Blends of Polyamide 6 with Low-Density Polyethylene Compatibilized with Ethylene–Methacrylic Acid Based Copolymer Ionomers: Effect of Neutralizing Cations

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ABSTRACT: Blends of polyamide 6 with low-density polyethylene compatibilized with sodium-, zinc-, and lithium-neutralized ethylene—methacrylic acid ionomers were investigated at 11, 33, and 55 wt % neutralization of the ionomers. Blends of polyamide 6 with low-density polyethylene without a compatibilizer had poor properties characteristic of incompatible polymer–polymer blends. After the addition of a compatibilizer, tensile properties improved, the modulus drop associated with melting increased to higher temperatures, and the dispersed phase size decreased. The improvement of the mechanical properties and thermomechanical properties was less with the acid copolymer than with the ionomers. Overall, ionomers neutralized with sodium, zinc, or lithium showed little difference in their compatibilization efficiency. © 2007 Wiley Periodicals, Inc. J Appl Polym Sci 107: 3090–3098, 2008

Key words: blends; compatibility; ionomers; polyamides; polyethylene (PE)

INTRODUCTION

Blending polyolefins with engineering plastics offers an interesting route to achieving new materials with promising property combinations. Blends of polyolefins with polyamides, such as polyamide 6 (PA6), have been extensively studied because of their practical interest. Polyolefins are easy to process and insensitive to moisture, exhibit good flexibility, and are relatively inexpensive. Polyamides are rigid and more thermally stable and possess good barrier properties to oxygen and organic solvents. Therefore, the addition of a small amount of a polyolefin to a polyamide can improve the impact properties, whereas a polyamide dispersed in a polyolefin may enhance the oxygen resistance and hydrocarbon permeation of the polyolefin or act as a reinforcing agent.[1]

However, PA6 and polyolefins form thermodynamically immiscible blends, and hence blends show poor ultimate properties. Frequently, when an immiscible blend is subjected to stress, the stress concentrates at the polymer–polymer interface, which, for incompatible polymer pairs, is weak and unable to transfer the stress between the continuous and dispersed phases. One strategy to reduce the negative effects of immiscibility in PA6/polyolefin blends is to introduce acid groups onto the polyolefin to react with terminal primary amines[2] and to introduce the possibility of chemical interchange reactions involving the amide linkage.[3] Another strategy is to introduce a third component, a compatibilizer, to improve interfacial properties between PA6 and the polyolefin. As a result, an increase in stress transfer between the continuous and dispersed phases is produced, improving the mechanical properties of the blend.[4] Compatibilization of polyamide/polyolefin blends has been achieved through the use of various polymers, including ethylene—methacrylic acid copolymers,[5] ethylene–acrylic acid copolymers,[2] and ethylene homopolymers or copolymers with grafted maleic anhydride.[6,7] The reaction between the terminal amine group of PA6 and the acid and/or anhydride causes graft copolymer formation during extrusion, which has been theorized to significantly strengthen the interface.[5]

Copolymers of ethylene with monomers containing acid groups are important commercial products. These materials are sold commercially with either hydrogen or a metal cation as the neutralizing agent

for the acid group. The latter are termed *ionomers*, and typically the number of acid groups neutralized with a metal cation is less than stoichiometric; that is, some of the acid groups are neutralized with a metal cation, whereas others are neutralized by hydrogen. One commercial ionomer is a copolymer of ethylene and methacrylic acid marketed by DuPont under the trademark Surlyn. Three of the most common neutralizing cations are lithium (Li^+), sodium (Na^+), and zinc (Zn^{2+}). The properties of the alkali-neutralized and zinc-neutralized materials are different; for example, sodium or lithium ionomers absorb significantly more water and tend to have higher fractional crystallinities than zinc ionomers.

Zinc-neutralized and sodium-neutralized ionomers have been extensively studied in previous work as blend compatibilizers for the polyamide–polyethylene system.[8–12] The addition of a compatibilizer has been shown repeatedly to increase compatibility between the two components, including improvements in mechanical properties[13] and barrier properties[9] and smaller dispersed domain sizes.[11,13,14]

The purpose of this study is to compare the effects of sodium-, zinc-, and lithium-neutralized ethylene-*co*-methacrylic acid as a compatibilizer for PA6/low-density polyethylene (LDPE) blends and also to study the effect of the neutralization level (%) on PA6/LDPE blends. The acid copolymer is also included as a reference. This study expands significantly what has been done in previous studies because (1) different neutralization levels are compared with the same starting copolymer resins and (2) a direct comparison between three different cations is made with the same starting copolymer resin.

EXPERIMENTAL

Materials

The PA6 employed in this study was an injection-molding grade (1013B) supplied by UBE Polyamide (Bangkok, Thailand). The supplier reported a molecular weight for this material of 12,000 g/mol, and it is reported to have approximately equal numbers of amine and carboxylic acid end groups. LDPE (LD 1450J; density = 0.914 g/cm³) was also an injection-molding-grade polymer graciously supplied by Thai Polyethylene Co., Ltd. Poly(ethylene-*co*-methacrylic acid), marketed under the trademark Nucrel 0903 (density = 0.93 g/cm³), was supplied by DuPont (Bangkok, Thailand).

Neutralization of the ethylene–methacrylic acid copolymer (EMAA)

EMAA (20 g) was dissolved at 140°C in 200 mL of a solution that contained toluene and *n*-butanol (3/1).

Twenty-five milliliters of either sodium or lithium hydroxide (the concentration of the hydroxide was adjusted according to the neutralization level desired) was added, and the system was refluxed at 140°C for 3 h. The solvent was evaporated, then 150 mL of fresh solvent was added and evaporated, and then the fresh solvent addition and evaporation steps were repeated two more times. The total time of the four evaporation steps and three addition steps was approximately 1 h; that is, the polymer was in contact with the solvent for approximately 4 h. The ionomer was dried overnight at 60°C.

For the zinc-neutralized materials, the appropriate amount of zinc acetate was mixed with 400 g of EMAA by a tumble mixer for 10 min. The materials were then blended in a Collin D-8017 T-20 twin-screw extruder (Bangkok, Thailand) with a screw speed of 35 rpm, which corresponded to a residence time of approximately 1 min in the extruder.

To determine the neutralization level of the ionomers, the carboxyl contents of ethylene methacrylic acid were determined by a titration method[15] that involved titration of a hot *n*-butanol and toluene solution containing the polymer by 0.1*N* sodium hydroxide in an aqueous solution with phenolphthalein as the indicator. Pure EMAA was also titrated to determine the starting carboxyl content of the material. The amount of acid monomer on the copolymer was 9 wt % (3 mol %) as determined by titration. Neutralization levels were 11, 33, and 55 wt % (±0.5%) for the three cations, again as determined by titration.

Blend preparation

Pellets were mixed in a tumble mixer for 10 min, and this was followed by drying *in vacuo* at 60°C for 24 h. The materials were then blended in a Collin D-8017 T-20 twin-screw extruder with a screw speed of 35 rpm. Blends were extruded through a single-strand die; the extrudates were cooled in a water bath, dried at the ambient temperature, and then pelletized. The pellets were dried and kept in sealed plastic bags before compression molding to minimize moisture absorption. Ionomer-compatibilized blends were made in a two-step process; first, the ionomer and LDPE were extruded together as a 50/50 master batch, and then LDPE, PA6, and an appropriate amount of the 50/50 master-batch mix were extruded together. EMAA-compatibilized blends were made in one shot; that is, LDPE, PA6, and EMAA were extruded together. It is not thought that this change in procedure affected the results.

Specimen preparation

Test specimens were prepared with a Wabash V 50 H 50-ton compression-molding machine
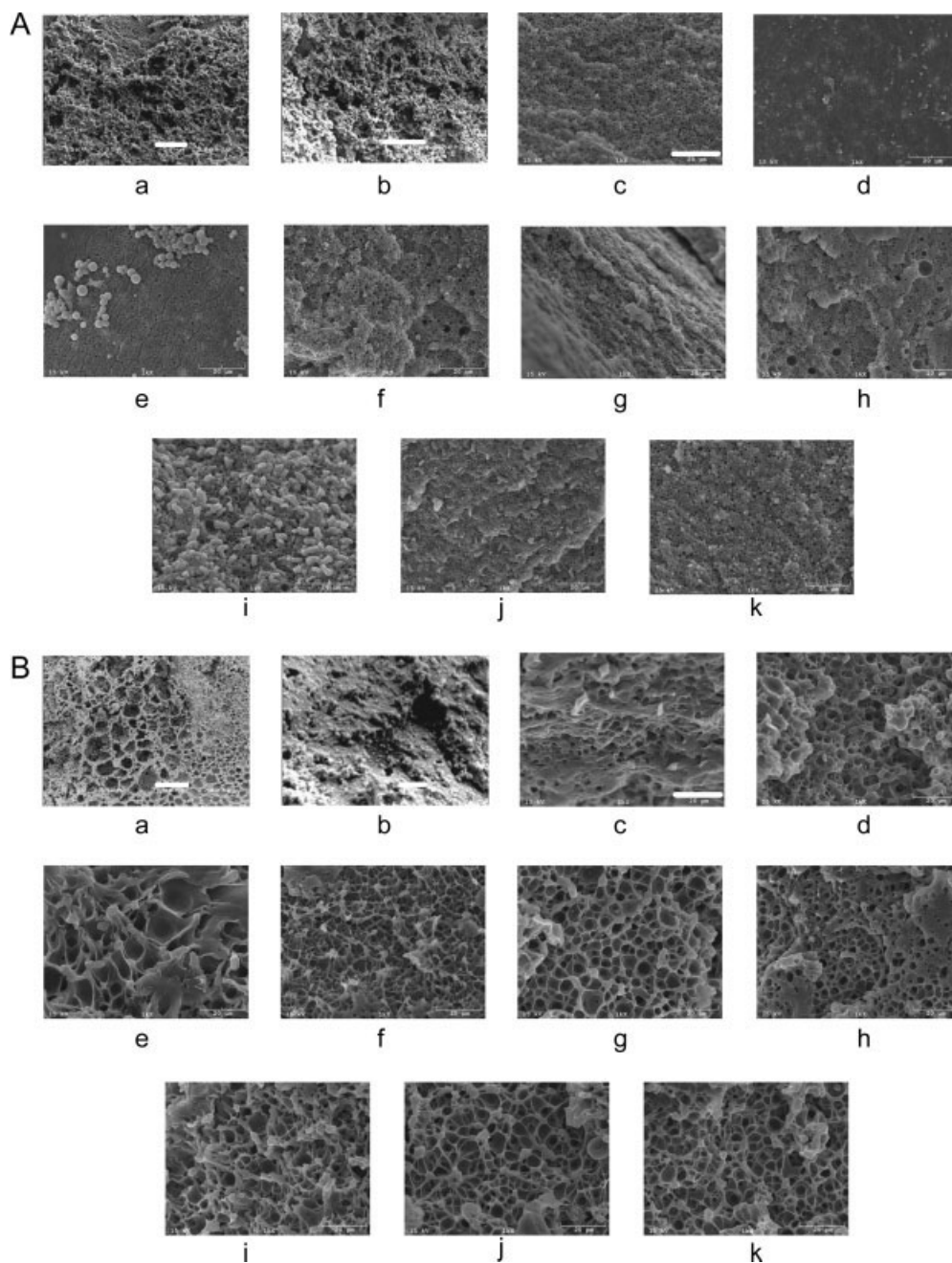
Figure 1 (A) SEM micrographs of 80/20 PA6/LDPE blends: (a) no compatibilizer, (b) EMAA (1.5 phr), (c) 11 wt % Na–EMAA (1.5 phr), (d) 33 wt % Na–EMAA (1.5 phr), (e) 55 wt % Na–EMAA (1.5 phr), (f) 11 wt % Zn–EMAA (1.5 phr), (g) 33 wt % Zn–EMAA (1.5 phr), (h) 55 wt % Zn–EMAA (1.5 phr), (i) 11 wt % Li–EMAA (1.5 phr), (j) 33 wt % Li–EMAA (1.5 phr), and (k) 55 wt % Li–EMAA (1.5 phr). Scale bars are shown in each micrograph but are difficult to discern; the white bars in panels a and b represent 50 µm, whereas the white bar in panel c represents 20 µm and is approximately the same for all the other panels. (B) SEM micrographs of 20/80 PA6/LDPE blends: (a) no compatibilizer, (b) EMAA (1.5 phr), (c) 11 wt % Na–EMAA (1.5 phr), (d) 33 wt % Na–EMAA (1.5 phr), (e) 55 wt % Na–EMAA (1.5 phr), (f) 11 wt % Zn–EMAA (1.5 phr), (g) 33 wt % Zn–EMAA (1.5 phr), (h) 55 wt % Zn–EMAA (1.5 phr), (i) 11 wt % Li–EMAA (1.5 phr), (j) 33 wt % Li–EMAA (1.5 phr), and (k) 55 wt % Li–EMAA (1.5 phr). Scale bars are shown in each micrograph but are difficult to discern; the white bar in panel a represents 50 µm, the white bar in panel b represents 10 µm, and the white bar in panel c represents 20 µm and is approximately the same for all the other panels.

(Bangkok, Thailand). Pellets were placed in a picture frame mold, and the mold was preheated at 240°C for 3 min in the press without application of pressure. The mold was then compressed under a force of 10 tons for a further 3 min, after which the mold was cooled to 40°C under pressure. Test specimens were cut from the molded sheets with a die cutter.
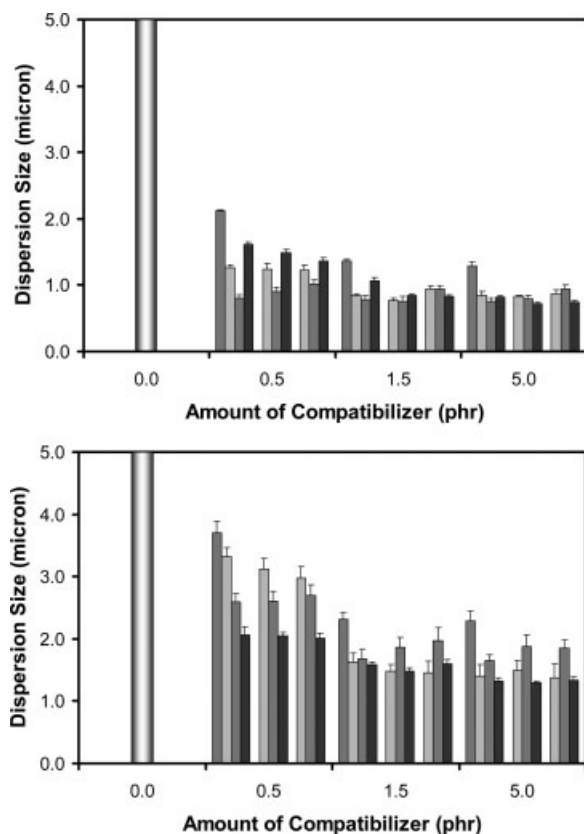
Figure 2 Dispersion size of 80/20 PA6/LDPE blends (top) and 20/80 PA6/LDPE blends (bottom). Data at zero represent no compatibilizer, but the full scale is not shown; the actual amounts are 16.0 and 18.0 μm, respectively. Light gray represents Na–EMAA, dark gray represents Zn–EMAA, and black represents Li–EMAA. For a given compatibilizer amount, groups of bars represent 11% neutralization (this group also contains pure EMAA as the leftmost bar), 33% neutralization, and 55% neutralization from left to right.

Scanning electron microscopy (SEM)

SEM [5200-2AE (MP152001), JEOL] (Bangkok, Thailand) was used to study phase morphologies of the blends. The specimens were fractured in liquid nitrogen and etched with (1) hot decalin (for high-density polyethylene minor phase blends) and (2) formic acid (for PA6 minor phase blends). The specimens were then coated with gold *in vacuo*. All SEM specimens studied were characterized with a magnification of 1000× at 15 kV.

Dynamic mechanical analysis (DMA)

An RSA II solids analyzer (Rheometric Scientific) (Piscataway, New Jersey) was used to measure the storage and loss moduli as a function of temperature. Film geometry and 4°C temperature steps were used. Samples were molded to a thickness of

around 0.5 mm. All experiments were performed with a 1-Hz frequency and 0.03% strain and with static force tracking dynamic force.

Tensile testing

A D1708 (Norwood, Massachusetts) microtensile die was used to cut the samples for tensile testing, and an Instron universal testing machine was used to measure the tensile strength with a crosshead speed of 1.30 mm/min. Samples were molded to a thickness of approximately 0.5 mm. At least five samples were used for each composition to determine an average and standard deviation.

Differential scanning calorimetry (DSC)

A TA Instruments Q1000 differential scanning calorimeter (New Castle, Delaware) with liquid nitrogen cooling was used for this study and was routinely calibrated with four different standards (cyclopentane, biphenyl, indium, and tin) at a 10°C/min heating rate. Polymer samples were cut from the same sheet used to cut samples for tensile testing, placed in aluminum DSC pans, and scanned at a rate of 10°C/min. The melting characteristics of the two components were determined during this initial scan, whereas the glass-transition temperature was determined during a second scan after the material was held at 250°C for 5 min and rapidly cooled to −100°C to ensure good sample–pan contact and eliminate complicating enthalpy relaxation effects. The fractional crystallinity was determined by integration of the area under the respective melting exotherms and conversion of these enthalpies to fractional crystallinities with a melting enthalpy of 282 J/g for polyethylene and 190 J/g for polyamide.[16] Crystallinities were calculated with eq. (1):

$$\chi_c = \frac{\Delta H \times 100\%}{\Delta H_f \times \text{Weight fraction}} \tag{1}$$

where χ_c is the crystallinity fraction (wt %), ΔH is the melting enthalpy of the components present in the blend, and ΔH_f is the heat of fusion for 100% crystallinity of the pure component (190 J/g for PA6 and 282 J/g for LDPE).

Dynamic stress rheometer

An SR 5000 dynamic stress rheometer (Rheometric Scientific) was used to measure the steady shear viscosity as a function of the shear rate. Steady-state conditions were known to occur because the viscosity was measured as a function of time and only when the viscosity was constant were measure-
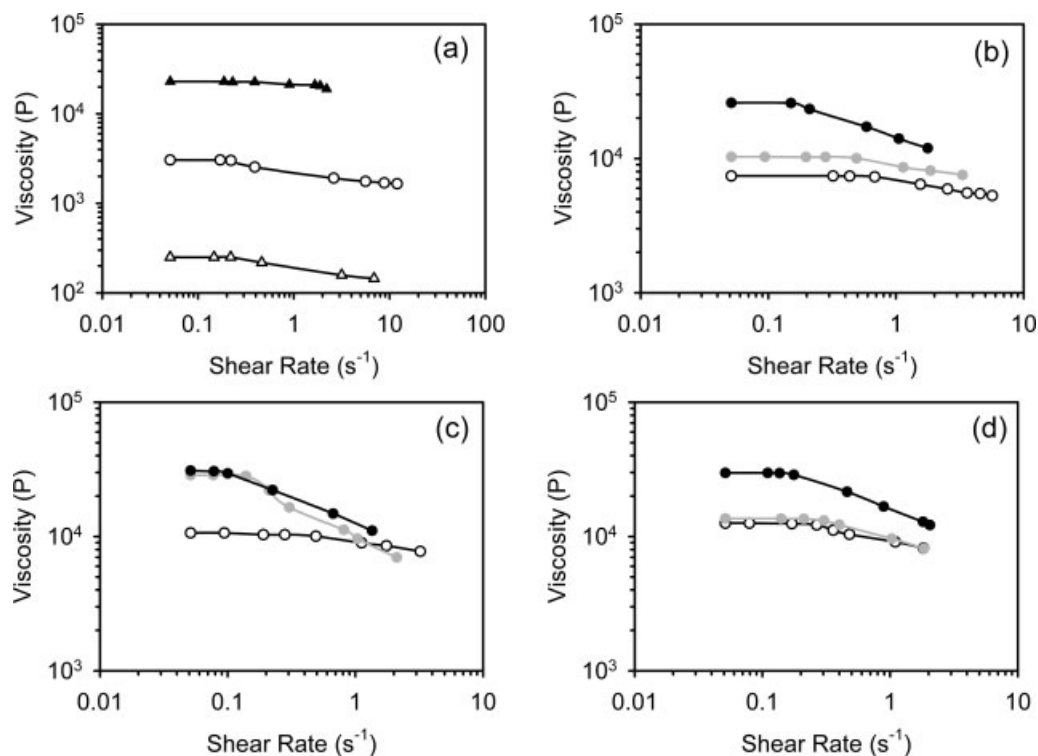
Figure 3 Steady-state shear viscosity versus the shear rate: (a) LDPE at 230°C (open triangles), PA6 at 230°C (open circles), and EMAA at 140°C (filled hexagons); (b) 50 wt % master batch of Na–EMAA; (c) 50 wt % master batch of Zn–EMAA; and (d) 50 wt % master batch of Li–EMAA. White represents 11 wt % neutralization, gray represents 33 wt % neutralization, and black represents 55 wt % neutralization. All master batches were measured at a temperature of 140°C.

ments recorded. Cone and plate geometry (cone angle = 0.0393 rad) was used with this experiment. Samples were molded to a thickness of around 0.5 mm and a plate diameter of 40 mm and then melted in the instrument, and the two plates were brought to the proper height as determined by the manufacturer. The condition for PA6 and LDPE was 230°C, whereas the pure EMAA material and ionomer master batches (50/50 LDPE/ionomer) were measured at 140°C because the materials were unstable at 230°C for the long periods of time required to measure the viscosity. Master batches of ionomers were used rather than pure ionomers because all of the ionomer was converted into master batches.

RESULTS AND DISCUSSION

Figure 1(A,B) shows micrographs of the 80/20 and 20/80 PA6/LDPE blends and indicates predominantly spherical droplets imbedded in a matrix. As expected, adhesion between the PA6 phase and the LDPE phase is poor in the uncompatibilized blends [panel a in Fig. 1(A,B)], as confirmed both by the large size of the phases and by the smoothness of hole surfaces. After the introduction of a compati-

bilizer, the size of the dispersed phase becomes much smaller. This reduction in size suggests that drop coalescence in the extruder is being reduced. Figure 2 shows the effect of the compatibilizer concentration and neutralization level on the size of the dispersed phase. As shown in Figure 2, only 1.5 phr Na–EMAA, Zn–EMAA, or Li–EMAA is sufficient to produce the maximum reduction in dispersed phase size. In all cases, the reduction is significantly better for the ionomer than for the acid copolymer.

For 80/20 PA6/LDPE blends with ionomers at 0.5 phr, the dispersed phase size tends to decrease with increasing neutralization level for Li–EMAA. With Zn–EMAA, the dispersed phase size tends to increase with increasing neutralizing level. For Na–EMAA, the dispersed phase size is independent of the neutralizing level. At higher neutralization levels, the dispersed phase size does not seem to depend on the neutralization level. In terms of cations, the efficiency goes as Zn > Na > Li as measured by the dispersed phase for the 0.5 phr sample; at higher compatibilizer levels, there seems to be no difference. For the high LDPE content materials, Li > Zn > Na for the 0.5 phr sample; for higher levels, Li = Na > Zn. Our hope was that one cation would clearly be better than another in promoting compa-
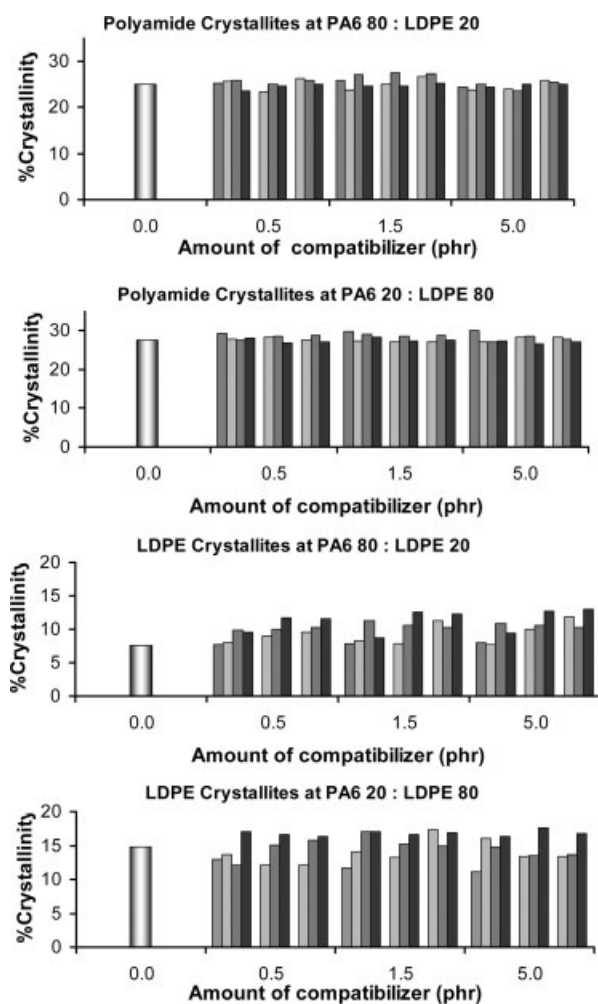
Figure 4 Percent crystallinity of PA6 and LDPE in blend ratios of 80/20 and 20/80 from DSC. Data at zero represent no compatibilizer. Light gray represents Na–EMAA, dark gray represents Zn–EMAA, and black represents Li–EMAA. For a given compatibilizer amount, groups of bars represent 11% neutralization (this group also contains pure EMAA as the leftmost bar), 33% neutralization, and 55% neutralization from left to right.

tibilization; however, the results clearly are inconsistent with respect to the dispersed phase size at least. However, it is well known that the viscosity of ionomers depends on the neutralizing cation, and perhaps the relative viscosities of the three components might help explain the results.

Figure 3 shows steady shear viscosities for the homopolymers (PA6, LDPE, and EMAA) and master batches. Neutralization of the acid has been shown to increase the viscosity of ionomers, and the viscosity continues to increase as the neutralization level increases.[17,18] This behavior is shown in Figure 3(b–d), although the increase is not uniform at all shear rates; for example, at shear rates greater than 1 s^{-1}, the 11% zinc-neutralized sample has higher viscosity than the 33% sample. Looking only

at zero-shear viscosities, we find that the increase with neutralization is very nonlinear (even in a log sense) for a given cation. Although the range of cation levels studied was very different and pure ionomers were used previously, such nonlinearity is not unique.[15] However, what is unique is that the zero-shear viscosities is only slightly larger for Zn versus Na; a factor of 4 was seen in an earlier study that used pure ionomers.[15] The fact that the viscosity is relatively inconsistent with respect to the neutralizing cation supports the inconsistency noticed in Figure 2. A more quantitative assessment is not possible because the relevant shear rate at which such a comparison should take place is not clear.

The addition of a compatibilizer had no effect on the melting temperature as measured by DSC for any samples; the melting temperature of the polyethylene was 107.1 ± 0.7°C, whereas that for PA6 was 221.3 ± 1.1°C. Figure 4 shows the percent crystallinity for each blend. The addition of a compatibilizer had no effect on the percent crystallinity of PA6, except possibly that the addition of the EMAA compatibilizer may have slightly increased the percent crystallinity of the PA6 component in the low PA6 content blends. In the low LDPE content material, LDPE crystallinity increases with the addition of a metal-neutralized compatibilizer at low compatibilizer amounts versus the EMAA compatibilizer, with Li–EMAA producing the highest increase. This effect is difficult to rationalize; compatibilization should reduce chain mobility, and this in turn should reduce crystallization. Perhaps the metal cation is nucleating crystallinity, which in turn outweighs the small reduction in chain mobility that occurs from 0.5 to 1.5 phr. Consistent with previous work that has shown that crystallinity decreases with an increase in an ionomeric compatibilizer,[10,11] the crystallinity decreases at 5.0 phr versus 1.5 phr when we consider both the compatibilizer and LDPE because the denominator of the weight fraction used in the fractional crystallinity calculation considers LDPE only. In the high LDPE content material, LDPE crystallinity is retarded by EMAA, and Li–EMAA increases LDPE crystallinity consistently. There is no consistent crystallinity increase or decrease with respect to the other two cations across compatibilizer amounts/neutralization levels.

The mechanical properties of the blends were tested, and the tensile strength, elongation at break, and modulus are shown in Figure 5. For the low LDPE content materials, ionomers significantly outperform EMAA as a compatibilizer with respect to failure stress and failure strain, whereas the difference among the zinc-, sodium-, and lithium-neutralized materials is not outside the range of experimental
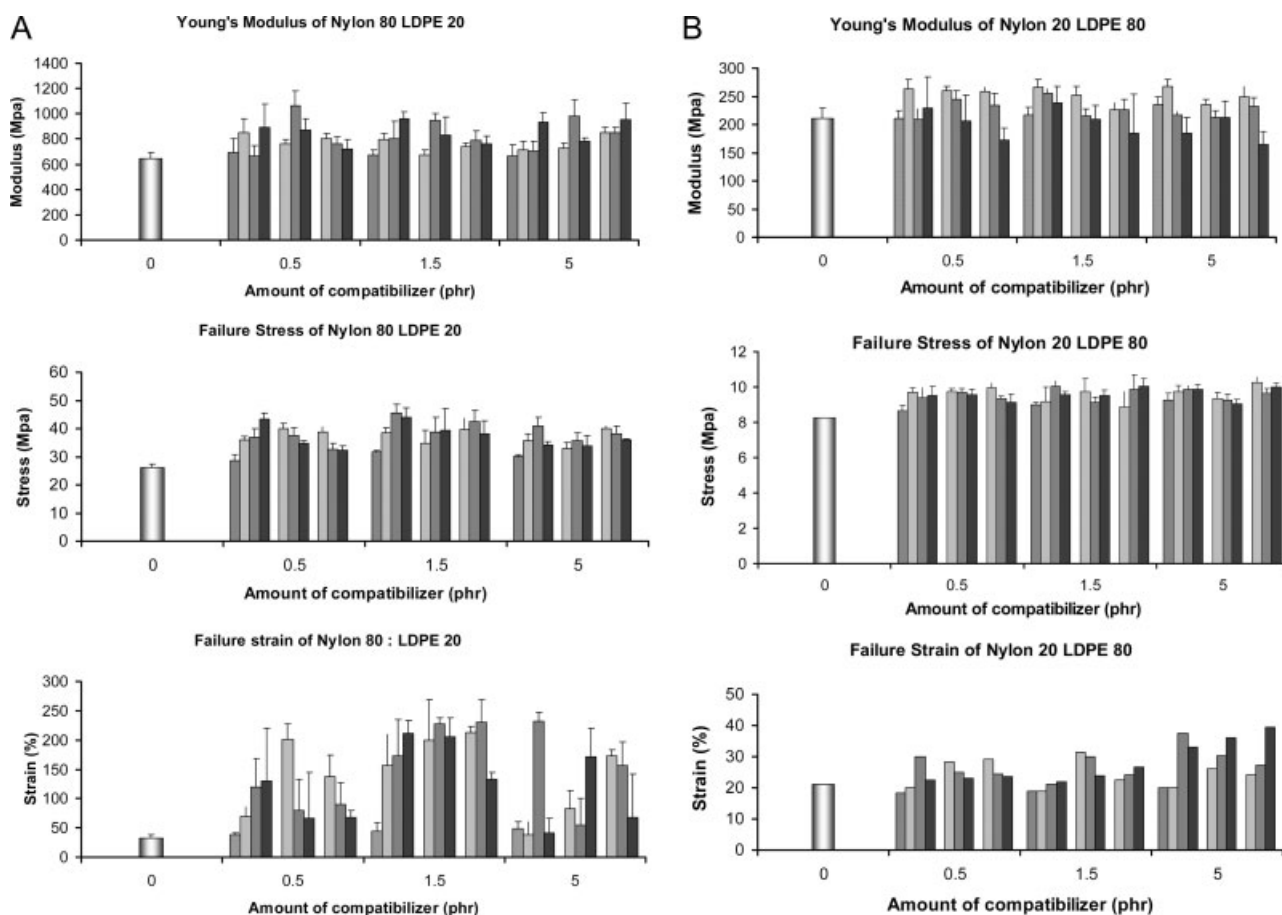
Figure 5 Tensile results (Young's modulus, failure stress, and failure strain) for (A) 80/20 PA6/LDPE blends and (B) 20/80 PA6/LDPE blends. Data at zero represent no compatibilizer. Light gray represents Na–EMAA, dark gray represents Zn–EMAA, and black represents Li–EMAA. For a given compatibilizer amount, groups of bars represent 11% neutralization (this group also contains pure EMAA as the leftmost bar), 33% neutralization, and 55% neutralization from left to right.

error, except for the failure strain at 5% compatibilizer content. The behavior at this high content seems to be quite random, suggesting perhaps that the nature of the LDPE phase is changing (because the ionomer should be 25% of the dispersed phase). In agreement with SEM data, 1.5% compatibilizer content seems to be sufficient for optimal performance. Surprisingly, the modulus for the materials with a metal-neutralized compatibilizer seems to be higher than that of the uncompatibilized or EMAA-compatibilized blends, even though PA6 crystallinity (which should be the most important factor with respect to the modulus in these high PA6 content materials) is independent of these factors.

The benefits of adding a compatibilizer are extremely small, if present at all, for the high LDPE content blends. There might be a small increase in failure stress and failure strain with the addition of a metal-neutralized compatibilizer, but the increases are marginal. No consistent effects are found in the modulus, which does not match the higher LDPE

crystallinity for the material compatibilized with Li–EMAA.

DMA spectra for ternary blends are shown in Figure 6. The addition of EMAA, Na–EMAA, Zn–EMAA, or Li–EMAA leads to samples that have mechanical stability at much higher temperatures for both blend compositions; that is, the storage modulus drop-off corresponding to the melting transition is shifted to a higher temperature as compatibilizer is added. As a measure of the melting temperature as probed by DMA, Figure 7 shows the temperature at which the storage modulus dropped to 10^7 dyn/cm^2. The melting temperature of 80/20 PA6/LDPE is lower when EMAA or no compatibilizer is used versus Na–EMAA, Zn–EMAA, or Li–EMAA as compatibilizer. No consistent melting behavior difference was found for the three neutralization levels or for the three different cations.

There is essentially universal agreement in the literature that the mechanism of compatibilization is a reaction involving carboxylic acids. Yet, as this work
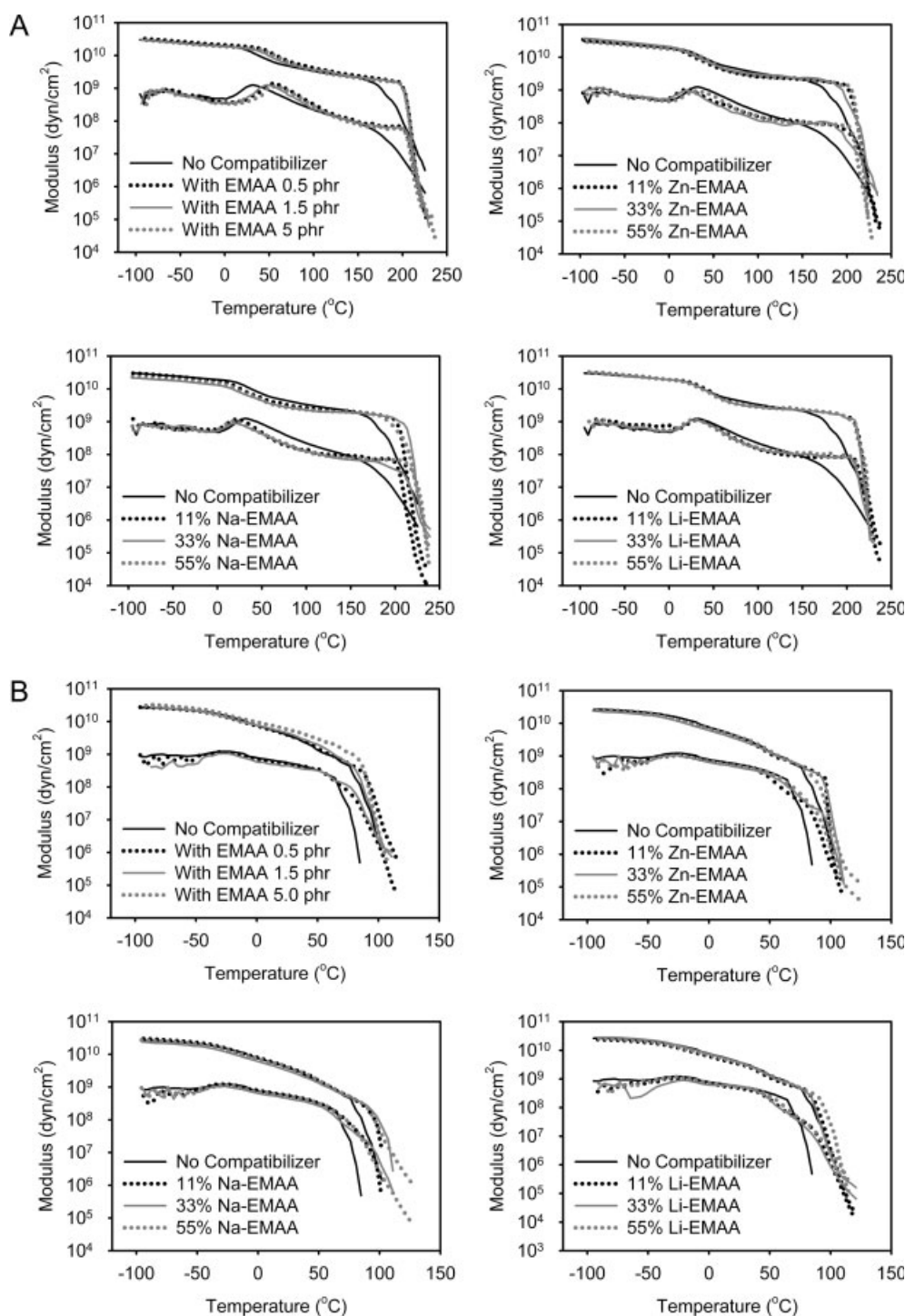
Figure 6 DMA results for (A) 80/20 PA6/LDPE and (B) 20/80 PA6/LDPE.

shows (and other work has before[19–21]), it seems quite unusual that decreasing the concentration of one of the reactants (i.e., the carboxylic acid) increases compatibilization performance. Two possibilities could explain this observation. The first is that the substantially higher viscosity of the ionomer causes improved compatibilization. However, if increased compatibilizer viscosity were the only

reason, then the performance at 55% neutralization should have been better than the performance at 11% neutralization, and this was not found. The other possibility is that the phase separation that the metal cation induces, or the fact that the metal cation itself is present in the sample (e.g., a catalytic effect), somehow enhances the number of covalent bonds at the PA6/LDPE interface. However, in either of these
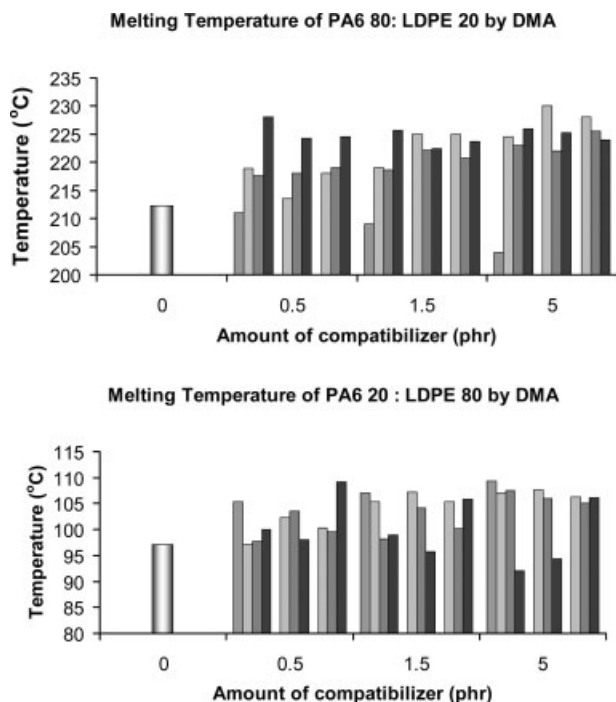
Figure 7 Melting temperature (determined as the temperature at which the storage modulus was 10^7 dyn/cm^2) of PA6 and LDPE in blend ratios of 80/20 and 20/80 from DMA. Data at zero represent no compatibilizer. Light gray represents Na–EMAA, dark gray represents Zn–EMAA, and black represents Li–EMAA. For a given compatibilizer amount, groups of bars represent 11% neutralization (this group also contains pure EMAA as the leftmost bar), 33% neutralization, and 55% neutralization from left to right.

cases, one would expect to see significant differences between either the cation types or amounts, and such a difference was not seen.

CONCLUSIONS

Adding an ionomeric compatibilizer improves blend properties in PA6/LDPE blends. Comparing the efficiency among sodium, zinc, and lithium shows some differences in the efficiency as measured by mechanical properties, dispersed phase size, and thermome-chanical properties; however, the differences are not consistent with respect to the neutralization level or compatibilizer amount. It is possible that this inconsistency might be due to the inconsistency in viscosity; although direct comparison is not possible because of different shear rate dependences of viscosity. As compatibilizers for PA6/LDPE blends, the data clearly show, however, that metal-cation-neutralized materials are, on average, better than the acid copolymers.

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