# Publication IV

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IV

Full Paper

Summary: The compatibilization of polyethylene/polyaniline (PE/PANI) blends and the preparation of plasticized PANI/camphorsulfonic acid (CSA) complexes suitable for melt blending were studied. Rheological properties of the components essentially affected the morphology of the blend and thereby the electrical conductivity. The hydrogen bonds between the PANI complex and the functionalized metallocene PE used as compatibilizer compensated the unfavorable viscosities of the components. Mechanical properties of PE/PANI blends were improved, and electrical conductivity of the blends remained the same or increased through addition of functionalized metallocene polyethylene. Plasticized PANI/CSA complex with good electrical conductivity was successfully prepared.
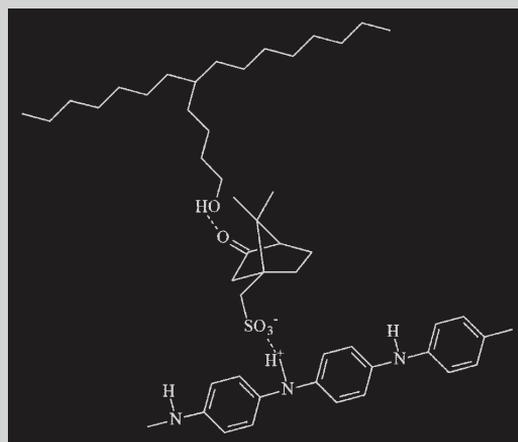
Compatibilization of PANI/CSA complex and OH-functionalized polyethylene.

Compatibilization of Conductive Polyethylene/Polyaniline Blends

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Introduction

Intrinsically conductive polymers have been studied intensively during recent decades. Among these special polymers, polyaniline (PANI) has attracted attention for its excellent chemical, electrical, and optical properties. Although addition of sulfonic acids as dopants causes intractable PANI to become soluble[1] and, after plasticizing,[2] it can even be melted, the PANI complex still remains brittle. In general, applications of PANI, including conductive paints and thin films as in organic LEDs, batteries, and sensors, are based on solution processing. The field of applications of PANI can be extended by blending it with polymers having good mechanical properties. The properties of the blends are a combination of those of the individual components and depend on the morphology of the blend. Compared with the matrix polymer, the blends often show an undesirable loss of

thermoplastic/elastomeric behavior, usually proportional to the PANI content in the mixture.[3] The research on PANI blends has mainly focused on solution blending, where a solvent is used as a third component.[4−6] It is then easier to control the morphology and minimize the PANI content so as to achieve good electrical and mechanical properties of the blend.

Though melt mixing in extruders has provided a reasonably economical way to produce a wide range of polymer blends, it has less often been applied for PANI blends due to the difficulty of producing a truly plasticized PANI complex and controlling the morphology of the blend. Improvement in the mechanical properties of a blend requires that the phase of the matrix polymer be continuous, and that there is sufficient adhesion between the phases. In melt mixing, phase separation can be controlled by adjusting the viscosity ratios[7] and shear rates to achieve a

morphology, in which both the phases are continuous. In some studies, PANI complex was first solution-blended with one polymer, and this blend was then melt-blended with a second polymer.[8] In such ternary blends, double percolation can be exploited to minimize the PANI content in the blend.

There are only a few studies on the mechanical properties of PANI blends with polyethylene due to the lack of adhesion between the PANI phase and polyethylene matrix in the melt processing. Mostly the studies are concentrated to enhance the conductivity of the blend, which is done by using dopants that own long alkyl chains.[9] Since adhesion is essential to achieving good mechanical properties of polymer blends, compatibilization is a necessary part of blending.[10] Adhesion in PANI blends is usually achieved with polar polymer matrices since the PANI complex itself is polar. With a nonpolar polymer matrix, PANI is generally doped with dodecylbenzene sulfonic acid (DBSA), which has a nonpolar tail. Zilberman et al.[11] have used copolyamide 6/6.9 as a compatibilizer in the blends of PANI/TSA and LDPE. The highest conductivity they reached was ca. 10^{-4} S · cm^{-1}, when blend composition was PANI/TSA 20 wt.-%, and the ratio of LDPE/copolyamide 75/25, but no mechanical properties of the blends were reported. Wan-Cheng et al.[12] have used ethylene/vinyl acetate copolymer (EVA) as a compatibilizer in the blends of PANI/TSA and LDPE. They managed to reach excellent conductivities of the blends, up to 10^{-1} S · cm^{-1}, but the mechanical properties of the compatibilized blends were worse than in pure LDPE. The tensile strength was reduced ca. 26% and elongation 66% at PANI content of 15 wt.-%. Zhang et al.[13] have used EVA as compatibilizer in blends of PANI/DBSA and LDPE. In the blends of 15 wt.-% PANI and 10 wt.-% EVA, the conductivity was ca. 10^{-6} S · cm^{-1}. But again the addition of EVA did not improve the mechanical properties of the blends. The yield strength dropped from 7.75 to 5.75 MPa after the addition of 15 wt.-% of PANI. The addition of EVA slightly affected the yield elongation, but the mechanical properties of the blends were worse than in pure LDPE.

The primary aim of this study was to investigate the use of functionalized polyethylene as a compatibilizer between the PANI complex and polyethylene matrix in melt processing. Since polyolefins polymerized with metallocenes,[14,15] have better mechanical properties than polyolefins prepared with conventional Ziegler–Natta catalysts, owing to the better controllability of molar mass and distribution, the functionalized polyethylenes were polymerized with metallocene catalysts. Another advantage of metallocene catalysts is that functionalized monomers can be polymerized directly. The compatibilization is based on hydrogen bonding between PANI complex and functionalized polyethylene (Scheme 1).

In general, PANI exhibits the best conductivity in solution processing when doped with camphorsulfonic acid (CSA). The high conductivity of PANI/CSA complex is achieved due to the molecular recognition with solvent, i.e.,
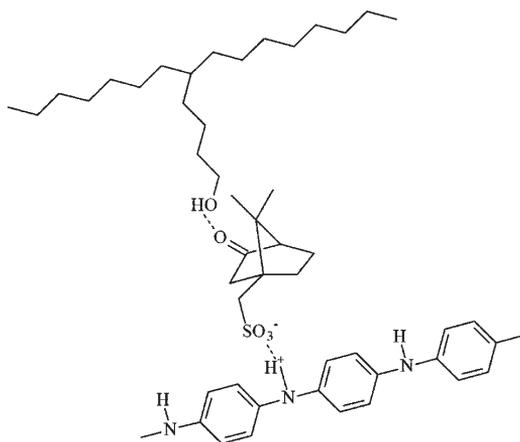
Scheme 1. Compatibilization of PANI/CSA complex and OH-functionalized polyethylene.

the solvent acts as a secondary dopant.[16–18] Relevant to this, a further aim was to study the possibility of preparing a plasticized PANI/CSA complex suitable for melt blending.

Experimental Part

Materials

The polyethylenes used as matrix component, LDPE (FA3220) and metallocene polyethylene (ME8168), and the maleic anhydride (MAH)-functionalized polyethylene PE-g-MAH (ME0420) used as compatibilizer, were supplied by Borealis Polymers Oy. Functionalized metallocene polyethylenes were prepared by the method described previously.[19] Functionalities in the prepared metallocene polyethylenes were hydroxyl groups (OH; $\bar{M}_w$ 144 100 g · mol^{-1}, 0.2 mol-%) and carboxylic acid groups (COOH; $\bar{M}_w$ 117 100 g · mol^{-1}, 0.2 mol-%). Comonomers 10-undecen-1-ol (96%) and 10-undecenoic acid (97%) were purchased from Fluka. The rac-ethylenebis(1-indenyl)zirconium dichloride catalyst [Et(Ind)2ZrCl2] and cocatalyst methylaluminoxane (10 wt.-% solution in toluene) were obtained from Witco. Plasticized PANIs doped with p-toluenesulfonic acid (p-TSA) and phenolsulfonic acid (PSA), and two different unplasticized PANIs doped with CSA, designated PANI/CSA1 and PANI/CSA2, in powder form were supplied by Panipol Ltd. Unplasticized PANIs were plasticized in cooperation with Panipol Ltd. The plasticization of PANI/CSA complexes was carried out in a melt mixer with ZnO$_2$ and DBSA as plasticizing agents.

Blending

The blends were prepared with a co-rotating twin-screw midiextruder (DSM; capacity = 16 cm^3; screw length $L =$ 150 mm) under nitrogen atmosphere. The screw speed was 60 rpm and temperature 200 °C. The mixing time was 3 min.

The content of the PANI complex in blends was 15 wt.-% in studies on interactions between the phases. The functionalized metallocene polyethylene content was 2 or 18 wt.-% in investigations of the effect of compatibilizer on the rheological properties. In Table 1 the blend compositions are depicted with the sample designations.

Characterization

Electrical conductivity was measured from extrudates by the four-probe method (Keithley Current Source 220 and Keithley Electrometer 6514).

The viscosity of the components was measured with a Goettfert Rheograph 2002 capillary rheometer according to standard ISO 11443:1995(E). The diameter of the die was 1 mm and length 30 mm. Viscosities were measured at 200 °C.

Mechanical properties of the blends were characterized from injection-molded samples (mini-injection molding machine DSM). Tensile tests were done with an Instron 4202 testing machine using a test speed of 5 mm · min^{-1} and with specimen type 1BA according to the standard ISO 527:1993(E).

Thermal analyzes with differential scanning calorimetry (Mettler Toledo DSC 821) using a heating rate of 10 °C · min^{-1} from 0 to 220 °C were carried out to study the miscibility and interactions of the blend components.

The morphology of the blends was characterized with field emission scanning electron microscope JEOL JSM-6335F. The extrudates and tensile test specimen were fractured after cooling in liquid nitrogen, and the fractured surfaces were sputter-coated with chromium under argon. The electron micrographs were taken using an acceleration voltage of 5.0 kV.

Results and Discussion

Conductivity

PANI/TSA, PANI/PSA, and PANI/CSA complexes were prepared in the laboratory scale following the recipes of industrial processes of Panipol Ltd., and the plasticization of the complexes was more important than conductivity. In blending, the PANI content was made high enough that the

Table 1. Blend compositions for various samples.

Sample	Blend composition	Amount in the blend
		wt.-%
PE	LDPE FA3220	–
ME	HDPE ME8168	–
OH	PE-*co*-OH	–
COOH	PE-*co*-COOH	–
MAH	PE-*g*-MAH	–
PE/OH2	LDPE/PE-*co*-OH	98/2
PE/COOH2	LDPE/PE-*co*-COOH	98/2
PE/MAH2	LDPE/PE-*g*-MAH	98/2
PE/OH18	LDPE/PE-*co*-OH	82/18
PE/COOH18	LDPE/PE-*co*-COOH	82/18
PE/MAH18	LDPE/PE-*g*-MAH	82/18
ME/TSA	HDPE/(PANI/TSA)	85/15
PE/TSA	LDPE/(PANI/TSA)	85/15
PE/TSA/OH2	LDPE/(PANI/TSA)/PE-*co*-OH	83/15/2
PE/TSA/COOH2	LDPE/(PANI/TSA)/PE-*co*-COOH	83/15/2
PE/TSA/OH18	LDPE/(PANI/TSA)/PE-*co*-OH	67/15/18
PE/TSA/COOH18	LDPE/(PANI/TSA)/PE-*co*-COOH	67/15/18
PE/PSA	LDPE/(PANI/PSA)	85/15
PE/PSA/OH2	LDPE/(PANI/PSA)/PE-*co*-OH	83/15/2
PE/PSA/COOH2	LDPE/(PANI/PSA)/PE-*co*-COOH	83/15/2
PE/PSA/OH18	LDPE/(PANI/PSA)/PE-*co*-OH	67/15/18
PE/PSA/COOH18	LDPE/(PANI/PSA)/PE-*co*-COOH	67/15/18
PE/CSA1	LDPE/(PANI/CSA1)	85/15
PE/CSA1/OH2	LDPE/(PANI/CSA1)/PE-*co*-OH	83/15/2
PE/CSA1/COOH2	LDPE/(PANI/CSA1)/PE-*co*-COOH	83/15/2
PE/CSA1/MAH2	LDPE/(PANI/CSA1)/PE-*g*-MAH	83/15/2
PE/CSA1/ME2	LDPE/(PANI/CSA1)/ME	83/15/2
PE/CSA1/OH18	LDPE/(PANI/CSA1)/PE-*co*-OH	67/15/18
PE/CSA1/COOH18	LDPE/(PANI/CSA1)/PE-*co*-COOH	67/15/18
PE/CSA1/MAH18	LDPE/(PANI/CSA1)/PE-*g*-MAH	67/15/18
PE/CSA1/ME18	LDPE/(PANI/CSA1)/ME	67/15/18
PE/CSA2	LDPE/(PANI/CSA2)	85/15
PE/CSA2/OH2	LDPE/(PANI/CSA2)/PE-*co*-OH	83/15/2
PE/CSA2/COOH2	LDPE/(PANI/CSA2)/PE-*co*-COOH	83/15/2
PE/CSA2/OH18	LDPE/(PANI/CSA2)/PE-*co*-OH	67/15/18
PE/CSA2/COOH18	LDPE/(PANI/CSA2)/PE-*co*-COOH	67/15/18

blends would be conductive, and the research was focused on compatibilization properties of functionalized metallocene polyethylenes. Conductivity of PANI complexes could not be measured with the same instrument construction used for extrudates, due to the brittleness of PANI complexes. The conductivities of the blends were surprisingly high for melt blending (Figure 1). And unexpectedly, blends with PANI/CSA complexes had the best conductivities even without solvent as a secondary dopant. The conductivities obtained in PANI/CSA1 blends were slightly above electrostatic dissipation.

It is interesting that the addition of functionalized metallocene polyethylene did not disturb the percolation of the PANI complex, though the viscosity of the functionalized metallocene polyethylenes differs from that of the LDPE matrix. With metallocene polyethylene as matrix and without compatibilizer, the blend was an insulator even with a content of 15 wt.-% PANI complex. From Figure 2 it can be seen that the viscosities of LDPE and PANI/TSA complex are closer than the viscosities of the metallocene polyethylene and the PANI/TSA complex, and this explains the conductivity obtained in blends of LDPE and PANI complexes and not in blends of metallocene polyethylene and PANI complexes. Further experiments were made only with the LDPE-matrix, and mechanical properties were tested only from the conductive blends as we aimed for a combination of appropriate electrical conductivity with good mechanical properties of the blend.

In blends with PANI/TSA complex, a small amount of functionalized metallocene polyethylene increased the conductivity. The PANI complex is probably located in the functionalized metallocene polyethylene phase or at the interphase, and double percolation occurs. With 18 wt.-% of polyethylene with carboxylic acid functionality, conductivity dropped due to the unfavorable rheological properties of the blend components. The conductivity of PANI/PSA blends, in contrast, did not change much with the addition of compatibilizers, and remained at the same level even at the higher content of compatibilizers. The PANI/PSA complex contains more functional groups capable of forming hydrogen bonds with the functionalized metallocene polyethylene, and these bonds compensate the unfavorable rheological properties. The same is seen with PANI/CSA1 blends. Unless OH- or COOH-functionalized metallocene polyethylene was used, even a 2 wt.-% of unfunctionalized metallocene polyethylene in the blend disturbs the percolation of PANI/CSA1 complex and the blend is an insulator. The PANI/CSA2 complex, in turn, behaves more like the PANI/TSA complex: the conductivity of the PANI/CSA2 blends increases with the addition of functionalized metallocene polyethylene, but is lower than that of PANI/CSA1 blends, probably due to the different effect of the plasticizer on the PANI/CSA complexes.

Domenech et al.[20] found that MAH forms covalent bonds with the PANI backbone in PANI/DBSA complex. Our studies show that the drop in conductivity is significant
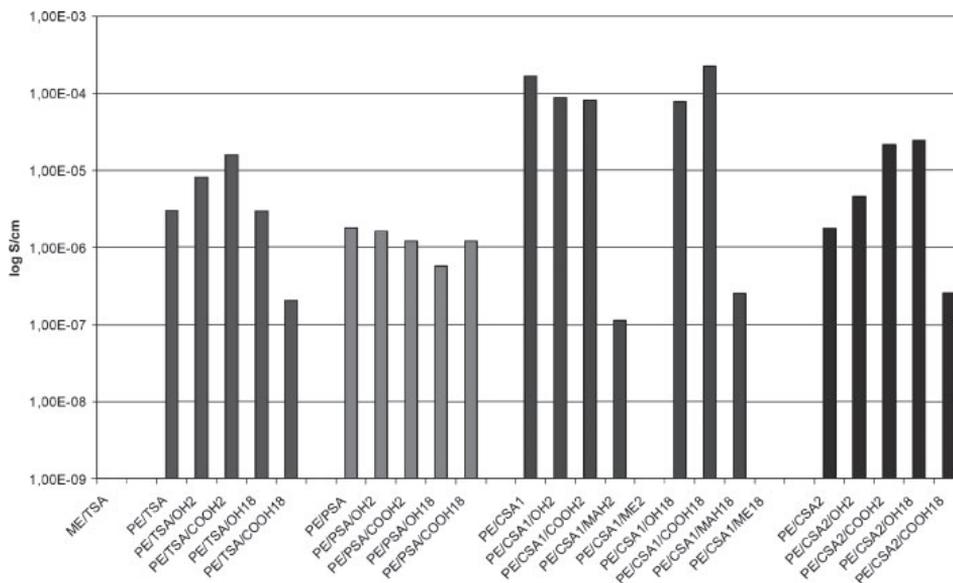

Figure 1. Electrical conductivity of PANI blends. The PANI complexes are designated with the dopant, the functionalized metallocene polyethylene with the functionality and amount in the blend, and LDPE matrix with PE.
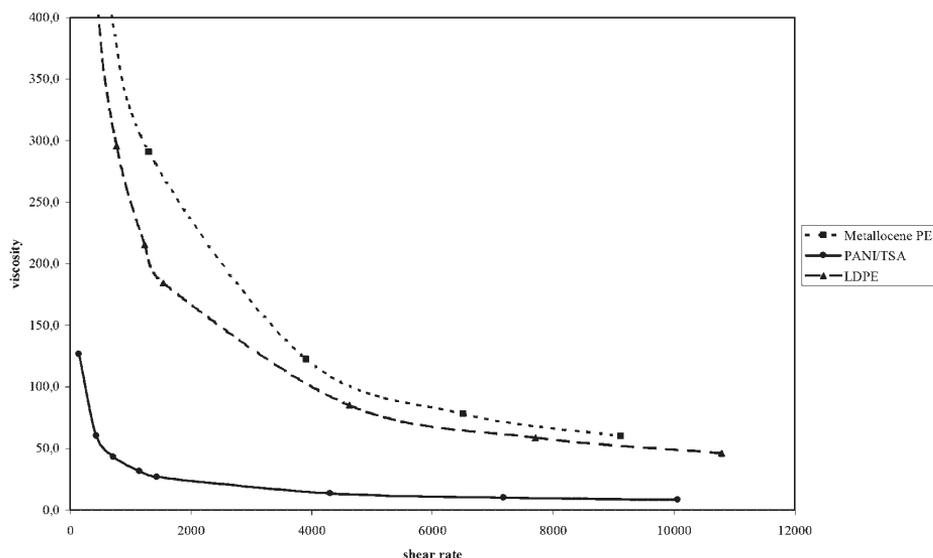
Figure 2. Viscosities of the blend components, metallocene polyethylene, PANI/TSA complex, and LDPE.

with MAH-functionalized polyethylene as compatibilizer. The result confirms that covalent bonds with PANI reduce the number of delocalized electrons in the PANI chain. Zhen et al.[21] reported that even hydrogen bonds may have a negative effect on the delocalization of electrons in the PANI chain. Our functionalized metallocene polyethylenes contain only 0.20 mol-% functionalities and the amount of hydrogen bonds formed is so low that the effect on the delocalization of electrons in the PANI chains is minimal. The possible hydrogen bonds with functionalized polyethylenes are difficult to observe with normal spectroscopic methods due to the large amount of N–H bonds formed by PANI backbone and sulfonic acids. However, the minimal amount of functionalities in polyolefins changes the polarity of the polyolefin. The surface of OH-functionalized metallocene poly(propylene) has 9% smaller contact angle than pure poly(propylene), with only 0.4 mol-% of functionality.[22]

Mechanical Properties

Mechanical properties of polyethylene decrease with the addition of the PANI complexes as shown in Figure 3. PANI complexes used in this study were so brittle that test specimens could not be prepared. Because the PANI complex is polar and the polyethylene matrix is nonpolar, there is a lack of adhesion between the different phases and the mechanical properties of the polymer matrix are negatively affected by the PANI complex. Compared with conventional Ziegler–Natta polyethylene LDPE, metallocene-catalyzed

polyethylenes have better mechanical properties. In the blends of LDPE matrix and functionalized metallocene polyethylene without PANI complex, the tensile modulus and strain are improved after 18 wt.-% addition of functionalized metallocene polyethylene (Figure 3).

In the PANI/TSA and PANI/PSA blends, the tensile modulus increases with the addition of functionalized metallocene polyethylenes. Slight increase can be seen in both the strength and strain. It is notable that when the PANI/PSA blend was compatibilized with 18 wt.-% of COOH-functionalized polyethylene, the mechanical properties were greatly improved without reduction of the electrical conductivity of the blend (see Figure 1).

Tensile modulus and strength are worse in the PANI/CSA1 blends than in the other PANI complex blends. The PANI/CSA complexes were post-plasticized with ZnO_2 and DBSA, and free DBSA has migrated into the matrix and functionalized polyethylene phases and plasticized them as well.[23] This did not occur when the PANI complexes were simultaneously doped and plasticized. The strain of the blends increases due to the plasticization effect. Better mechanical properties are achieved in PANI/CSA1 blends with MAH-functionalized polyethylene as compatibilizer, where the free plasticizer forms covalent bonds with MAH-functionalized polyethylene, but then the conductivities decrease.

The plasticizer is evidently better bonded in the PANI/CSA2 complex than in PANI/CSA1 complex, and mechanical properties of the blend are, therefore, clearly improved after the addition of functionalized polyethylenes. With
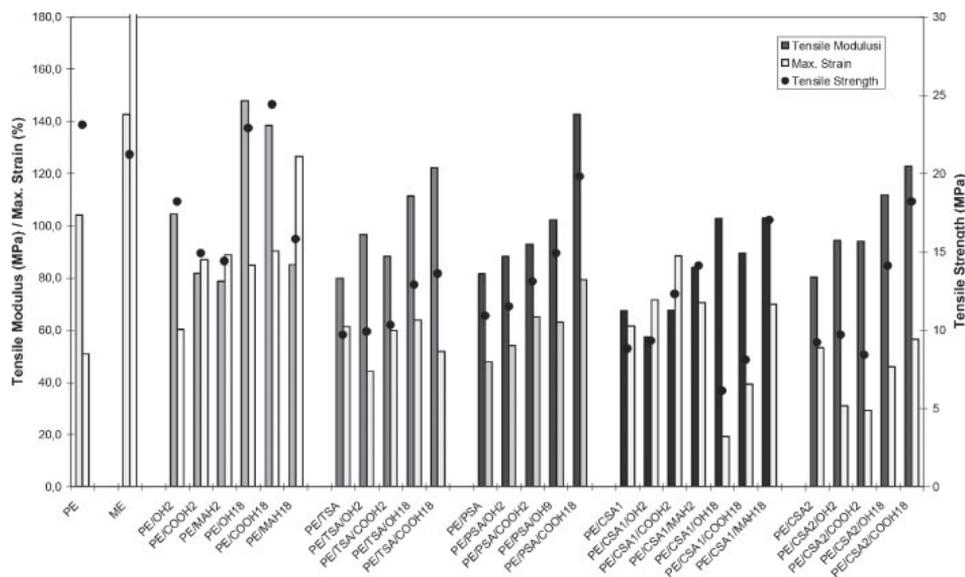
Figure 3. Mechanical properties of PANI blends. The PANI complexes are designated with the dopant, the functionalized metallocene polyethylene with the functionality and amount in the blend, and LDPE matrix with PE.

18 wt.-% OH-functionalized polyethylene as compatibilizer, both the modulus and strength are improved over that of PANI/CSA1 blends.

Thermal Properties

As shown in Figure 4, the melting temperature of polyethylene matrix is not affected by blending with the PANI complex. However, the crystallinity of the polyethylene matrix is decreased. As already noted above, free DBSA in the PANI/CSA1 complex plasticizes the matrix. The same

behavior is seen in the DSC measurements: Crystallinity of the polyethylene matrix is lowest when the blend contains PANI/CSA complexes.

The DSC curves (Figure 5 and 6) reveal that the LDPE matrix and functionalized metallocene polyethylenes are immiscible by showing two clear melting peaks. The melting temperatures of functionalized metallocene polyethylenes are shifted towards lower temperatures after the addition of PANI complex. It can be assumed that the PANI complex is located in the same phase as the functionalized metallocene polyethylene, which partly explains the high
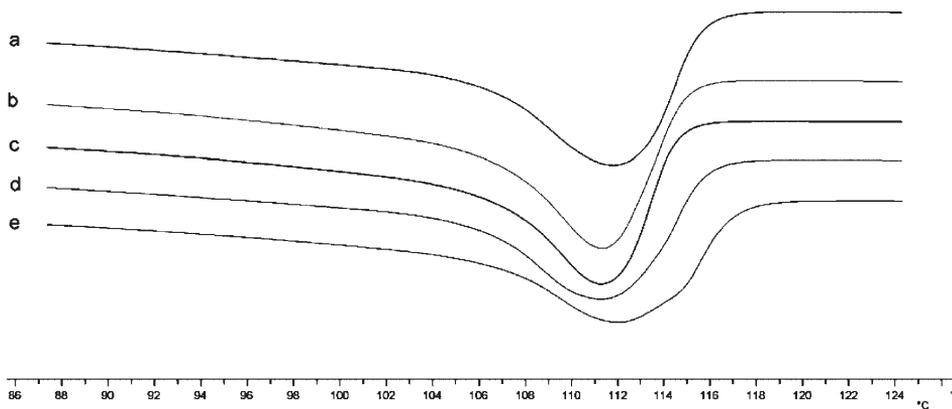

Figure 4. DSC curves of blends without compatibilizers: (a) LDPE, (b) PE/(PANI/TSA), (c) PE/(PANI/PSA), (d) PE/(PANI/CSA1), and (e) PE/(PANI/CSA2).
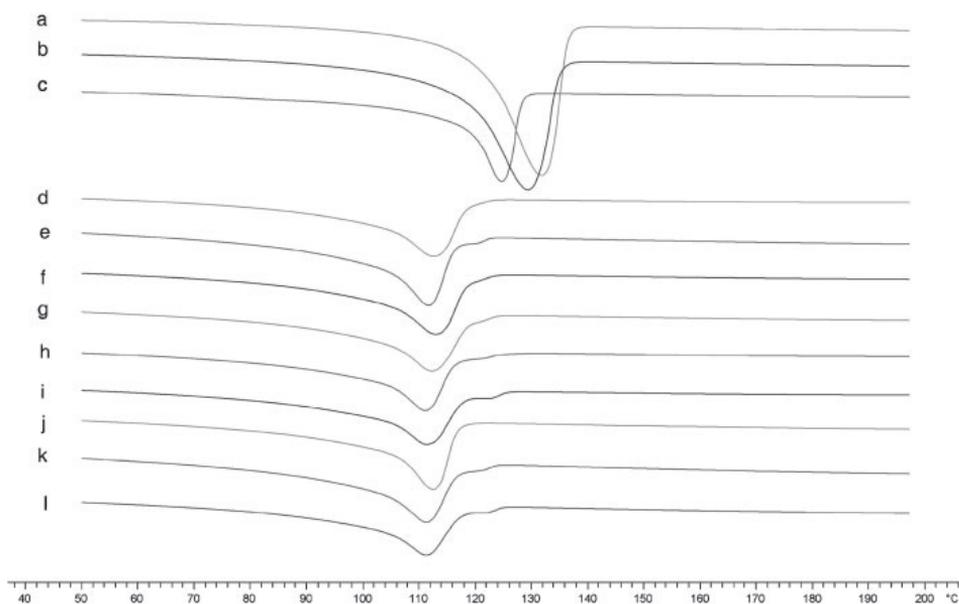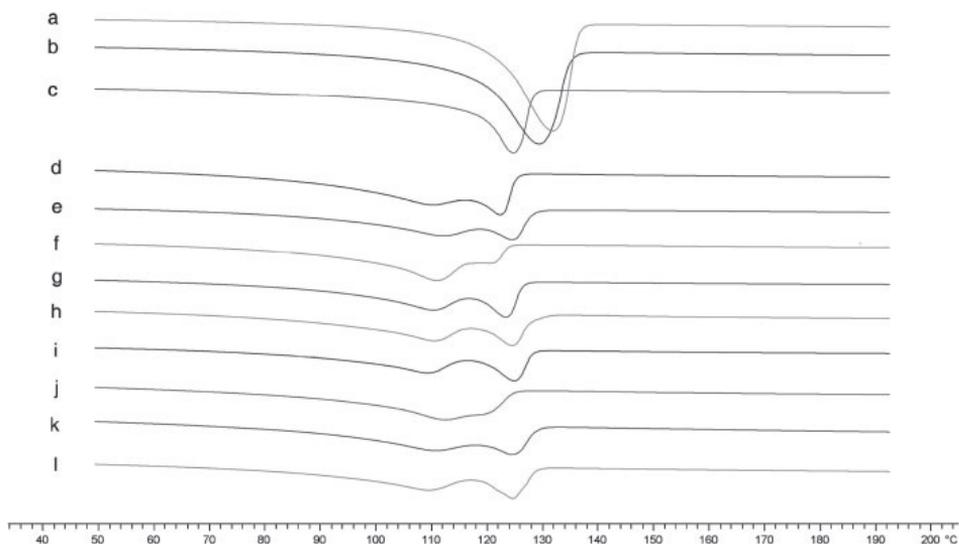
Figure 5. DSC curves of blends containing 2 wt.-% of functionalized polyethylene as compatibilizer: (a) OH-functionalized metallocene polyethylene, (b) COOH-functionalized metallocene polyethylene, (c) MAH-functionalized polyethylene, (d) PE/(PANI/TSA)/–OH, (e) PE/(PANI/TSA)/–COOH, (f) PE/(PANI/PSA)/–OH, (g) PE/(PANI/PSA)/–COOH, (h) PE/(PANI/CSA1)/–OH, (i) PE/(PANI/CSA1)/–COOH (j) PE/(PANI/CSA1)/–MAH, (k) PE/(PANI/CSA2)/–OH, and (l) PE/(PANI/CSA2)/–COOH.

Figure 6. DSC curves of blends containing 18 wt.-% of functionalized polyethylene as compatibilizer: (a) OH-functionalized metallocene polyethylene, (b) COOH-functionalized metallocene polyethylene, (c) MAH-functionalized polyethylene, (d) PE/(PANI/TSA)/–OH, (e) PE/(PANI/TSA)/–COOH, (f) PE/(PANI/PSA)/–OH, (g) PE/(PANI/PSA)/–COOH, (h) PE/(PANI/CSA1)/–OH, (i) PE/(PANI/CSA1)/–COOH, (j) PE/(PANI/CSA1)/–MAH, (k) PE/(PANI/CSA2)/–OH, and (l) PE/(PANI/CSA2)/–COOH.
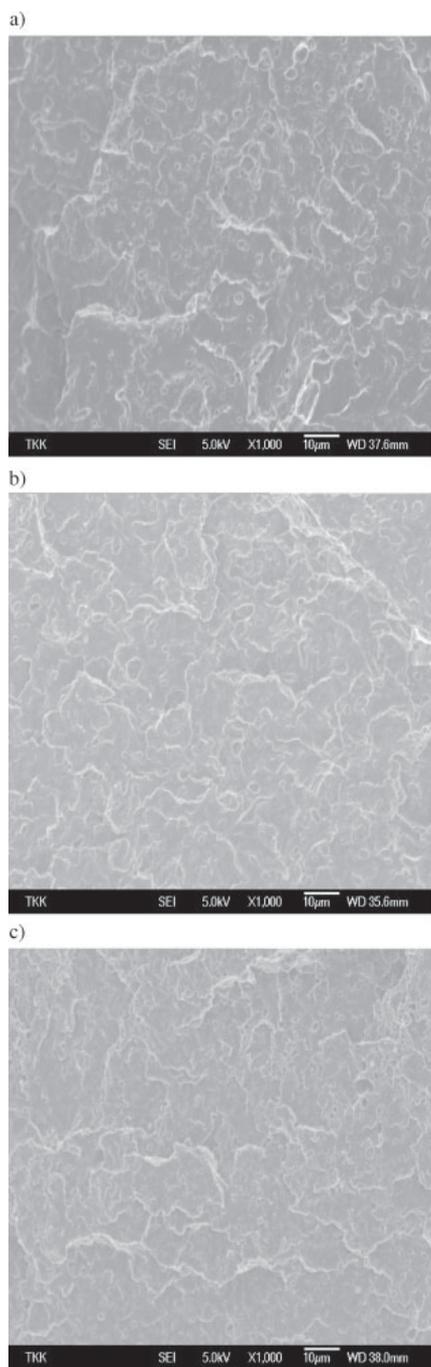
Figure 7. SEM images of PANI/PSA blends: (a) PE/(PANI/PSA), (b) PE/(PANI/PSA)–OH (2 wt.-%), and (c) PE/(PANI/PSA)–OH (18 wt.-%).
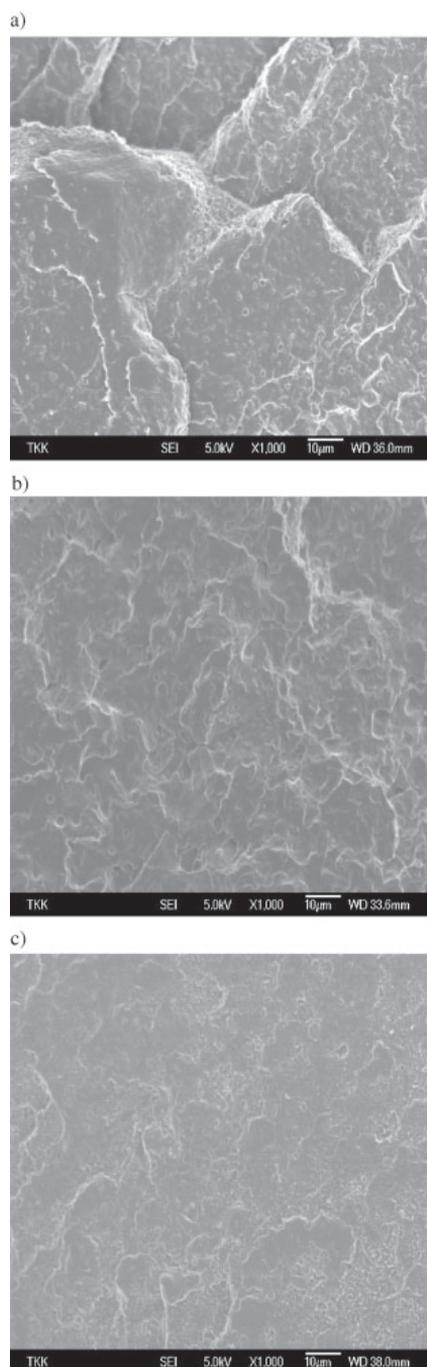
Figure 8. SEM images of PANI/TSA blends: (a) PE/(PANI/TSA), (b) PE/(PANI/TSA)–OH (2 wt.-%), and (c) PE/(PANI/TSA)–OH (18 wt.-%).
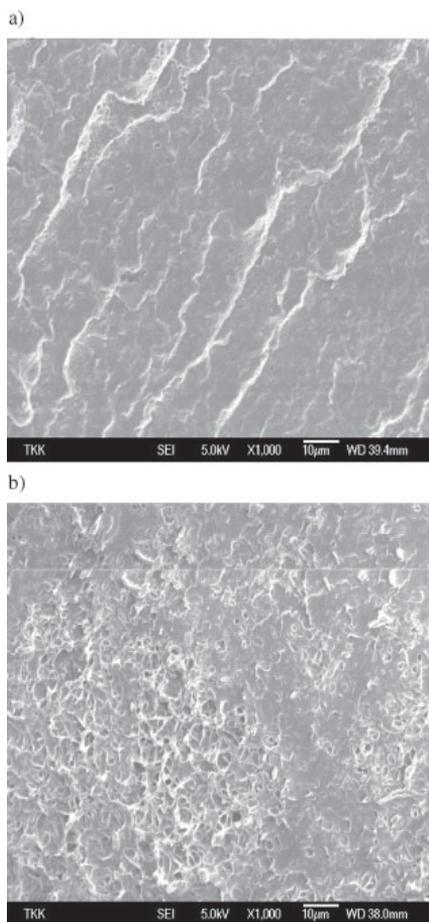
a)

b)

Figure 9. SEM images of PANI/CSA1 blends with PE-*g*-MAH and metallocene polyethylene: (a) PE/(PANI/CSA1)/–MAH (2 wt.-%) and (b) PE/(PANI/CSA1)/ME (2 wt.-%).

conductivity of the blends with 2 wt.-% of functionalized metallocene polyethylene. In the blends with 18 wt.-% of functionalized metallocene polyethylene, the unfavorable rheological properties hinder the double percolation. There are only small differences between hydroxyl and carboxylic acid functionalities.

Morphology

Scanning electron microscope (SEM) images show two different morphologies in the fracture surfaces of extrudates (Figure 7 and 8). In the blends without functionalized polyethylene, the PANI complex is mostly in the dispersed phase and, in the blends with functional polyethylene, network structure can be seen more clearly. The finer network structures are in the blends of PANI/PSA (Figure 7)
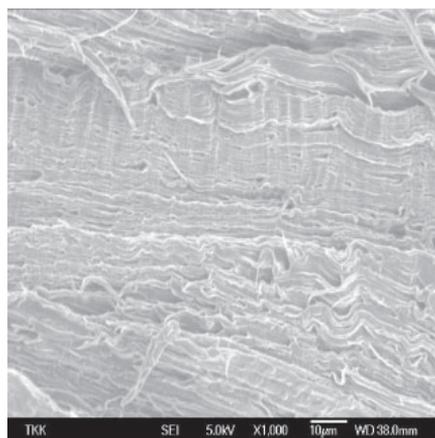

Figure 10. SEM image of PANI/CSA1 blend from the tensile test specimens.

than in PANI/TSA blends (Figure 8), which explains the differences in the conductivities of the blends. There are no distinguished differences in morphology between blends of OH- or COOH-functionalized polyethylenes.

In the blends with MAH-functionalized polyethylene the same kind of dispersion or network structure of PANI complex is not seen and the fracture surface is very smooth [Figure 9(a)]. In the blends containing metallocene polyethylene without functionalities, the surface is very rough and there is no adhesion between the two phases [Figure 9(b)].

Figure 10 depicts the fracture surface of tensile test specimen of PANI/CSA1 blend after the test. The lamellar structure of phases is clearly seen, which is formed during the tensile test. The same kind of lamellar structure is seen in blends with compatibilizers. Mechanical properties are greatly affected by adhesion of the lamellar phases.

The SEM images reveal that the PANI/CSA complex is plasticized. There are no more nonfusable particles in the PANI/CSA blends than in the other PANI complex blends.

Conclusion

Mechanical properties of polyethylene/polyaniline (PE/PANI) complex blends were improved through the addition of functionalized metallocene polyethylene as compatibilizer. The rheological properties of the components affected the morphology of the blend. With metallocene polyethylene as matrix, PANI complexes were dispersed in the blend and the percolation of the PANI complexes was prevented and the blends acted as insulators. However, in LDPE/PANI complex blends where OH- and COOH-functionalized metallocene polyethylenes acted as compatibilizers, electrical conductivity of the blends was maintained or even

increased. The conductivity of these blends was based on the hydrogen bonding between the PANI complex and functionalized metallocene polyethylene, which compensated the unfavorable rheological properties of the components. There was no clear difference between hydroxyl and carboxylic acid functionalities; both improved the mechanical properties and in some cases even the conductivity. This work shows that it is possible to prepare PANI/CSA complexes for melt processing with suitable electrical conductivity. Improvement of the mechanical properties of the blend requires that the plasticizer be better bonded to the PANI/CSA complex, but without at the same time degrading the conductivity of the complex.

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