

Properties and Preparation of Compatibilized Nylon 6 Nanocomposites/ABS Blends Using Functionalized Metallocene Polyolefin Elastomer. I. Impact Properties

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ABSTRACT: The impact behaviors of nanoclay-filled nylon 6 (nano-nylon 6) blended with poly(acrylonitrile–butadiene–styrene) terpolymers (ABS) prepared through a twin screw mixing process were investigated here using metallocene polyethylene grafted maleic anhydride (POE-g-MA) as a compatibilizer to enhance the interface interaction. No clear effect of compatibilizer on the dispersion of clay and crystalline structure of nano-nylon 6 has been observed. In view of morphology and rheological behaviors, the effect of compatibilizer on the mechanical properties could be elucidated. It is found that impact strength increases with the addition of compatibilizer at various ABS compositions. Similar effects are also observed with decreasing test temperature at the nano-nylon 6/ABS blend composition of

80/20. As for thermal properties, the heat distortion temperature shows a marginal decrease in the nano-nylon 6/ABS blends. Rheological behavior indicates that increased viscosity is found for the investigated compatibilized systems. Through morphology observations, the etched ABS particle sizes tend to decrease with the addition of compatibilizer for the blends, but are larger with higher contents of ABS concentrations. Those observations account for impact behaviors of the investigated blends. © 2006 Wiley Periodicals, Inc. *J Appl Polym Sci* 100: 1364–1371, 2006

Key words: nylon 6 nanocomposite; poly (acrylonitrile–butadiene–styrene) terpolymers; metallocene polyethylene grafted maleic anhydride

INTRODUCTION

Polymer composites featuring both stiffness and strength have been of great commercial interest in the past decade. In particular, nanocomposites formed through hybrids of organic and inorganic materials dispersed in a nano scale have received much attention recently due to their outstanding performance in optical, electrical, mechanical properties, etc.

In such nanocomposites, nanoclay exfoliated nylon 6 (nano-nylon 6) first developed by Toyota Central Research Group to dramatically increase the heat distortion temperature (HDT) and hardness accompanying with improved flow behavior has led to many other advanced applications.^{1–3} Dubois et al. have thoroughly reviewed numerous approaches in other systems inclusive of epoxy, polyurethanes, polysiloxanes, etc.⁴

There is also of great commercial interest in polymer blends because the technology offers a potential and

economic route to new products with combined attractive features of each material.^{5–6} Nylon 6 is one of widely used engineering plastics. However, many drawbacks remain to be improved, such as brittleness and high moisture absorption. One of approaches was to blend nylon 6 with various types of rubbery polymers including styrene–ethylene–butylene–styrene block copolymer, etc. A compatibilizer has usually been incorporated to further improve impact resistance.⁷ To further endow nylon 6 with stiffness, ABS (acrylonitrile–butadiene–styrene terpolymer) is incorporated. However, the blend of nylon 6 and ABS gives relatively poor mechanical properties because of the low interfacial interaction. A compatibilizer is often required to enhance the interfacial stability. Several literatures have been reported on the efficiency of compatibilizer types.^{8–17} Paul et al. have carried out extensive studies on nylon 6/ABS blend in terms of compatibilizers, morphology, processing, etc.^{14–18}

The above compatibilizers are based on the interaction with acrylonitrile portion in most cases. It is interesting to see how other types of compatibilizers perform for an interaction with other portions such as high concentration of polybutadiene domain. A maleated metallocene polyethylene elastomer (POE) is chosen here for testing this hypothesis. The successful

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development of metallocene catalyzed PE was considered to be one of the most significant achievements in the polymer history for the past 20 years.^{19–21} Toughened nylons using maleated POE have been carried out by several authors.^{22–24} For the current work, POE serves as a compatibilizer as well as an impact modifier for nano-nylon 6 blends.

Owing to the inorganic dispersed phase, nanocomposites often exhibit outstanding stiffness with exceptional heat resistance. However, one of major mechanical properties, toughness, for those nanocomposite blends as normally seen in a conventional nylon system has not been thoroughly studied, especially for a melt blending process.^{25–30} Tjong et al. has recently investigated impact fracture toughness of polyamide-6/montmorillonite nanocomposites toughened with a maleated styrene–ethylene–butylene–styrene elastomer.³¹

In the first part of this study, the focus would be made on the mechanical properties of nano-nylon 6/ABS blends to demonstrate the effectiveness of POE-g-MA as a compatibilizer. Mechanical properties including tensile strength and Izod impact strength were investigated at various concentrations of ABS in the blends. A separate study to further characterize the detail dispersion of clay, crystallization, and thermal degradation properties through the addition of the compatibilizer is in progress and will be reported in the near future.

EXPERIMENTAL

Materials

The materials used in this study were nano-nylon 6, nylon 6, poly(acrylonitrile–butadiene–styrene) terpolymers (ABS), and polyethylene elastomer (POE). Nano-nylon 6 prepared by in situ polymerizing ϵ -caprolactam, and 4 wt % clay (swellable fluoromica treated with acid) was supplied from Unitika Co. under the trade name of M1030D. The density is 1.15 g/cm³ and the melt index is 11 g/10 min (2.16 kg, 190°C), as provided by the supplier. Nylon 6 (Sunylon 6N) was received from Formosa Chem. Fiber Co. The apparent density is 1.14 g/cm³. ABS (acrylonitrile : butadiene : styrene = 57 : 18 : 25 by wt %) was a commercial grade of Great Eastern Resins Co. under the trade name of ABS547P. Metallocene Polyethylene (POE) containing 24% octene comonomer (EG 8100) was manufactured by Du Pont Dow Elastomer Co.

Sample preparations

All pristine resins and blends were predried 4 h at 90°C in a dehumidified air-circulated oven prior to mixing. The grafting of maleic anhydride onto metallocene polyethylene (POE-g-MA) was prepared as a

compatibilizer through a single screw extruder. The grafting ratio is ~ 1 wt % determined by a titration method. The mixing of nano-nylon 6 and POE-g-MA formed a modified nano-nylon 6 at the first extruding step. This was followed by blending with ABS in the second extruding step using a corotating twin-screw extruder of type Kobe KTX-30 (screw diameter = 30 mm, length to diameter ratio L/D = 43.5). The screw speed was maintained at 100 rpm and the barrel temperatures were ranging from 170 to 230°C. The extrudate was pelletized and then oven-dried under the same condition. Tensile and Izod impact test specimens complied with ASTM standards D638 and D256, respectively, were then prepared using an injection-molding machine (Battenfield UNILO G4000).

Measurements

Structure and thermal characterizations

All test specimens were preconditioned at 23°C and 50% humidity for 24 h before tests. The Infrared spectra were recorded on a Fourier Transform Infrared Spectrophotometer (JASCO, 460 PLUS) at a resolution of 4 cm⁻¹ for 64 scans from 4000 to 700 cm⁻¹. X-ray diffraction (XRD) technique was employed to assess the level of clay destructure in the composites. The nano-nylon 6 crystal structure in different samples was confirmed by XRD as well. A Siemens D5005 X-ray unit operating at 40 kV and 30 mA was used to carry out the experiments at room temperature. The X-ray source was Cu K α radiation with a wavelength of 0.154 nm. The diffractograms were scanned in the 2 θ range from 1.5° to 30° at a rate of 0.01°/s. HDTs were recorded with a 4.6 kg/cm² load under a heating rate of 2°C/min using Toyoseiki Instrument model S-6M. The dynamic mechanical properties of the compression-molded samples were measured using a Perkin-Elmer DMA 7e system. The measurements were carried out in the three-point bending mode at a frequency of 1 Hz from –105°C to 150°C at a heating rate of 5°C/min in an air atmosphere.

Mechanical and rheological properties

Tensile measurements were conducted at a crosshead speed of 25 mm/min using an Instron Machine model 5567. Tensile strength, elongation, and Young's modulus were recorded. Notched Izod impact test was performed using ATLAS CS-137–25 at various temperatures. Rheological measurements were carried out using Gottfert 1501 to determine shear viscosity against shear rates ranging from 100 to 1000 s⁻¹ at 240°C.

Morphological observations

Morphology of fractured impact test specimens at –40°C was elucidated using a scanning electron micro-

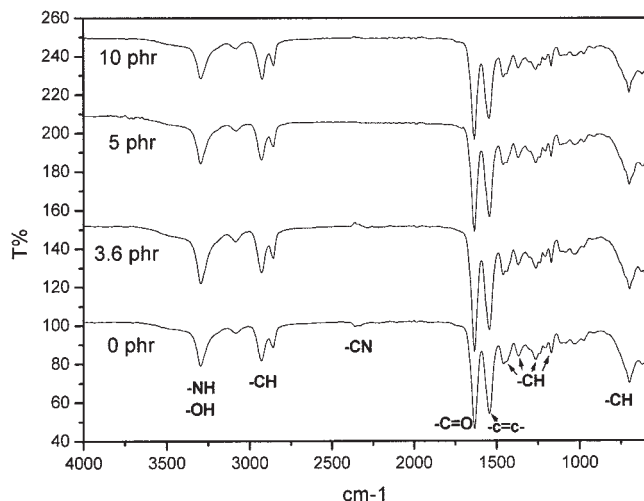


Figure 1 FTIR spectra of different functional groups of nano-nylon 6/ABS = 80/20 blends as a function of the compatibilizer contents of POE-g-MA.

scope (Topcon SM300). All samples were sputtered with gold before further characterization.

RESULTS AND DISCUSSION

In the first part of this study, the focus would be made on the mechanical properties of nano-nylon 6/ABS blends to demonstrate the effectiveness of POE-g-MA as a compatibilizer. A separate study to further characterize the detail dispersion of clay, crystallization, and thermal degradation properties through the addition of the compatibilizer is in progress and will be reported in the near future.

Structure characterization

Major regions of the FTIR spectra of nano-nylon 6/ABS blends are depicted in Figure 1 for a comparison, including absorption regions of —NH/—OH (hydrogen-bonded 3295 cm^{-1}), —CN (2256 cm^{-1}), C=O (1633 cm^{-1}), —C=C— (1543 cm^{-1}), and —CH ($2928, 1510\text{—}1450, 1264, 1185, 699\text{ cm}^{-1}$). Basically, these results for those systems indicate that hydrogen bonding exists within molecular chain for the blends. As the compatibilizer was incorporated, most of typical absorption bands remained unchanged. No appearance of new absorption band is observed. In general, the reaction of amine group on nylon and maleic anhydride on the compatibilizer to form functional groups of —C=O and —NH is overlapped in the original absorption regions, and so it is not clearly distinguishable on the compatibilized blends. This indicates a direct interaction between amine group on nylon and anhydride group on the compatibilizer is enhanced through the formation of nylon-POE block copolymer. This reaction has been well recognized in

the literature.^{8–17} Other characterizations such as dynamic properties and morphological observation would be of help to provide a suggestive molecular interpretation on these blends and will be discussed later.

Figure 2 shows the XRD patterns of nano-nylon 6 and its composite blends for air quench samples. As seen, no visible diffraction peak for clay is found, which could be attributed to the formation of swollen and disordered intercalated tactoids or the exfoliation of clay. To ensure the dispersion of clay, transmission electron microscope is actually required to assess clay dispersibility throughout the polymer matrix. Our other study of scanning electron microscope/energy dispersive spectrometer, transmission electron microscope, and thermal weight loss has been conducted and will be reported in a separate paper. As a matter of fact, the HDT of nano-nylon 6/ABS composites discussed later is higher than that of conventional nylon 6/ABS blends, which also supports this finding. Further, diffraction peaks of nano-nylon 6 are found around 2θ at 11° and 21.6° , which are the characteristic peaks of unstable γ -form changed from stable α -form at 20° and 24° for nylon 6 with the incorporation of clay.³² A diffraction peak of compatibilizer is found with 2θ at 20.1° indicative of the side branches crystalline structure.³³ In addition, no clear effect of compatibilizer on the dispersion of clay and crystalline structure of nano-nylon 6 has been observed. Cooling effect on the crystallization of nano-nylon 6 and compatibilizer is in progress.

Mechanical properties

Effects of blends composition on the notched Izod impact strength of nano-nylon 6/ABS blends as a

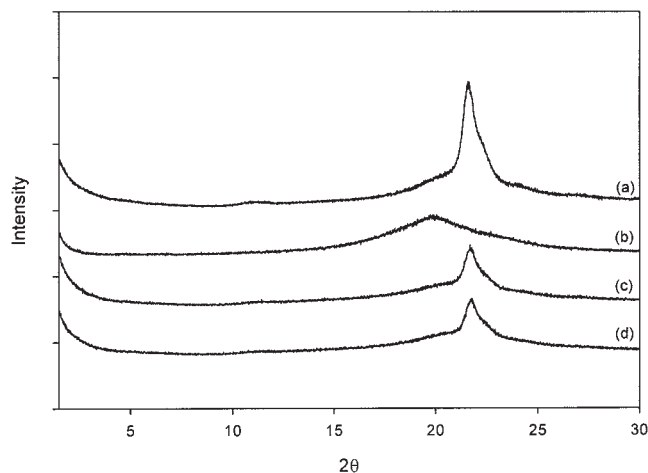


Figure 2 XRD patterns of (a) nano-nylon 6, (b) POE-g-MA, (c) nano-nylon 6/ABS, and (d) nano-nylon 6/ABS (10 phr POE-g-MA) under air quench condition.

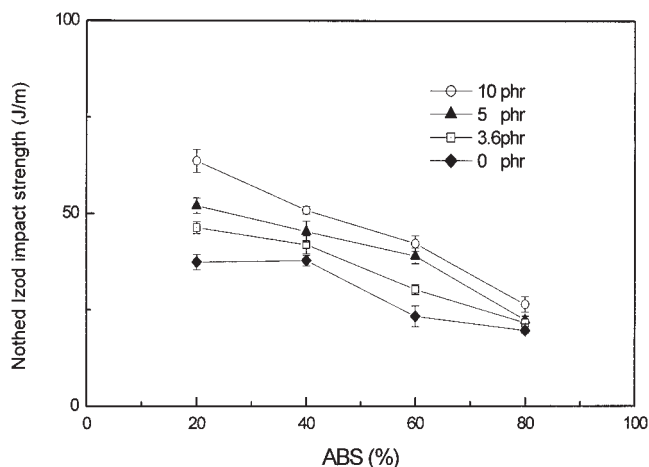


Figure 3 Effect of blends composition on the notched Izod impact strength of nano-nylon 6/ABS blends as a function of the compatibilizer contents of POE-g-MA.

function of the compatibilizer contents of POE-g-MA are shown in Figure 3. This incompatible nano-nylon/ABS system is found as expected, which leads to the decrement of impact resistance with increasing ABS content. By incorporating POE-g-MA into all nano-nylon/ABS blends, a positive effect in impact strength for these compatibilized composites has been seen when compared with an unmodified system. This indicates a direct interaction between amine group on nylon 6 and anhydride group on the compatibilizer is enhanced. This enhanced interfacial interaction between nano-nylon 6 and ABS will further be discussed and confirmed in detail later in the analysis of dynamic properties and fractography. Still, impact strength of compatibilized blend does not increase with increasing ABS content using this approach. Further study is necessary to elucidate this impact behavior. Note that there is about a 1.7 times maximum increase at nano-nylon 6/ABS composition of 80/20 containing 10 phr of compatibilizer, reaching the value of 64 J/m. Likewise, a moderate increase up to 3.4 times at nylon 6/ABS blends without nanoclay when a comparison is made at the same composition containing 10 phr of compatibilizer has been observed, reaching the value of 237 J/m.³⁴ This also indicated an effective role of POE-g-MA as a compatibilizer.

To ensure this compatibilized effect, one could leave out ABS resin and compare the impact strength of POE-g-MA modified nano-nylon 6. A conventional nylon 6 has been employed as a comparison here.³⁴ As mentioned in the introduction, the compatibilizer POE-g-MA can also serve as an impact modifier. Effects of modifiers contents on the notched Izod impact strength are shown in Figure 4 for nano-nylon 6 and nylon 6, respectively. Toughening effect is observed for both resins. Impact strength increases from 65 to 403 J/m at a modifier dosage of 0 to 10 phr in the case

of nylon 6. Though a supertough condition is not obtained even at 10 phr of modifier, the value is slightly higher than 370 J/m listed in the literature.²⁴ Similar findings are reached for nylon 6 systems as well, indicating a certain interaction between maleic anhydride on POE and amine group on nylon is reached. Interestingly, impact strength only increases from 40 to 101 J/m, a 2.5 times increase, at the corresponding amounts of modifier in the case of nano-nylon 6. Similar results were also found for impact fracture toughness of polyamide-6/montmorillonite nanocomposites toughened with a maleated styrene-ethylene-butylene-styrene elastomer, and brominated isobutylene/paramethylstyrene elastomer toughened polyamides as well as their toughened nanocomposite counterparts. Impact strength for toughened polyamides is 1.5–6 times higher than that of toughened polyamide nanocomposites.^{31,35}

Two possible reasons including nylon 6 sources and clay effect may account for this difference in impact enhancement for nylon 6 and nano-nylon 6 toughened with POE-g-MA, respectively. Yet, both pristine resins give a similar order of impact strength in our study. So, nylon sources could not solely explain the aforementioned phenomenon. Instead, nano-clay incorporated deteriorated toughening efficiency due to its rigidity.^{30–31} In their pioneer preparation of nylon 6 based nanocomposite, Na⁺-montmorillonite organically modified by protoned α,ω -amino acid was used to be swollen by ϵ -caprolactam monomer to initiate ring opening polymerization.^{1–2} Inevitably, the available amine group for the reaction with maleic anhydride group on the modifier becomes insufficient, which accounts for this relatively low degree of enhancement in impact strength. This is attributed to a loss of some amide and amine group forming a complex with hydroxyl group located within the gallery of

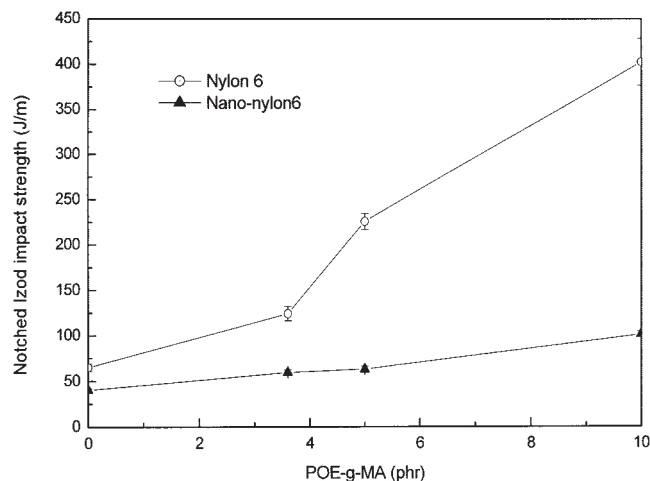


Figure 4 Effect of the compatibilizer contents on the notched Izod impact strength of nano-nylon 6/POE-g-MA and nylon 6/POE-g-MA.

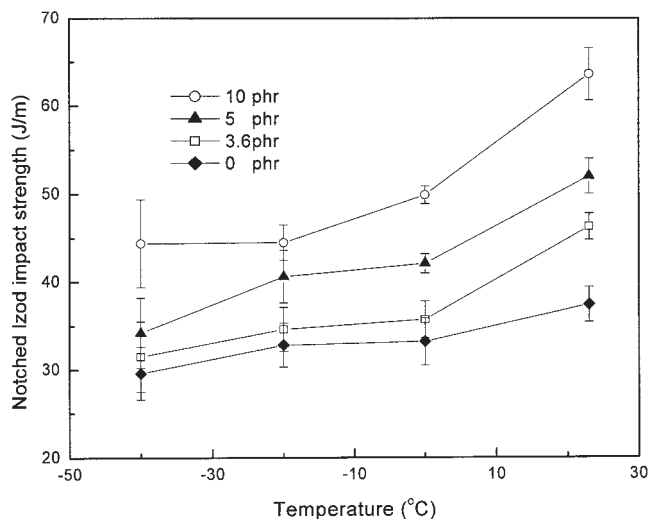


Figure 5 Effect of temperature on the notched Izod impact strength of nano-nylon 6/ABS = 80/20 blends as a function of the compatibilizer contents of POE-g-MA.

clay moiety during a preparation of nylon 6 nanocomposite. The impact strength of nylon 6/POE-MA/clay also changed through different mixing sequences.³⁰ This work demonstrates this possibility on the nylon 6 nanocomposite and its blend as well. Further study is in progress to elucidate the dispersion of clay based on the TEM and X-ray diffraction technique under different cooling condition for gaining an insight of these observations.

Effect of temperature on the impact strength of nano-nylon 6/ABS at a composition ratio of 80/20 as a function of the compatibilizer contents of POE-g-MA is depicted in Figure 5. Impact strength continues to decrease with decreasing test temperature due to a restriction in molecular motion. Impact strength at low temperature remains at certain level, about two third of value at room temperature for blend composition containing 10 phr of compatibilizer. In addition, impact strength increases as the contents of compatibilizers increase for all test temperatures. With such an increase in impact strength, POE-g-MA does not only employ as a compatibilizer but also serve as an impact modifier because of its elastomeric nature. The rationale for this observed behavior has been described previously.

Figure 6 shows the effect of the compatibilizer contents on tensile strength and tensile modulus of nano-nylon 6/ABS = 80/20 blends. Regarding the variation of tensile strength after the incorporation of compatibilizer, a decrement for nano-nylon/ABS blends is ~25%. Likewise, a 20% decrease in modulus is found for the corresponding increase of compatibilizer. These observations are indebted to an elastomeric nature of metallocene polyethylene. Noted that tensile strength of nano-nylon is about 1.6 times higher than that of ABS. The blends

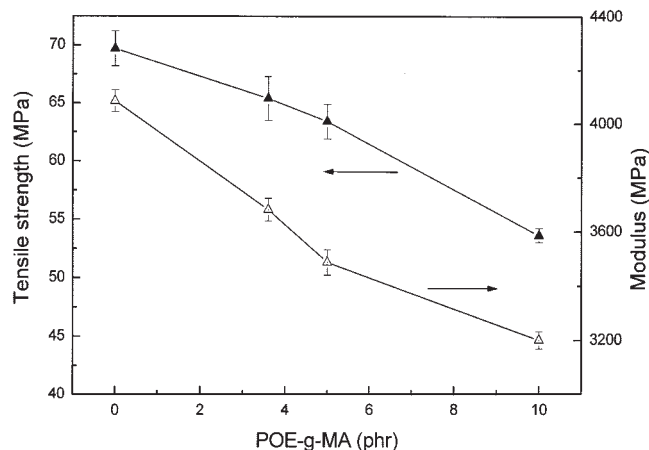


Figure 6 Effect of the compatibilizer contents on tensile strength and tensile modulus of nano-nylon 6/ABS = 80/20 blends.

apparently suffer a slight loss of tensile strength when blended with ABS. The enhancement in impact resistance is obviously more effective than that in tensile strength and tensile modulus.

Thermal and rheological properties

To further elucidate the nanoclay in the role of thermal resistance, the HDT recorded for nano-nylon 6/ABS blends as a function of the compatibilizer contents of POE-g-MA is shown in Figure 7. The HDT decreases with increasing the loadings of the compatibilizer. The value of HDT decrease from 183°C to 166°C, only a 17°C decrease for nano-nylon 6 blends. HDT appears to level off at compatibilizer contents above 5 phr. Apparently, clay demonstrates its unique feature in enhancing thermal behavior even if blends contain certain amounts of elastomeric type of compatibilizer.

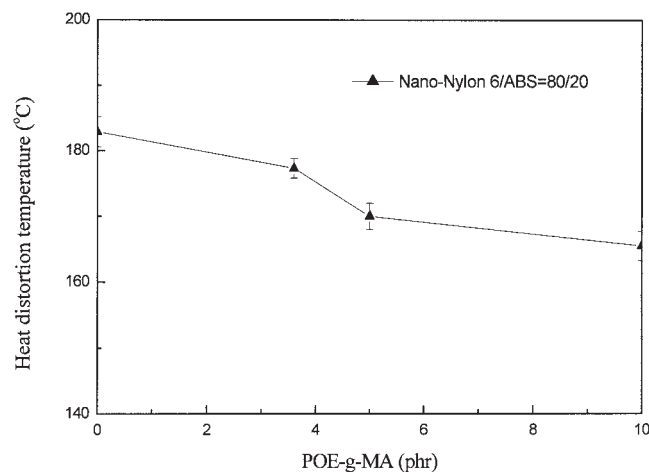


Figure 7 Effect of the compatibilizer contents on the HDT of nano-nylon 6/ABS = 80/20 blends.

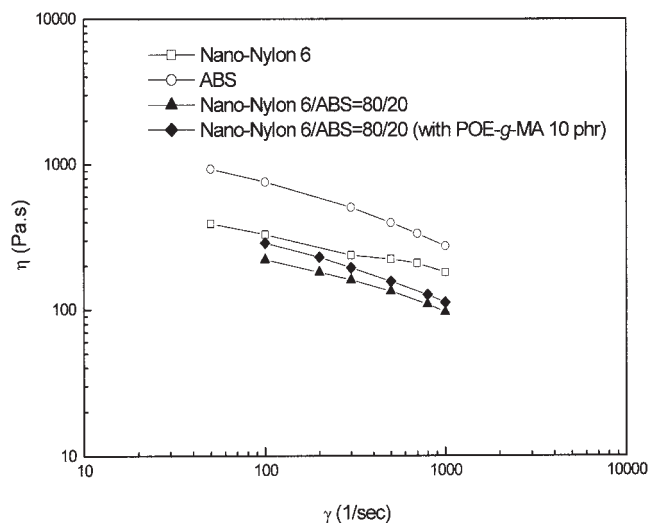


Figure 8 Effect of shear rate on the shear viscosity of nano-nylon 6/ABS blends as a function of the compatibilizer contents of the POE-g-MA.

Effect of shear rate on the shear viscosity of nano-nylon 6/ABS blends as a function of the compatibilizer contents of the POE-g-MA is shown in Figure 8. The viscosity of all resins follows non-Newtonian flow behavior of general polymers and decreases with increasing shear rate. The viscosity of ABS is about two times higher than that of nano-nylon 6. Based on this difference in the viscosity ratio and chemical affinity, the mixing of nano-nylon 6 and ABS gives an incompatible blend, as expected. Therefore, the viscosity of blend is generally lower than that of each base resin due to the lack of strong interaction within interfaces. With the incorporation of compatibilizer, the viscosity increases again, which is indebted to the enhanced interfacial interaction through the formation of nylon-POE block copolymer. Previous enhancement in mechanical properties such as toughness has been observed. The investigated systems do show compatibilization effect in the rheological point of view.

Dynamic properties

Figure 9 depicts the $\tan \delta$ as a function of temperature for nano-nylon 6/ABS blends with and without compatibilizer from -105 to 150°C . There are three peak values at -75°C , 59°C , and 106°C , representing each glass transition temperature for polybutadiene, nano-nylon 6, and polystyrene segments, respectively. With the incorporation of 10 phr compatibilizer, two peak values shifts slightly and become -56°C , 58°C , and 119°C , correspondingly. As one expect, the interaction between maleic anhydride moiety grafted on metallocene POE and nano-nylon 6 is enhanced, which causes the glass transition temperature of polybutadiene domain shifted toward higher temperature. Yet, the peak

value of nano-nylon 6 remains largely unchanged. In addition, the clay nature of restricting molecular motion of acrylonitrile-styrene matrix seems to be possible due to the previous enhanced interaction, which causes the glass transition temperature of polystyrene increase slightly. Note that this slight increment does not contribute the marginal decrease in the HDT with the introduction of compatibilizer as seen in Figure 6.

There are two possibilities about the role of POE-g-MA played in this system. At first, POE-g-MA may form a discrete phase and reside in the matrix of nano-nylon as an impact modifier. Secondly, POE-g-MA may reside at the interface of nano-nylon and SAN matrix of ABS as a compatibilizer. In light of this good mixing performance provided by using twin screw extruder, the chance of POE on the compatibilizer to interact with polybutadiene should not be ruled out. Owing to a high compatibility of MA and SAN, MA onto POE could readily react with nylon at the interface of nano-nylon and SAN matrix to form reactive compatibilizing precursors. This argument is partly supported by the molecular motion described in dynamic properties and the decreased ABS domains in the following morphological observations, though a further study is needed to clearly elucidate the possible location of the compatibilizer.

Morphology

Figure 10 shows the distinct morphology of fracture surface of impact specimens for unmodified and compatibilized nano-nylon 6/ABS blends at a specific composition of 80/20 based on the SEM observations. Observed holes with a dimension of a few micrometer in size represent etched ABS particles. With the addition of the compatibilizer, particle sizes tend to decrease, suggesting a good interfacial bonding between

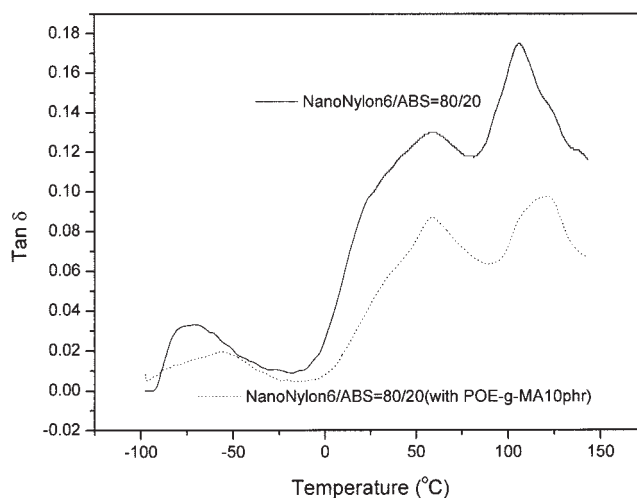


Figure 9 $\tan \delta$ for nano-nylon 6/ABS = 80/20 blends.

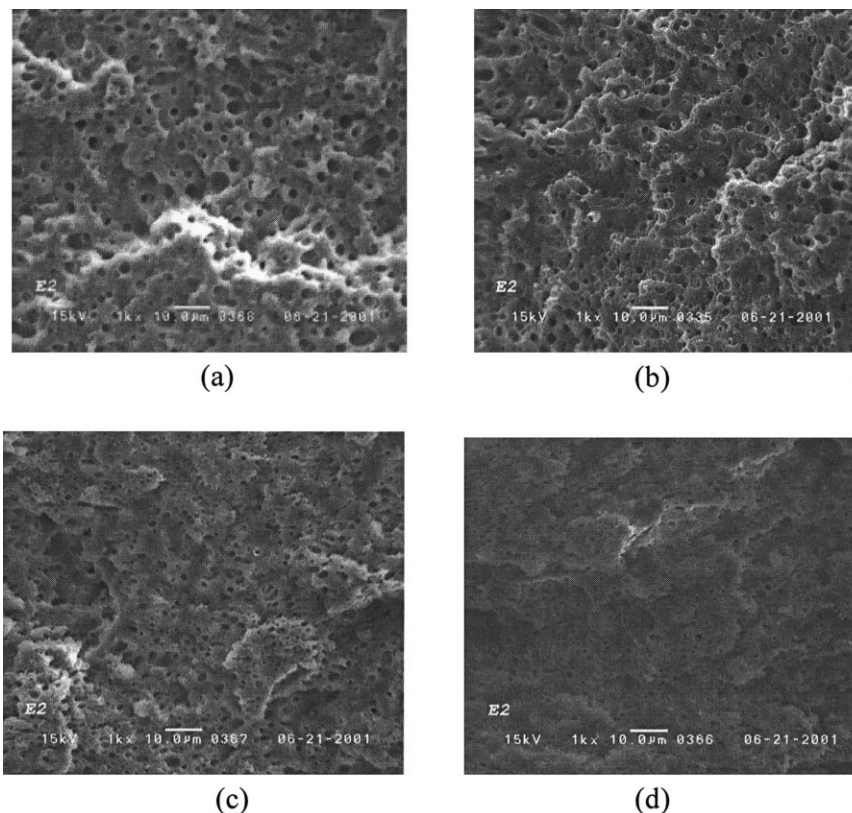


Figure 10 SEM photomicrograph of nano-nylon 6/ABS = 80/20 blends (a) 0 phr (b) 3.6 phr (c) 5 phr (d) 10 phr of POE-g-MA.

nano-nylon 6 matrix and ABS dispersed phase through the formation of nylon-POE block copolymer. Other compositions of blends also indicate a similar behavior. The result is parallel to the previous finding in the enhancement in impact strength. Compatibilized blends confer smaller particle sizes in all other compositions. One may argue that POE-g-MA is only served as an impact modifier. However, if this is the case, then the hole sizes should not decrease with increasing POE-g-MA content and molecular interaction should not occur in the aforementioned dynamic properties. Thus, the current finding suggests that POE-g-MA serves not only as an impact modifier because of its elastomeric nature but also as a compatibilizer in this system. Our other work also has come to a preliminary result to support this finding, using a different type of compatibilizer with nonpolar base resin exhibiting nonelastomeric nature.³⁴

Figure 11 shows the SEM micrographs of the effect of ABS composition on the peculiar morphology for compatibilized nano-nylon 6/ABS blends. Basically, ABS forms dispersed phase within nylon matrix at a low concentration of ABS composition, as one would expect. Likewise, one might expect a continuous phase for a higher loading of ABS resin. However, a dispersed phase with relatively large dimension in size is observed, which could be ascribed to a low viscosity

ratio of nano-nylon 6 to ABS. Basically, with increasing content of ABS composition, ABS dispersed phases tend to increase in size, up to 10 μm at the composition of 20/80. This unexpected results account for lower impact strength with increasing dosages of ABS, which indicates that the system unfortunately does not exploit the high impact feature of ABS.

CONCLUSIONS

The impact behaviors of nanoclay-filled nylon 6 blended ABS were investigated here using metallocene polyethylene grafted maleic anhydride (POE-g-MA) as a compatibilizer to enhance interface interaction through the formation of nylon-polyolefin reactive compatibilizing precursors. No clear effect of compatibilizer on the dispersion of clay and crystalline structure of nano-nylon 6 has been observed. Impact strength increases for nano-nylon 6/ABS blend systems with the addition of compatibilizer at various ABS compositions. Similar effects are also found with decreasing test temperature at the representative blend composition of 80/20. As for the effects of ABS compositions, impact strength and tensile strength decreases with increasing the content of ABS. The HDT shows a marginal decrease. Rheological behavior indicates that increased viscosity is found for compati-

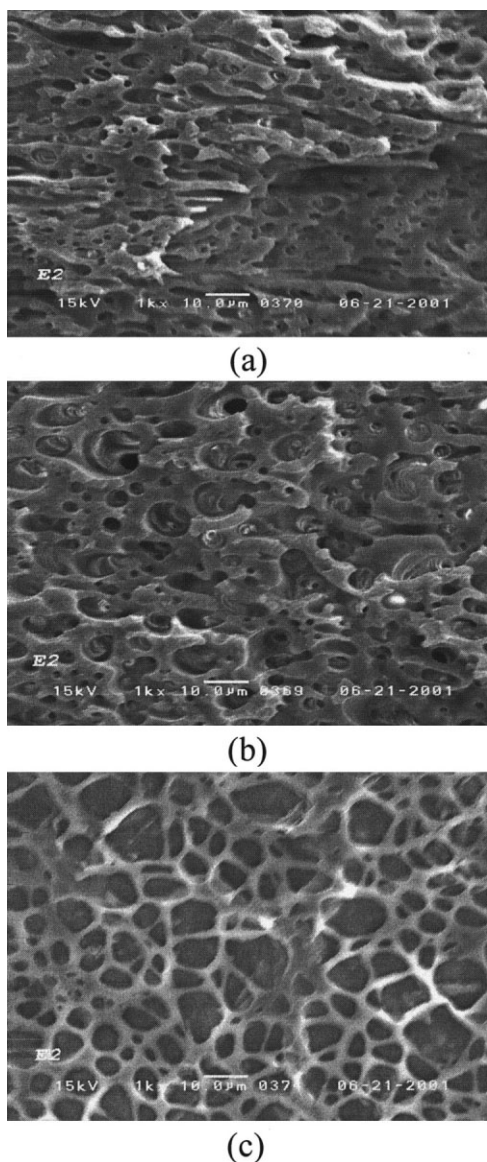


Figure 11 SEM photomicrograph of nano-nylon 6/ABS blends at different compositions with 10 phr of POE-g-MA. (a) 60/40, (b) 40/60, and (c) 20/80.

bilized systems, which confirms the positive effect of compatibilizer. Dynamic properties indicate an enhanced molecular interaction. Through morphology observations, the etched ABS particle sizes tend to decrease with the addition of compatibilizer for the investigated blends, but are larger with higher contents of ABS concentrations. This study basically paves the way for a better understanding of further approaches in improving toughness of nano-nylon 6 nanocomposite blends in an attempt to reach optimum properties inclusive of its unique feature in rigidity and thermal properties. A separate study to further characterize the dispersion of clay, crystallization, and thermal behaviors through the addition of the compatibilizer is in progress and will be reported in the near future.

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