Mechanical Properties of Compatibilized Nylon 6/Polypropylene Blends; Studies of the Interfacial Behavior Through an Emulsion Model

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ABSTRACT: The high interfacial tension between two immiscible phases in a polymer blend often prevents a homogenous stress distribution. Therefore, blends of Nylon 6 (Ny6) and polypropylene (PP) were compatibilized using two commercially available types of PP grafted with maleic anhydride (MAPP) with a low (∼2 %) and a high (∼7%) grade of maleation. The interfacial tension of compatibilized and non-compatibilized blends of PA6/PP was calculated from the recoded data of oscillatory rheological measurements using an emulsion model. Both compatibilizers showed similar improvements in tensile strength of up to 25%, but the one low maleation grade compatibilizer showed improved impact properties (>200%). It could be shown that, despite, being more effective in reducing the interfacial tension, using a high grade of maleation in the compatibilizer causes no additional improvement in tensile strength over a low grade of maleation and even has a negative effect on the PA6/PP blend impact strength. © 2014 Wiley Periodicals, Inc. J. Appl. Polym. Sci. **2014**, *131*, 40792.

INTRODUCTION

Blending of polymers offers a significant advantage over individual polymers as it allows the compensations and/or combination of individual properties that are in high demand by industry.[1] However, the combination of different polymers can prove difficult if the combined materials are thermodynamically immiscible, causing a separation of the two phases during blending. As a result the interfacial tension between the polymer phases is too high to allow an even stress distribution without disrupting the blend morphology.[2,3] Thus, to achieve a reduction of the interfacial tension and to improve phase dispersion, compatibilizers are employed that increase the interfacial adhesion.[2–5]

Blends of Nylon6 (Ny6) and polypropylene (PP) promise the benefits of a high usage temperature and stiffness combined with good impact properties, as well an improved moisture resistance and dimensional stability.[6,7] While Ny6 offers high mechanical properties and thermal stability, PP shows easier processability, improved moisture resistance and is commercially more attractive.[8] However, as Ny6 and PP are inherently immiscible, a compatibilizer is required to achieve those synergistical properties.[9]

The compatibilizer improves the interfacial adhesion by lowering the interfacial tension, reducing the particle size of the dispersed phase and as a result leads to improved mechanical properties compared to the non-compatibilized binary blends.[10–13] PP grafted with maleic anhydride (MAPP) has been shown to be an effective compatibilizer in PP-based binary blends.[14–17] Several types of MAPP are commercially available and often differ in their maleation content and molecular weight which can be expected to significantly influence the effectiveness of the applied compatibilizer. However, the improvement in interfacial adhesion between the phases in a blend is often validated exclusively indirectly by a change in mechanical properties and/or a microscopial analysis of the blend surface fracture.

Rheological, oscillatory measurements on polymer blend melts have been successfully applied to determine morphological and interfacial properties of non-compatibilized and compatibilized polymer blends based on emulsion models.[18–23]

Therefore, in the present work two types of a MAPP-based compatibilizer with different degrees of maleation will be analyzed regarding their influence on the mechanical and morphological properties of Ny6/PP blends. Additionally, the effectiveness of the used compatibilizer will be analyzed by calculating the interfacial tension of the blends and put in relation with the mechanical properties. While differences in molecular weight are an additional factor that could have an influence on the effectiveness of a compatibilzer, this work will focus exclusively on the grade of maleation.

THEORY

An emulsion of Newtonian liquids shows the rheological behavior of a viscoelastic liquid.[19] A model developed by Oldroy for dilute emulsions allows the prediction of the viscoelastic behavior over a broad frequency range.[24] Oldroy's model was developed further by Palierne for the application on concentrated emulsions.[25] Based on Oldroy's and Palierne's observations, Graebling et al. developed an emulsion model for two viscoelastic fluids, making it particularly interesting for binary polymer blends.[26]

Based on Oldroy's calculation for inclusions of monodisperse size and spherical shape, the complex viscosity $\bar{\eta}^*$ can be expressed as,

$$\bar{\eta}* = \eta_M^* \left(\frac{1+3\Phi H}{1-2\Phi H}\right) \qquad (1)$$

with Φ being the fraction of the included phase, η_M^* being the complex viscosity of the matrix phase and

$$H = \frac{4\frac{a}{R}\left(2\eta_M^*+5\eta_I^*\right)+i\omega\left(\eta_I^*-\eta_M^*\right)\left(16\eta_M^*+19\eta_I^*\right)}{40\frac{a}{R}\left(\eta_M^*+\eta_I^*\right)+i\omega\left(2\eta_I^*-3\eta_M^*\right)\left(16\eta_M^*+19\eta_I^*\right)} \qquad (2)$$

with α being the interfacial tension, η_I^* the complex viscosity of the dispersed phase, ω the frequency (that all complex viscosities are measured at) and R the radius of the inclusions.

As complex viscosity and complex modulus G^* are related by,

$$G^* = i\omega\eta^* \qquad (3)$$

Equation (1) can be rewritten as,

$$\bar{G}^* = G_M^*\left(\frac{1+3\Phi H}{1-2\Phi H}\right) \qquad (4)$$

With,

$$H = \frac{4\frac{a}{R}\left(2G_M^*+5G_I^*\right)+\left(G_I^*-G_M^*\right)\left(16G_M^*+19G_I^*\right)}{40\frac{a}{R}\left(G_M^*+G_I^*\right)+\left(2G_I^*-3G_M^*\right)\left(16G_M^*+19G_I^*\right)} \qquad (5)$$

As the factors G_M^*, G_I^*, and R can be determined experimentally, the interfacial tension α can be calculated using a non-linear regressional statistical analysis for any given Φ.

EXPERIMENTAL

Ny6 (Ultramid B24 N03), selected for its high melt flow index of approximately 47 g/10 min, measured according to ASTM standard D-1238 was supplied by BASF (Ludwigshafen, Germany). A homopolymer form of PP (PP1350N) was supplied by Pinnacle Polymers, (Garyville, LA). Both polymers were supplied in pellet form. Two different types of MAPP were used, Epolene E43, supplied by Westlake Chemical (Houston, TX) and Fusabond P353 supplied by DuPont (Wilmington, DE). Both compatibilizers were supplied in pellet form. The molecular weight of the used compatibilizers could not be measured experimentally. The molecular weight of Epolene E43 was given as 9100 by the supplier. Typical values for the molecular weight of Fusabond P353 are not available from the supplier. All polymers were dried for 72 h at 80°C before processing.

Determination of Maleic Anhydride in MAPP

To determine the level of maleation in the used compatibilizers a titration was conducted based on ASTM D 1386.

Approximately 1 g of MAPP was dissolved in 50 mL of boiling toluene and two drops of concentrated Hydrochloric acid. The solution was then titrated with 0.1N alcoholic Potassium hydroxide (KOH) using phenolphthalein as indicator. As the molar mass of maleic anhydride is 98.06 g/mol, the relative amount of maleic anhydride (MA) grafting can then be calculated by,

$$\%_{\text{MA grafting}} = \frac{N_{\text{KOH}} V_{\text{KOH}}\, 98.06}{2 W_{\text{sample}}} \times 100 \qquad (6)$$

With N being the normality in moles/liters, V is the volume in liters, and W being the weight of the sample in grams.

Each sample was tested in duplicate and average values were reported.

Sample Processing

The blend samples were prepared using micro-compounding equipment. The polymers were mixed using a HAAKE MiniLab II Micro Compounder (Thermo Scientific, Waltham, MA) at 100 rpm for 2 min at 240°C. The melt was then transferred to a HAAKE MiniJet mini injection moulder (Thermo Scientific, Waltham, MA) and injection moulded into a room temperature mold using an injection pressure of 220 bar and injection time of 10 s.

Three formulations of the non-compatibilized Ny6/PP blend in the ratios Ny6/PP of 90/10, 80/20, and 70/30 wt %, and will be referred to as NCB-1, NCB-2, and NCB-3. For the compatibilized blends 5 wt % of the PP phase were substituted with MAPP in each formulation, resulting in three formulations for each compatibilizer with the ratios of Ny6/PP/MAPP of 90/5/5, 80/15/5, and 70/25/5. The formulations employing Epolene E43 with be referred to as CBE-1, CBE-2, and CBE-3. Correspondingly, the blends containing Fusabond P353 will be referred to as CBP-1, CBP-2, and CBP-3. The relatively high amount of compatibilizer was chosen to emphasize its effect on the interfacial tension and resulting mechanical properties.

Mechanical Testing

All mechanically tested samples were dried at 80°C for 48 h before testing. Five samples of each formulation were tested to determine the tensile properties and 6 for the impact strength.

Tensile tests were performed using an Instron 3382 universal testing machine (Instron Norwood, MA) according to ASTM standard 638. Samples type V samples were tested using a crosshead speed of 10 mm/min. and a 100 kN load-cell. Manually controlled wedge action grips were used.

Izod impact testing was performed on notched samples using a TMI impact tester (Testing Machines Inc., New Castle, DE) supplied with a 0–5 × 0.05 ft.lbs hammer.

Microscopy

To determine the average radius of the inclusions samples of the blends were compression moulded into thin films of a thickness <0.2 mm at 240°C. The compression moulded samples were then etched in Xylene at 110°C for 180 min to remove
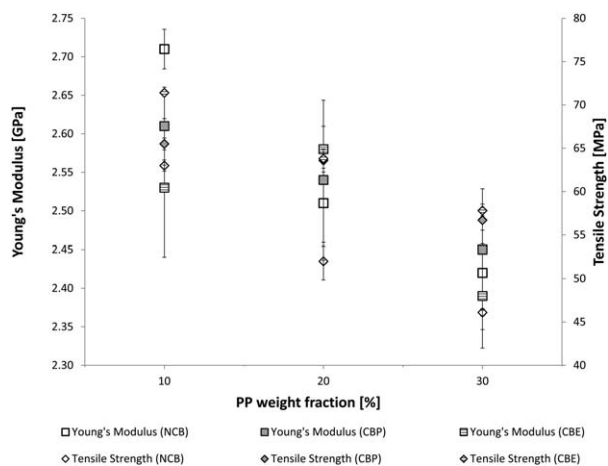
Figure 1. Young's modulus and tensile strength of the three blend formulations with and without compatibilizer. Shown are mean values with one standard deviation.

Table I. Young's Modulus and Tensile Strength of the Three Blend Formulations

Blend	Young's modulus [GPa]	Tensile Strength [MPa]
NCB-1	2.71 ± 0.025	63.00 ± 0.64
NCB-2	2.51 ± 0.055	52.00 ± 2.15
NCB-3	2.42 ± 0.073	46.10 ± 0.42
CBE-1	2.53 ± 0.050	71.40 ± 0.27
CBE-2	2.58 ± 0.103	63.80 ± 1.11
CBE-3	2.39 ± 0.078	57.80 ± 0.75
CBP-1	2.61 ± 0.089	65.50 ± 0.68
CBP-2	2.54 ± 0.030	63.60 ± 0.42
CBP-3	2.45 ± 0.068	56.7 ± 1.15

Shown are mean values with standard deviation.

the PP and PP/MAPP phase, respectively. The etched samples were rinsed in water and dried at 85°C for 24 to 48 h.

Eight to 10 images of the etched samples were then recorded for each blend using a FEI Inspect S50 (FEI Hillsboro, OR) scanning electron microscope (SEM) with an acceleration voltage of 20 kV. The size of the cavity created by the removal of the PP/MAPP phase was measured using ImageJ 1.47v (National Institutes of Health) to calculate the average radius of the inclusion necessary for the modelling.

The fracture surface of impacted tested samples was analyzed using the SEM with an accelerating voltage of 20 kV. All samples used for SEM were sputter coated for 60 sec using a Cressington Sputter Coater 108 auto (Cressington Scientific Instruments, Watford, UK) and a gold target.

Samples for Atomic Force Microcospy (AFM) were prepared from pieces cut from processed but untested impact test bars. Samples were cut using a Leica EM UC7 Ultramicrotome (Leica Microsystems, Wetzlar, Germany) with a Diatome 3 mm diamond knife (Diatome AG, Biel, Switzerland).

Tapping mode scans were performed on a Bruker Multimode 8 Atomic Force Microscope (Bruker, Billerica, MA) using a scanning TESPV2 tip.

Rheological Measurements
All rheological melt measurements were performed on an Anton Paar MCR302 rheometer (Anton Paar GmbH, Graz, Austria) with a parallel plate set-up with a plate diameter of 25 mm. The used measurement distance was 1 mm. All measurements were performed at 240°C. The linear viscoelastic range of all formulations was determined experimentally and, accordingly, a strain value of 1% was selected for the oscillatory measurements.

Frequency sweeps were performed on PP samples within a frequency range of 100 to 0.1 rad/s. However, it is known that nylon melts show an increase in complex viscosity over time.[27,28] To avoid an influence of this behavior on the conducted measurements, time sweeps were performed for 10 min with a constant strain of 1% at frequencies of 100, 39.8, 10, 5, 1, 0.251, and

0.1 rad/s on samples of the Ny6 and the Ny6/PP(MAPP) blends. The data for each was then extrapolated using a Careau-Yasuda model[28] to a theoretical time $T = 0$, to obtain a value for complex viscosity and complex modulus of the tested formulation at unaltered state. The calculated values can then be used to imitate a frequency sweep, necessary to calculate the interfacial tension using eqs. (4) and (5).

RESULTS AND DISCUSSION

Maleation Grade
As the molecular weight of the compatibilizers could not be determined and a literature survey proved to be inconclusive the analysis will focus exclusively on the effect of the maleation grade.

Based on our findings, the percentage of maleation in Epolene E43 was $6.71 \pm 0.33\%$ while Fusabond P353 only showed $1.95 \pm 0.23\%$ of grafting. The maleic anhydride content of Epolene E43 is given as <0.7% in the company's materials safety data sheet.[29] A number of published literature is stating the maleation content of Epolene E43 in the range from 2.9 wt %,[30] over 4.1 wt %[31] to as high as 8 wt %.[32] The exact
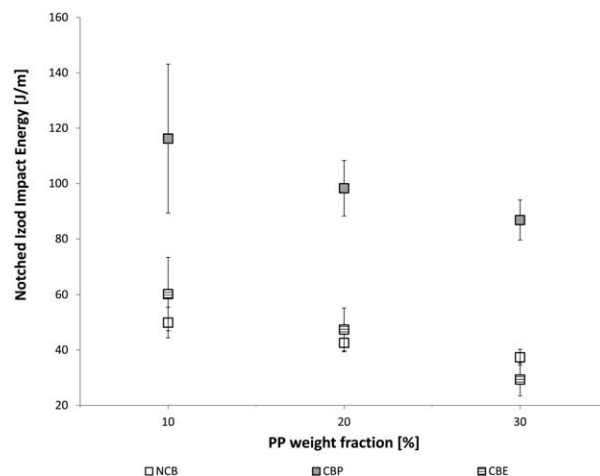
Figure 2. Notched Izod impact energy of the three blend formulations with and without compatibilizer. Shown are mean values with one standard deviation.
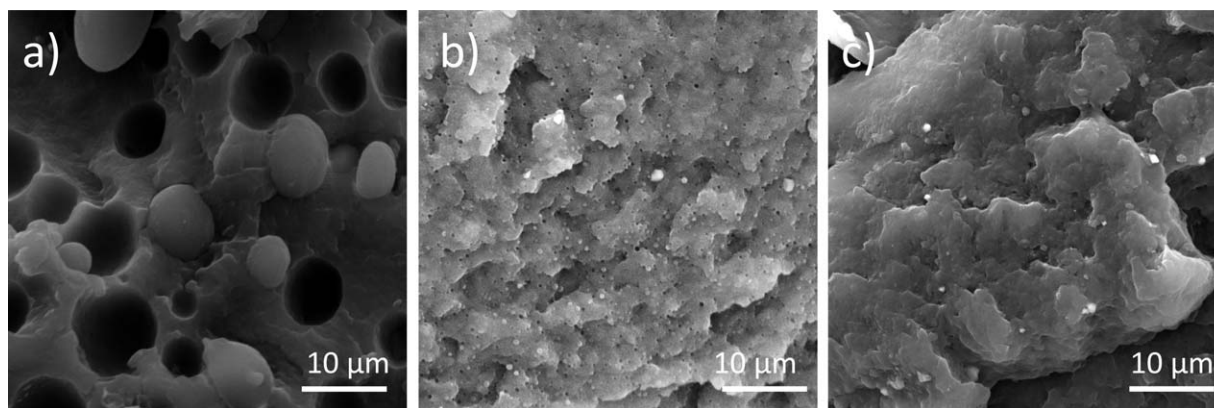
Figure 3. SEM micrographs of the fracture surface of an impact sample of NCB-1 (a), CBE-1 (b) and CBP-1 (c).

percentage of maleic anhydride in Fusabond P353 is not published by the supplier, but was given as 1.4 % by Tessier et al.[33]

Mechanical Properties

The effect of the compatibilizer on the tensile properties of the blends can be clearly seen by a strong increase in tensile strength of all compatibilized blends compared to their non-compatibilized counterparts (Figure 1 and Table I).

There is little difference in tensile strength between the formulations using 80 and 70 wt % Ny6, respectively compatibilized with Fusabond P353 (63.6 MPa and 56.7 MPa) and Epolene E43 (63.8 MPa and 57.8 MPa). However, a clear increase in tensile strength in combination with an almost constant Young's modulus compared to the non-compatibilized blends (53 MPa and 46.1 MPa) shows that a decreased interfacial tension due to compatibilization results in strongly improved tensile properties.[14] Based on the tensile test results it appears as if both compatibilizers works almost equally well in improving the tensile properties.

However, the influence of the MAPP on the notched Izod impact properties is less clear. While the application of Fusabond causes a strong increase in impact energy by more than 50 J/m in each formulation, Epolene shows only a small improvement compared to the non-compatibilized blend an in case of the 70 wt % Ny6 formulation even a small decrease (Figure 2).

While this would appear somewhat counter-intuitive given the positive effect of this compatibilizer on the tensile properties, it could possibly be explained the interfacial adhesion being too strong and therefore resulting in decreased impact properties[34] as for example reported for maleic anhydride compatibilized blends of polypropylene and EPDM (a copolymer of ethylene–propylene–diene monomer)[35] or compatibilized blends of Ny6 and very low density polyethylene.[36]

In fact, both compatibilized blends shows a strongly improved fracture mechanism compared to the non-compatibilized blend exemplarily shown for the NCB-1, CBE-1, and CBP-1 blends (Figure 3), showing that a weak interface cannot be the reason for the low impact properties of the blends compatibilized with Epolene E43.[12]

A better understanding of the effect of the used compatibilizers on the interfacial properties that could help explain the results of the mechanical testing can be achieved by calculating the
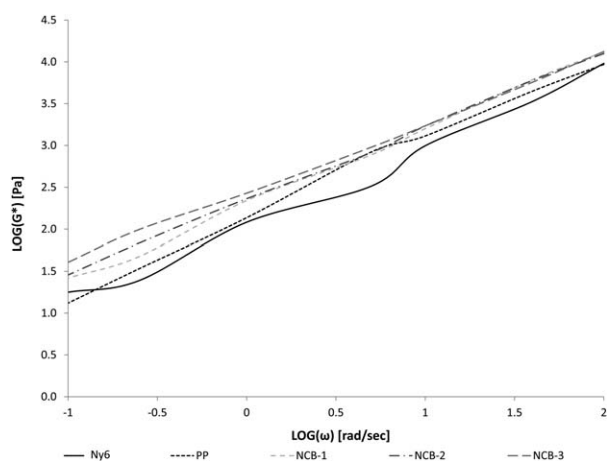
Figure 4. Complex moduli over angular frequency of neat Ny6 and PP compared to the non-compatibilized blends.
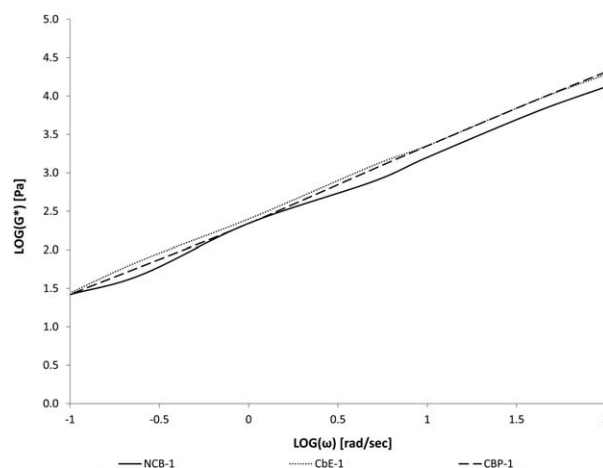
Figure 5. Complex moduli over angular frequency of the blends NCB-1, CBE-1 and CBP-1.
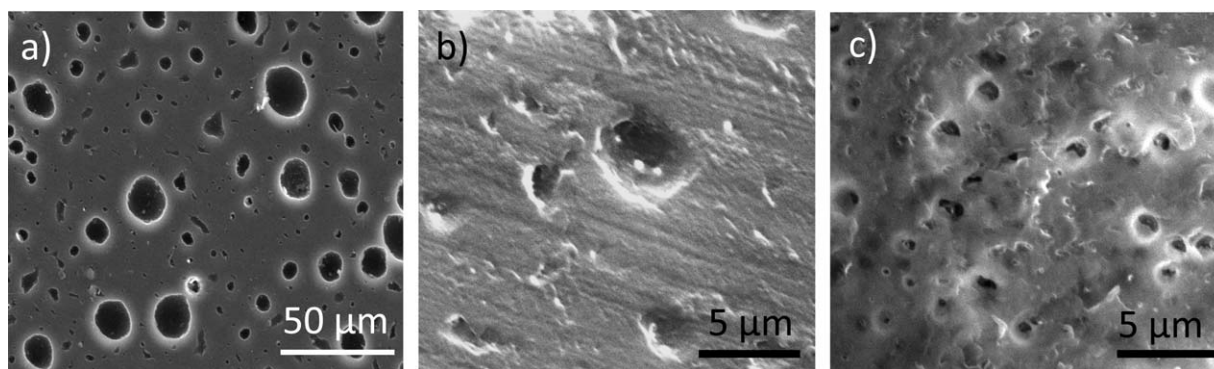
Figure 6. SEM micrographs of etched samples of NCB-1 (a), CBP-1 (b) and CBE-1 (c).

interfacial tension using the above described emulsion model and analyzing the blend morphology.
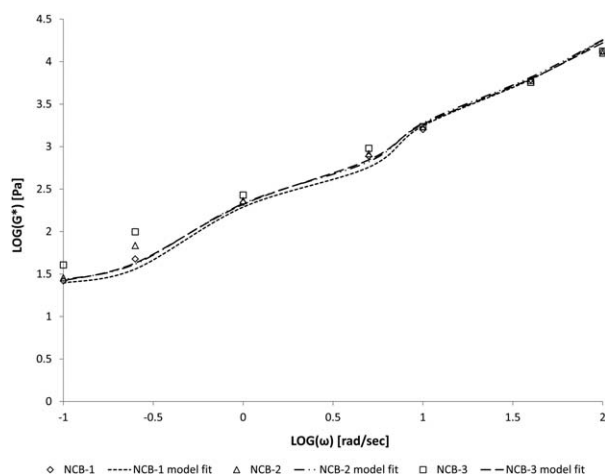
Rheology

The frequency sweeps of the pure Ny6, PP, and the non-compatibilized blends, shown in Figure 4, display a typical plateau region at lower frequencies,[37] starting at 10 rad/s. This is

especially pronounced for the neat Ny6. With increasing PP content in the blend, this behavior becomes less obvious.
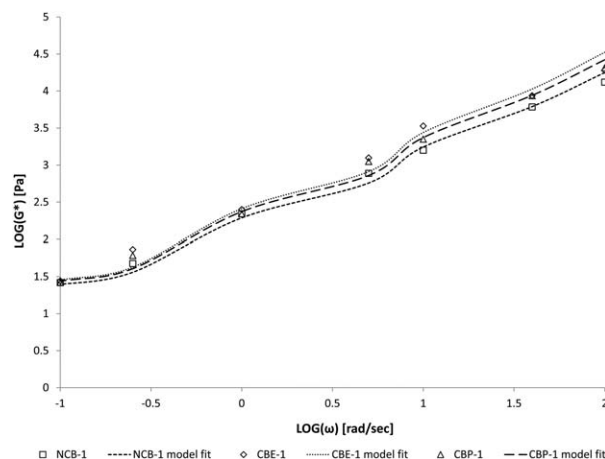
It is noteworthy that the complex modulus of the blends compared to the neat polymers is much higher at lower frequencies, and the difference is increasing with increasing PP content.
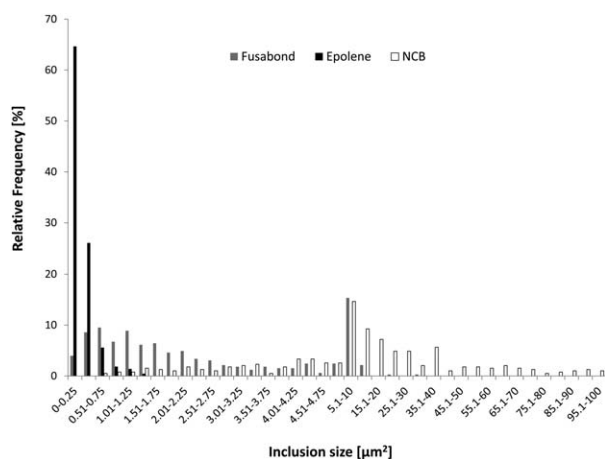

Figure 7. Size distribution of the inclusions in the blends CBP-1, CBE-1 and NCB-1.


Figure 9. Complex modulus over frequency calculated form rheological measurements of the non-compatibilized blend NCB-1 and the compatibilized blends CBE-1 and CBP-1 compared to the corresponding model fit.

Figure 8. Complex modulus over frequency calculated form rheological measurements of the non-compatibilized blends compared to the model fit.
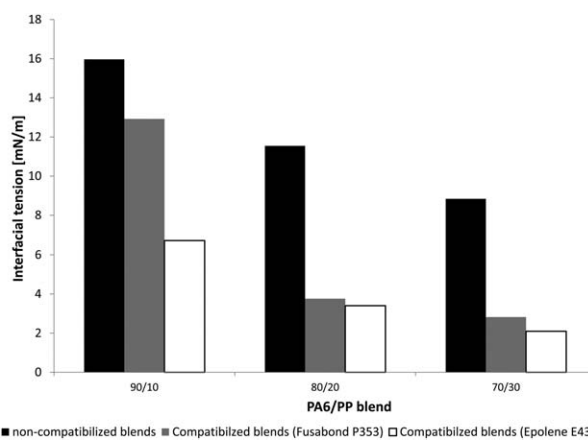
Figure 10. Calculated values of the interfacial tension between the Ny6 and PP phase in the non-compatibilized and compatibilized blends.
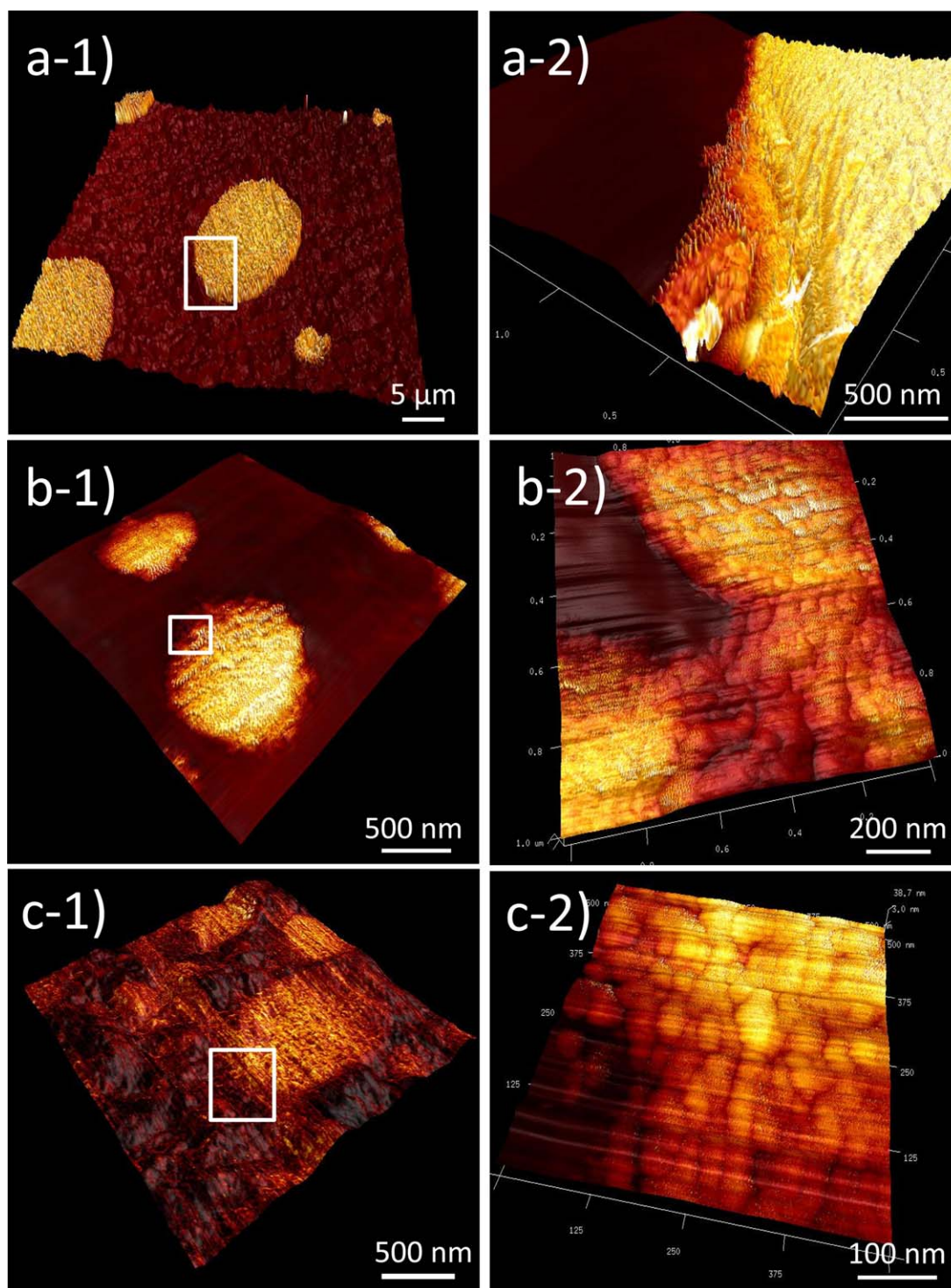
Figure 11. AFM tapping mode scans of samples taken from NCB-3 (a-1 and a-2), CBE-3(b-1 and b-2) and CBP-3(c-1 and c-2) showing the phase difference between Ny6 and PP. A clear distinction between the Ny6 phase and spherical PP phase can be seen. Both compatibilized blends show a more gradient transition between both phases than NCB-3, as shown in magnified scans. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

Gramespacher and Meissner explained this for polystyrene/poly(methylmethacrylate) blends, by an increasing contribution of the interfacial energy to the total stored energy of the system.[38] They hypothesized a shape change of the inclusion phase at low frequencies during oscillatory shear deformation, resulting in a change of interfacial area and hence, a change in interfacial energy. This energy contribution is however negligible at high frequencies.

The effect of the compatibilizer on the blends is exemplarily shown for the NCB-1, CBE-1, and CBP-1 in Figure 5. It can be seen that the plateau region is shifted to lower frequencies and also less distinct in the compatibilized blends, indicating a reduced interfacial tension.[39]

Blend Morphology

A clear size reduction close to a factor of 10 of the phase inclusion as a result of the applied compatibilizer can be seen in Figures 6 and 7 showing the etched samples for NCB-1 (a) compared CBP-1 (b) and CBE-1 (c) and the size distribution of the inclusions.[39]

An analysis of the measured size distribution of the inclusion showed only small difference for the blends CBE-2 and 3 and CBP-2 and 3 (data not shown). However, a big difference can be seen for the blends CBE-1 and CBP-1 as the application of Epolene E43 has caused a concentration of over 90 % of the inclusions in the region from 0-0.5 μm^2 (Figure 7).

Interfacial Properties

The model described by Greabling et al.[26] shown in eqs. (4) and (5) could be successfully applied to the non-compatibilized blends using a mean value for the inclusion radius calculated the carried-out measurements (Figure 8). The small deviation of the data from the model can be attributed to the model assumptions of a monodispersed size of the inclusion size. The need for extrapolation of the measured data as described above could be an additional factor for the slight discrepancy between observed data and the model.

The model can also be applied to the compatibilized blends as exemplarily shown for NCB-1 compared CBE-1 and CBP-1 (Figure 9).

The model then allows for a determination of the interfacial tension between the Ny6 and PP and Ny6/PP and MAPP phases. It is obvious from Figure 10 that the application of compatibilizers strongly reduces the interfacial tension. The calculated values of the interfacial tension of the non-compatibilized blends (19.7, 11.55 and 8.84 mN/m) are within range reported in literature confirming the effectiveness of the applied model.[39–41] Furthermore, Asthana and Jayaraman reported a drop in interfacial tension of 8 mN/m for a Ny6/PP(MAPP) blend using a compatibilizer with a maleation percentage of 0.3 wt % to 3 mN/m for a compatibilizer with a maleation percentage of 0.8 wt %, showing the importance of the grade of maleation on the interfacial tension.[40]

In our experiments those differences are most severe in the 90/10 blend as the amount of compatibilizer was deducted from the PP phase as a fixed percentage of the total blend, the amount of compatibilizer equals 50% in the blends CBE-1 and CBP-1. The data suggest that Epolene E43 shows increased interfacial interaction resulting in the decreased inclusion size.[42] Based on our measurements we have found the percentage of maleation in Epolene E43 to be 6.85 ± 0.48% and in Fusabond P353 to be 1.97 ± 0.25%, which corresponds well with our observations regarding the interfacial tension in the compatibilized blends. A similar behavior could also be observed in compatibilized Ny6/Polyethylene blends.[43]

As expected from the findings of the morphological analysis, the difference between the compatibilizers is most pronounced in the CBE-1 and CBP-1 blends, showing a reduction of the interfacial tension from 15.97 mN/m to 12.92 mN/m for Fusabond P353 and 6.83 mN/m for Epolene E43. However, the absolute reduction in surface tension is bigger for the blends with a higher content of PP, from 11.55 mN/m down to 3.76 and 3.4 mN/m and from 8.84 mN/m to 2.82 and 2.10 mN/m for Fusabond and Epolene, respectively (Figure 10). This can be explained by the increased ratio PP/MAPP in those formulations, indicating that the amount of compatibilizer is actually too high in the blends with the lowest PP content. Given the small differences in interfacial tension of the blends containing 80 and 70 wt % Ny6 it can be assumed that a full saturation with the compatibilizer has been reached at those formulations, and the interface is less affected by an increase in PP phase.

The improved interfacial adhesion as a result of compatibilization can also be seen in the AFM scans. Next to the already discussed noteworthy decreased in inclusion size with compatibilization[44] a high magnification scan of the Ny6/PP interface in the non-compatibilized blend [Figure 11(a-1, a-2)] shows a clear line between the two phases showing clear phase separation as a result of poor interfacial adhesion.[45] However, for both compatibilized blends [Figure 11(b,c)] a small gradient can be seen between the two distinct phases, indicating the formation of a true "interphase" between Ny6 and PP can be controlled by the introduction of MAPP.[7,12] This is the result of strongly anchored PP particles and an increased coalescence due to the presence of the compatibilizer.[42]

Those results show that judging the effectiveness of a compatibilizer merely by its effect on the mechanical properties can be misleading regarding the resulting interfacial adhesion between the used blend components. A more detailed and direct analysis of the effect of a compatibilizer on a blend system as done here can provide valuable detail that allow for a more effective selection of compatibilizer necessary to achieves the wished for mechanical properties.

CONCLUSIONS

MAPP can drastically reduce the interfacial tension of Ny6/PP blends resulting in an improved tensile strength. However, the type of compatibilizer and its grade of maleation were shown to have a strong effect on the interfacial tension and therefore the mechanical properties of the blends.

The interfacial tension of non-compatibilized and compatibilized blends of Ny6 and PP could be determined from oscillatory rheological measurements using an emulsion model and it could be shown that the compatibilizer with a higher degree of maleation (Epolene E43) shows a stronger reduction in interfacial tension. However, a too strongly reduced interfacial tension could be detrimental to the impact strength. Therefore care should be taken when assessing the effect of a compatibilizer indirectly by analysis of the mechanical properties.

ACKNOWLEDGMENTS

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