not fulfilled. Mechanical properties of their blend (impact resistance, tensile strength, tensile or elastic elongation etc.) are much worse compared to pure polymers, because mixing of the components is extremely weak, interfaces are easily separated, so the load transfer between phases is very limited [7]. Addition of polyamide increases crystallisation tendency of polycarbonate causing brittleness [8]. Improvement of the compatibility and thus the mechanical properties of PC/PA blends can be achieved with various compatibilizing additives.

Compatibility takes place by mixing process, also known as addition process, where a steric stabilizing effect can be achieved with the help of a third component added during processing in order to reduce tension between the interfaces [9]. That component is usually a pre-synthesized

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block or graft copolymer composed of the polymers that make up the blend. The compatibilizing effect of ABand ABA type block copolymers prepared from mono or diamine terminal PA homopolymer and high molecular weight PC can significantly reduce the size of PC particles dispersed in polyamide and achieve greater adhesion to the matrix [10]. An extremely high quality PA-PC block copolymer alone can be prepared in the reaction of PA 6 and polycarbonate diol (PCD) with a diphenylmethane diisocyanate (MDI) coupling agent. The resulting copolymer has an elongation at break of over 900% [11]. However, the disadvantages of addition compatibilisation are that it is often difficult to synthesize copolymers in blends of different compositions, and the additives prone to form micelles, thereby reducing their efficiency, increasing viscosity of the melt and deteriorating mechanical properties

of the blend [8]. Nowadays in-situ also known as reactive compatibilization has been widely used and the essence of that technique is to form block or graft copolymers on the interface of the phases directly by means of covalent (rarely ionic) bonds [9]. The additive forms a direct bond with one component while mixes with the other. A major advantage over the addition technique is that the copolymers are formed just at the interface of the phases during the mechanical processing. With epoxy resin additives a significant improvement can be achieved in PC and PA blend, as the oxirane rings are easily cleaved and react with hydroxyl, carboxyl, and amine groups, thus resulting in situ block copolymer formation at the surfaces of the segments [12]. As a result of the 1% additive concentration, elongation at break of the blend doubled, impact strength improved by 70%, the interfaces between the PC and PA segments became blurred and the structure became more coherent. A good compatibilizing effect also can be achieved with hyperbranched polyethylenimine (hPEI) in PC and amorphous PA blends [13]. Reactive compatibilization also affects the thermal properties of polymer blends. Plasticizer effect of the polypropylene-oxide (PPO) additive increases the mobility of PC and PA molecules in the melt, thereby promoting the formation of PC-PA copolymers that reduce interfacial tension and increase decomposition temperature of the blend [14]. Alumina nanoparticles are also effective in increasing thermal stability of PC/PA blends and have a positive effect on mechanical properties, especially on elongation at break [15].

The aim of our experimental work was to investigate the effect of experimental olefin-maleic-anhydride copolymerbased additives in PC/PA blends in order to improve compatibility of the components, thereby enhancing properties of the blends. In addition to the study of processing parameters and mechanical properties, rheological characteristics and morphology were also investigated.

Experimental

Materials

During the experimental work, Teijin Panlite L-1225L (MFI: 21.6 g/10 min (300 °C; 1.2 kg)) commercial polycarbonate granules and DOMO DOMAMID 6AV (MFI: 165 g/10 min (275 °C; 5 kg)) type PA 6 was used. Masterbatches (MB) were prepared from three types of HDPE (MB-1: TIPELIN 5700S (MFI: 1.7 g/10 min (190 °C; 5 kg)) MOL Petrochemicals, MB-2: TIPELIN FS 383-03 (MFI: 0.8 g/10 min (190 °C; 5 kg)) MOL Petrochemicals, MB-3: Hostalen GC 7260 (MFI: 23 g/10 min; (190 °C; 5 kg)) LyondellBasell). The investigated compatibilizing additives were olefin-maleic-anhydride copolymer-based self developed additives of the University of Pannonia. The experimental additives were prepared by functionalizing the same olefinmaleic-anhydride copolymer chain with alcohol and amine components (Table 1). Their effects were compared with commercial POLYBOND 3009 additive (PB), which is a polyethylene type modifier grafted with maleic-anhydride.

Preparation of PC/PA blends

The PC/PA blends were prepared in a LabTech LTE 20–44 twin screw laboratory extruder. The screw speed of the extruder was 50 rpm for each sample, thereby the residence time of the melt in the device was 100 to 110 s. The fiber exiting the tool was pulled at a constant speed and then shredded. The temperature profile varied between 232 °C and 250 °C depending on the composition of the blend, increasing with decreasing PC content.

PC/PE masterbatches containing the additives were prepared on a LabTech LRM-M-100 laboratory two roll mill. The composition of the masterbatches was 90 wt% PC and 10 wt% PE. The rolls were 230 °C and 200 °C, the friction ratio was 1.76 and homogenization time was five minutes. Masterbatches were then granulated and co-extruded with the polyamide.

 Table 1 Typical properties of the experimental additives

22.1	-
51.3	165.3
.3776	0.2713
.6224	0
)	0.3644
	0.3644
)	51.3 .3776 .6224

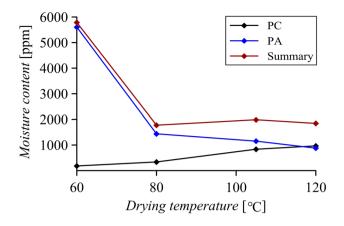


Fig. 1 Variation of PC and PA moisture content as a function of drying temperature

Characterization

Moisture content of the raw materials was measured with an Aboni HydroTracer FMX, and the rate of thermal degradation was examined with a SHIMADZU IRTracer-100 infrared spectrometer. 10 cm long test specimens were cut from the extruded strings. Tensile mechanical properties were measured under laboratory conditions (room temperature, 40% relative humidity) with an INSTRON 3345 universal tensile testing machine with a drawing speed of 10 mm/min. The extruded samples were conditioned at 60 °C for 4 h before measurements. The results were taken from the average of a minimum of seven parallel measurements. SEM images were taken of the samples fractured surfaces. The microstructure of the samples were tested by FEI/ThermoFisher Apreo S scanning electron microscope. Observation by SEM was carried out in low vacuum with an accelerating voltage of 10.0 kV. Since the samples are obviously non-conductive by nature, making the surfaces conductive for SEM analysis a Blazers Union SCD 020 type gold evaporator were used to apply a thin electron transparent layer to the broken surface. The EDX analysis was also performed with the SEM instrument. The residue of the extruded samples were granulated and then, after identical sample conditioning, rheological characteristics of the blends were identified with a CEAST Smart RHEO 2000 capillary rheometer (at 255 °C, two minutes preheating time, shear rate below 10,000 1/s). Viscoelastic properties were determined by an oscillatory rheometer (MCR 302, Anton Paar) at 220 °C after conditioning. Plate-plate (PP) geometry was applied for the measurement with a constant gap of 1.0 mm between the plates. Firstly, amplitude sweep test was carried out for

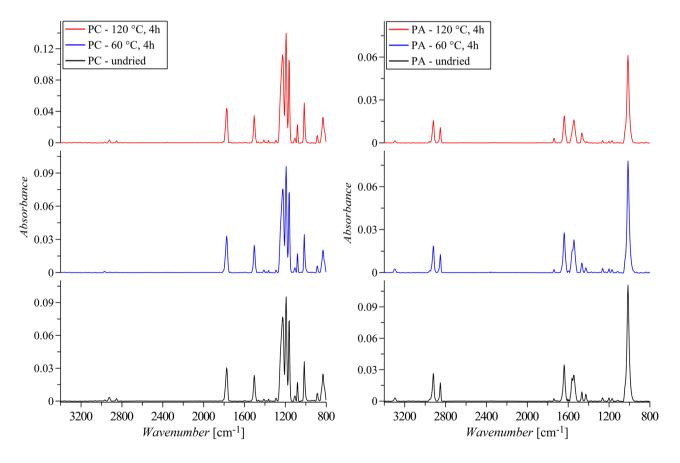


Fig. 2 FT-IR spectra of PC (left) and PA (right) for undried, dried at 60 °C and dried at 120 °C raw materials

determination of linear viscoelastic range for all of the specimens using 10 Hz frequency. Angular frequency was changed between 0.01 and 100 rad/s during frequency sweep test applying the 10% strain amplitude determined by amplitude sweep. X-ray diffraction (XRD) analyses were performed using a Philips PW 3710 type diffractometer (Philips Analytical, Almelo, The Netherlands) equipped with a PW 3020 vertical goniometer and a curved graphite diffracted-beam monochromator. The applied radiation was CuK α from a broad-focus Cu tube, operating at 50 kV and 40 mA. The samples were measured in a continuous scan mode with 0.02°/s scanning speed. Data collections were carried out with X'Pert Data Collector software. Profile fitting option of PC-APD software was applied to determine the integral intensity of individual reflections.

Results and discussion

During the experimental work, the appropriate preparation and process parameters were set, depending on the measurement results of the pure polymers and blends, as well as the optimal polymer and additive concentrations were determined. The first step of the experimental work was investigating effects of the pre-conditioning parameters on the mechanical and rheological properties of the raw materials supplemented with FT-IR and SEM measurements. Based on the results of the first step PC/PA blends were produced in the presence of polyethylene and without it. Introduction of polyethylene into the blends governed by the experimental additives as their physical required carrier material. Both mechanical and rheological parameters were studied for all the blends supported by SEM (EDX) and XRD.

Effects of the preparatory operations

Having high and relatively high moisture adsorption capacity both PA and PC raw materials were needed to be dried before processing. Water content of the granules evaporates at processing temperature above 200 °C resulting in the appearance of bubbles in the extruded strings, thereby mechanical properties of the product deteriorate.

To determine the optimal parameters for pre-conditioning of the raw materials, the granules were dried in an oven at 60 °C, 80 °C, 105 °C and 120 °C for 4 h, and then the moisture content was measured. Figure 1 showed increasing moisture content of the PC at higher drying temperature that was considered to be caused by evaporation of light components and plasticizers in the polymer during drying. PA moisture content continuously reduced with increasing drying temperature but above 80 °C the process was no longer so significant. The whole water content of PC and PA reached the minimum value at a drying temperature of 80 °C. Extent of possible thermal degradation in the presence of oxygen combined with long drying time and high temperature was examined by FT-IR spectroscopy at each drying temperature before and after moisture content measurement [7]. No significant thermal degradation for either PA or PC was revealed on FT-IR spectra for the undried granules and for the two outer temperatures showed in Fig. 2. as new absorption peaks did not appeared. Based on moisture contents and FT-IR results and considering economic aspects of preconditioning, the PC and PA granules were dried at both 60 °C and 80 °C for 4 h before processing and definitive drying temperature was determined from characteristics of the extruded samples from pure polymers detailed in the followings.

Mechanical properties (tensile strength (Fig. 3–top); elongation at break (Fig. 3–bottom)) were significantly improved for both PC and PA after the higher drying temperature. The tensile strength increased by 15–20% for both polymers, up

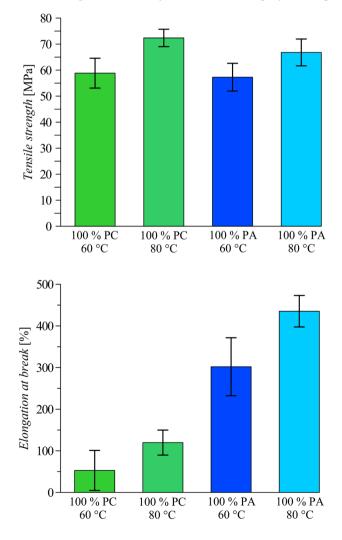


Fig. 3 Mechanical properties of the raw materials at different drying temperature

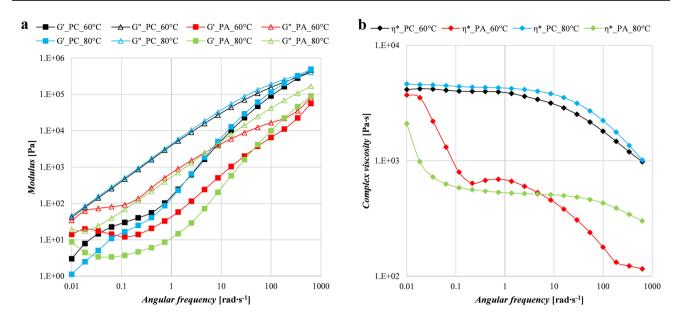


Fig. 4 a Storage and loss modulus b complex viscosity of PC and PA conditioned at different temperatures as a function of angular frequency; T = 230 °C, $\gamma = 10\%$

to 72.4 MPa for PC and 66.8 MPa for PA. Furthermore the standard deviation of the results also decreased. Elongation at break of PA increased from 300% to over 430%, and the elongation of PC more than doubled. Elongation at break of the raw materials dried at 60 °C was characterized by extremely high standard deviations. Significant difference among the parallel measurements was probably caused by bubbles in the extruded samples derived from evaporation of the residual moisture content of the granules during process-ing. Samples having low tensile strength and elongation at break were broken exactly at the bubbles being the weakest parts during tensile testing.

Considering the results of frequency sweep of raw materials (Fig. 4), polycarbonate was concluded to be less sensitive to the temperature of pre-conditioning inspite of the fact that both PC and PA have basically hygroscopic behaviour. Generally, moduli and complex viscosity curves of the PC were more balanced compared to the curves of PA.

Crossover frequency was observed only in the case of polycarbonate (Fig. 4a) at both temperatures of preconditioning indicating the more dominant elastic response over the crossover point. PA possessed more viscous behaviour than PC since loss moduli were higher than storage moduli as the function of angular frequency in the whole range examined.

Rising of the conditioning temperature did not make complex viscosity of PC (Fig. 4b) significantly changed and PC showed a Newtonian plate typical for un-crosslinked thermoplastic materials. Great differences in the storage moduli and complex viscosities of polyamide conditioned at different temperatures and measured over 200 °C were supposed to be related to post-condensation reactions and/or degradation in initial stage not being detectable by FT-IR at room temperature [16].

Properties of uncompatibilized blends

In the next step blends in different composition without additive (PC/PA-80/20; 60/40; 40/60; 20/80) were prepared in order to determine a relevant PC:PA ratio applying preconditioning at both 60 °C and 80 °C. Regardless of the drying temperature extruded sample suitable for mechanical tests could only be drawn from the composition of PC/PA80/20 wt%. For the other compositions polymer melt exiting the

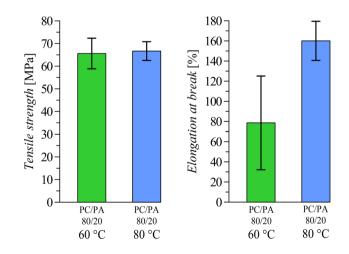


Fig. 5 Mechanical properties of the 80/20–PC/PA blend at different raw material drying temperature

tool immediately began to distended (even up to 2 cm in diameter), the string suddenly thinned and snapped caused by pulling.

Mechanical properties (tensile strength (Fig. 5–left); elongation at break (Fig. 5–right)) improved at the higher drying temperature meanwhile standard deviation of the results decreased for both. Tensile strength of the blend made from raw materials dried at 80 °C decreased by 8% compared to pure PC and was almost identical to pure PA. Elongation at break of the blend having high PC content was closer to elongation at break of PC but for PA it was still improved by 33% compared to pure PC.

SEM images were also taken of the fractured surface of the blends supplementary to the tensile tests. Figure 6a showed that the specimen fractured at the bubble during tensile testing, therefore probably the presence of bubbles and large radial cracks reduced the mechanical properties. Tensile strength of the blend achieved the strength of pure PA while elongation at break was better than that of the neat PC but significantly lower compared to PA. To determine the qualities of the domains with different morphology PC was selectively extracted from the blend with dichloromethane [17]. Figure 6c and d showed that granular particles with highly inhomogeneous size distribution disappeared from the blend. Selective extraction was supported by EDX analysis. The N content of the original 80/20 composition blend was only 0.11 wt% while after the dichloromethane treatment it increased to 1.58 wt% confirming the selective dissolution of granular PC particles. PA segments in the PC matrix Fig. 6b located in a roughly concentric circle within the sample indicated inadequate dispersion and separation of the two components in the melt. The crystallinity of the

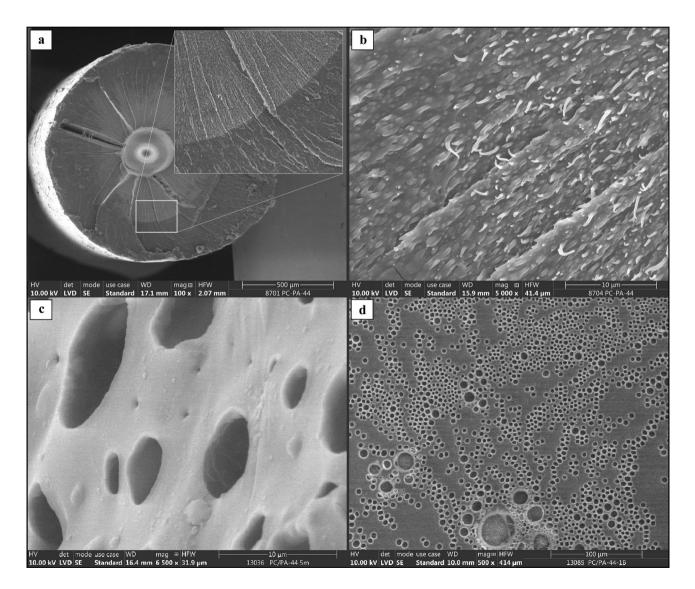


Fig. 6 SEM images from the fractured **a**, **b** and extracted **c**, **d** surface of the $\frac{80}{20-PC/PA}$ blend (left- $100 \times and \frac{800 \times zoom}{right-5000 \times zoom}$; c- $6500 \times zoom$; d- $500 \times zoom$)

blend was determined by X-ray diffraction analysis [18]. Due to addition of the semi-crystalline PA, the blend containing 80 wt% PC had only 66% amorphous phase so it can be concluded that the crystallization tendency of the polycarbonate increased in the presence of the polyamide. As a conclusion of the results achieved sofar raw materials were decided to be dried at 80 °C for 4 h before extrusion moulding of compatibilized blends in the next step of the experimental work.

Effect of a third component

Having a concept of the compatibilization to apply the experimental additives in the form of a masterbatch based on polyethylene; PE was added to the PC/PA blends as a third component in order to investigate effects of the polyethylene itself. The concentration of PE was set to 10 wt% of PC in all cases. PE was aimed to be selected by its characteristics,

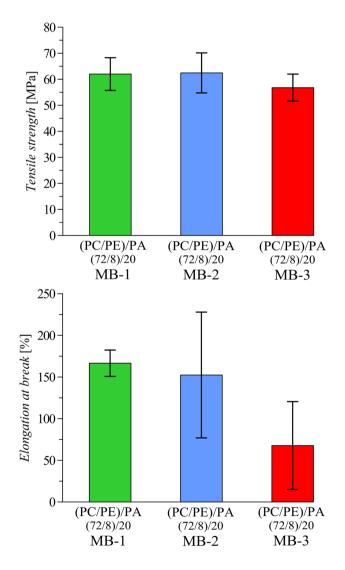


Fig.7 Effect of PE types on the mechanical properties of PC/PA blend

primarily based on melt flow index (MFI). Three polymers with different viscosities derived from different molecular weights were selected to be studied. Masterbatches were dried at 80 °C for 4 h similar to the neat polymers before extrusion moulding. Taking processing experiments conducted on PC:PA ratios into consideration only blends with 20 wt% PA were investigated. Mechanical and rheological characteristics were measured (Figs. 7, 8 and 9) in order to select the HDPE type for preparation of masterbatches containing the experimental compatibilizing additives.

Regarding to tensile strength (Fig. 7–top) however the blend with MB-2 had the highest value but that was only 0.45 MPa higher than the strength of the MB-1 containing blend, moreover the standard deviations of the results were also higher. In terms of elongation at break (Fig. 7–bottom) MB-1 had the best result and the smallest standard deviation. Elongation at break of the MB-1 containing PC/PA blend was almost 15% higher than in case of MB-2 and the standard deviation was nearly one-fifth. The worst mechanical properties belonged to the blend extruded with MB-3 masterbatch probably because of the lowest molecular weight among the polyethylenes resulting in plasticizing effect.

Capillary rheometry was used to study characteristics of the blends in the shear rate range below 10,000 1/s, completely covering the extrusion moulding range, moreover provides information on injection moulding. Viscosity (Fig. 8) decreased almost linearly in the investigated range. There was a slight difference only in the case of MB-3, accompanied by the steepest drop of the viscosity curve. Results of the blends with MB-1 and MB-2 run roughly in parallel correlating with the MFI values of the polyethylenes.

Slight differences were noticed obviously either in the values of moduli or complex viscosities of blends with masterbatches (Fig. 9) containing various types of polyethylene. It was important to emphasize that the curves of every masterbatch containing blend basically show similarity with the curves of polyamide instead of polycarbonate. That

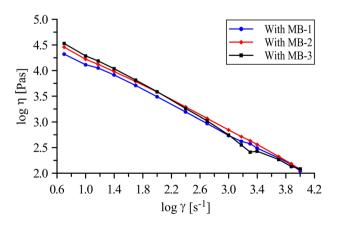


Fig. 8 Effect of PE types on the viscosity curves of PC/PA blend

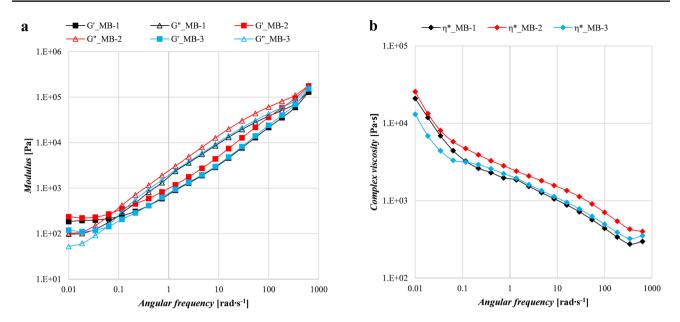


Fig. 9 a Storage and loss modulus b complex viscosity of PC/PA masterbatches differed in polyethylene type as a function of angular frequency; T = 230 °C, $\gamma = 10\%$

interesting phenomenon was supposed to indicate higher degree of plasticizing effects in the blends.

Both storage and complex viscosities of masterbatch based blends (Fig. 9) were higher than the neat PC and PA (Fig. 4) conditioned at any temperatures. The reason behind the increment of complex viscosity and storage modulus at lower frequencies was supposed to be the polyethylene content that may contribute to better homogenization of polyamide and polycarbonate. However, according to Wang et al. [13] the formation of PC-PA copolymer at high processing temperature may cause the increase of G' and η^* . Furthermore, values of G' were highlighted to be higher than G' at low frequencies indicating the pseudo-solid behaviour [19] of PC/PA blends also confirming the better interaction between the components in the presence of polyethylene.

PE content improved processability of the blend by widening the processing window as well as some of its mechanical properties but did not eliminate separation of the PC and PA segments. In Fig. 10 SEM images supported that statement by showing inhomogeneity of the structure and weak interfacial connection between the segments. Similar to a blend without PE both a fibrous and a granular region was separately noticed.

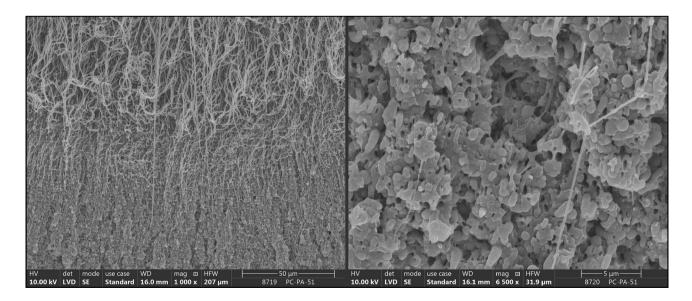
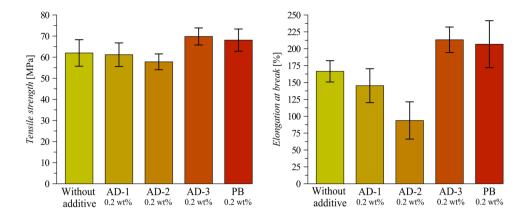


Fig. 10 SEM images from the fractured surface of PC/PA blend extruded with MB-1 (left– $1000 \times zoom$; right– $6500 \times zoom$



Effect of compatibilization

The best mechanical properties belonged to the MB-1 containing blend while comparing the effect of the three HDPE types therefore that type of HDPE was selected for masterbatches with compatibilizing additives. PC:PE ratio was 9:1 and the processing parameters were the same as aformentioned. Three olefin-maleic-anhydride copolymer based experimental compatibilizing additives and the commercial one were used in 0.2 wt% concentration in the PC/PA blends. Mechanical properties of the compatibilized blends (Fig. 11) were compared to the extruded blend without additive considering as the reference.

As a result of masterbatches containing AD-1 and AD-2 experimental additive both tensile strength (Fig. 11-left) and elongation at break (Fig. 11-right) of the blends decreased related to the reference. Best mechanical properties were achieved in the presence of AD-3 since tensile strength was improved by 12% (to 69.8 MPa) and elongation at break was increased by 20% (to 206.7%). As a result of addition of the AD-3, tensile strength was about 5% higher than the strength of neat PA and only 2.5 MPa less than the strength of neat PC. Commercial compatibilizing additive slightly increased strength and elongation at break and standard deviation of the results was also higher than in case of AD-3. Considering the structural characteristics of the additives it can be concluded that the presence of half-ester functional groups was not advantageous in terms of tensile strength, but it was particularly disadvantageous in terms of elongation at break (AD-1, AD-2). The presence of N-containing functional groups in the additives was proved to be favourable with the anhydride groups (AD-3), it even surpassed effect of the commercial additive at the same concentration.

Viscosity curves of Fig. 12 increased as a result of the compatibilizing additives compared to the blend without additive. AD-1 increased the viscosity to the lowest extent and the highest values belonged to the blend prepared with the commercial additive. Trend of the viscosity curves

was not being substantially modified in the presence of additives over the additive-free blend suggested that the processing parameters are not required to being changed during injection moulding of the blends when experimental compatibilizers are used.

Addition all of the compatibilizing additives led to the increase of moduli (Fig. 13a) and especially the complex viscosities (Fig. 13b) of PC/PA blends compared to the additive-free one. Enhanced interfacial interaction promoted the entanglement of the two polymers in the presence of the compatibilizing additives. Increased storage moduli and complex viscosities indicated increased melt strength of the blend as a result of compatibilizing, and therefore, providing higher stability of the polymer melt during processing and higher resistance against shearing effects. Compatibilizers made properties measured more balanced especially in the run of complex viscosity curves recorded at lower frequencies.

Property modifying effect of the additives were supported by FT-IR spectra of additive free and compatibilized blends as the most relevant characteristic peaks for

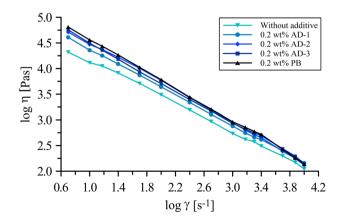


Fig. 12 Effect of compatibilizing additives on viscosity curves of PC/ PA blends

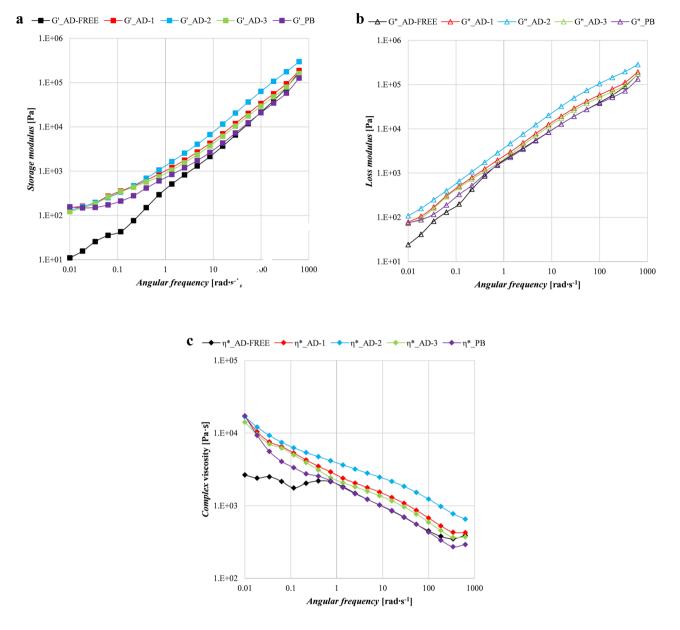


Fig. 13 Effect of experimental additives on the **a** storage modulus **b** loss modulus and **c** complex viscosity of PC/PA blends as a function of angular frequency; T = 230 °C, $\gamma = 10\%$

both hydrocarbon and carbonyl functional groups were the same without indicating any chemical reactions independently from the presence of the additive (Fig. 14).

Based on the results of mechanical and rheological properties it can be concluded that property modifying effects of the additives in the investigated concentration presumably originated from the physical character of the compatibilizers caused by their chemical structures. Even compatibilizing additives maintained the granular and fibrous domains either supported by SEM images (Fig. 15) so that the components still separated in the melt and the sharp interfaces could not be completely eliminated. However, the regions with different morphologies were not so sharply separated in the presence of additives compared to the samples without additive, as there were many fibrous segments in the granular domain probably explaining the improvement of elongation at break either.

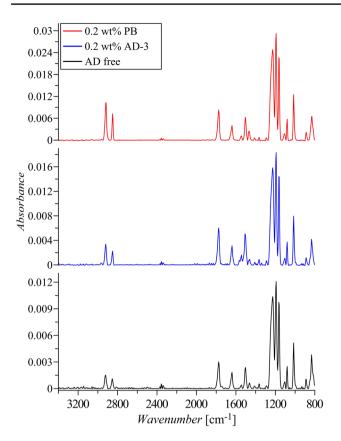


Fig. 14 FT-IR spectra of PC/PA blends without additive, with experimental additive and commercial compatibilizer

Conclusion

In that paper tailoring of polycarbonate and polyamide were investigated. The biggest barrier to combination of those polymers is the lack of dissolution in each other in any composition and temperature range, therefore, mechanical and rheological properties of their blend have to be improved with different compatibilizing additives.

Having high moisture adsorption capacity both PC and PA, optimal parameters for the pre-condition of the raw materials before processing were determined first. Several drying temperatures and duration were tested and possible thermal degradation was followed by FT-IR.

Blends were prepared by extrusion moulding and compatibilizing additives at a concentration of 0.2 wt% were added in the form of a PE based masterbatch. Effects of different types of HDPE were investigated and PE was selected based on the tensile and rheological test results for preparation of masterbatches containing the compatibilizers.

Two additives were found to be effective in improving the mechanical properties and one of them was an experimental olefin-maleic-anhydride copolymer based additive (AD-3). Tensile strength of the compatibilized blend approached the value of neat PC, while elongation at break improved by 20% compared to the blend without additive and by more than 80% compared to neat PC. However, sharp interfaces on SEM images were not completely eliminated in the compatibilized blends; but the domains with

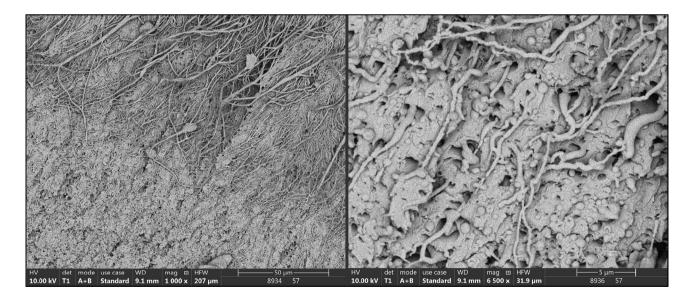


Fig. 15 SEM images from the fractured surface of PC/PA blend with AD-3 experimental compatibilizing additive

fibrous and granular morphology were less separated than in the blends without additive. Increased storage moduli and complex viscosities indicated increased melt strength of the blend as a result of compatibilizing, and therefore, providing higher stability of the polymer melt during processing and higher resistance against shearing effects.

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Authors' contributions All authors contributed to the study conception and design. Material preparation, measurements and analysis were performed by Tamás Kovács, Lilla Simon-Stőger, Balázs Heller and Csilla Varga. Rheological tests were performed by Lilla Simon-Stőger and FT-IR analysis were performed by Balázs Heller. The first draft of the manuscript was written by Tamás Kovács and all authors commented on previous versions of the manuscript. All authors read and approved the final manuscript.

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