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Evaluation of polypropylene grafted with maleic anhydride and styrene as a compatibilizer for polypropylene/clay nanocomposites

Jiseon Lee · Jin Kwang Kim · Younggon Son

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Abstract Among modified Poly(propylene)s (PPs) grafted with polar monomers, PP grafted with maleic anhydride (PP-*g*-MAH) is known to be the most efficient compatibilizer for PP/clay nanocomposites, since it provides well-dispersed nanostructures and yields optimal physical properties of the nanocomposites. One drawback of this material, however, is that it becomes brittle and its viscosity decreases drastically, leading to nanocomposites with low toughness as the graft degree of MAH increases. Therefore, there is a limitation to increasing both stiffness and toughness of PP/clay nanocomposites with PP-*g*-MAH. In this study, we investigated the performance of a PP grafted with maleic anhydride and styrene (PP-*g*-MAH-St) as compatibilizers in PP/clay nanocomposites. It was found that the incorporation of styrene as a comonomer prevents molecular weight reduction of the PP main chain upon high loading of a radical initiator for high graft degree of MAH. The compatibilizers (PP-*g*-MAH-St) thus obtained show good compatibilizing performance in PP/clay nanocomposites. The PP/clay nanocomposites compatibilized by PP-*g*-MAH-St show both high stiffness and toughness, which is accomplished by using a compatibilizer of higher viscosity compared with PP-*g*-MAH.

Keywords PP/clay nanocomposites · PP-*g*-MAH-co-styrene · Melt grafting

Introduction

Poly(propylene) (PP) is one of the most versatile commodity polymers. Owing to PP's low cost, relatively good properties, and steady improvement of its physical properties, new applications are continuously expanding, and it is replacing ABS,

J. Lee · J. K. Kim · Y. Son (✉)
Advanced Materials Science and Engineering, College of Engineering, Kongju National University, Cheonan, Chungnam 331717, South Korea
e-mail: sonyg@kongju.ac.kr

HIPS, and some engineering plastics in many fields [1]. Although PP has seen widespread application, its limited stiffness is an obstacle to broadening its utilization as a high performance plastic. The stiffness of PP is known to be considerably improved by the incorporation of well-dispersed nano silicates. PP is non-polar by its chemical nature whereas layered silicates are inherently polar materials, and thus dispersion of silicate layers is extremely difficult unless PP is appropriately modified. The most widely used method of modification for PP/clay nanocomposite applications is the grafting of polar monomers onto PP main chains in the presence of a radical initiator by melt grafting.

Because PP is the most widely used polymer, almost all polar monomers have been investigated for grafting onto it, and the performance of the modified PPs thus obtained as compatibilizers for PP/clay nanocomposites has been extensively evaluated. Such modified PPs include amine-functionalized PP (PP-*g*-NH2) [2], silane grafted PP [3], itaconic acid grafted PP [4], diethylmaleate grafted PP [5], carbamyl maleamic acid grafted PP [5], acrylic acid grafted PP [6], glycidyl methacrylate grafted PP (PP-*g*-GMA) [6], hydroxy ethyl methacrylate grafted PP (PP-*g*-HEMA) [7], etc. [8–11], but most studies have reported only moderate improvement compared to PP grafted with maleic anhydride (PP-*g*-MAH). In fact, the majority of studies on PP/clay nanocomposites have been carried out using PP-*g*-MAH, because it provides the best reinforcing effect among all modified PPs investigated to date.

PP-*g*-MAH has been comprehensively studied, especially as a compatibilizer in PP/clay nanocomposites [6, 12–16]. Although it provides the best reinforcing effect, it still has a major drawback. The grafting of MAH onto PP is generally carried out by a melt grafting method in the presence of radical initiators. The radical initiator, however, increases not only the formation of radicals, but also chain scission of a polymer. Therefore, the molecular weight of PP-*g*-MAH produced by the melt grafting method decreases with the MAH content, and PP-*g*-MAH becomes more brittle as the amount of grafted MAH increases. It have been widely reported that higher MAH content in PP-*g*-MAH increases the stiffness of PP/PP-*g*-MAH/clay nanocomposites but decreases the toughness of the nanocomposite due to reduced viscosity and the decreased molecular weight of the PP-*g*-MAH [13–17].

Recently, it has been found that the incorporation of styrene (St) as a comonomer in the melt grafting process prevents the chain scission and increases the grafting degree of glycidyl methacrylate (GMA) and MAH on polyolefin [18–22]. We anticipate that PP grafted with maleic anhydride/styrene (PP-*g*-MAH-St) thus fabricated will provide better efficiency than PP-*g*-MAH as a compatibilizer in PP/clay nanocomposites due to the higher degree of grafting and the increased viscosity of the compatibilizer. While a few studies have reported that PP-*g*-MAH-St could be successfully obtained by a melt grafting method, there has been no study reporting the use of PP-*g*-MAH-St thus obtained as a compatibilizer in PP/clay nanocomposites. In this study, we fabricated PP-*g*-MAH-St with incorporation of styrene comonomer with various MAH/St ratios and evaluated the efficiency as a compatibilizer in PP/clay nanocomposites.

Experimental

Materials

Two different PPs were used in this study. Commercial PP, used for fabrication of PP-*g*-MAH and PP-*g*-MAH-St, was obtained from Samsung Total Chemicals (grade name: BB110 MFI: 0.5 dg/min) and another PP with much lower viscosity (MI: 30 dg/min) purchased from Korean Petrochemical Co (grade name: CB5230) was used as a matrix phase in the PP/clay nanocomposites. Organoclay (trade name: Cloisite 20A) based on dimethyl, dehydrogenated tallow quaternary ammonium was purchased from Southern Clay Products, Inc. Maleic anhydride (MAH), styrene, and dicumyl peroxide were from the Aldrich Chemicals.

Preparations of the compatibilizers

Preparation of the compatibilizers and the nanocomposites, respectively, was carried out in an extensional batch mixer (see Ref. [23] for details on the extensional batch mixer). Prior to compounding, all the raw materials were dried in a vacuum oven at 80 °C for a minimum of 12 h. Melt compounding was performed using an extensional batch mixer with a shear rate of $\sim 2000\ s^{-1}$ at 180 °C for 5 min.

The powdery PP was tumble-mixed with a polar monomer (maleic anhydride) and dicumyl peroxide as an initiator in a sealed plastic bag. The mixture was immediately put into the batch mixer for compounding. The content of MAH was varied from 1.0 to 4.0 phr. The ratio of MAH/initiator was set to 10. The compatibilizers incorporating the styrene comonomer were also prepared in the same manner. In this article, the following code is used for the compatibilizer:

The code PP-*g*-MAH2.0St2.0 represents a compatibilizer prepared from a mixture of 100 g of PP, 2.0 g of MAH, 2.0 g of styrene, and 0.2 g of an initiator. By the same rule, PP-*g*-MAH1.0 represents a compatibilizer prepared from a mixture of 100 g of PP, 1.0 g of MAH, and 0.1 g of an initiator.

Preparation of the nanocomposites

Since nanocomposites prepared by the masterbatch method provide better micro-scale dispersion, all composites were prepared by a two-step masterbatch method. The first step entailed blending the PP and clay at a ratio of 8/2 w/w (PP/Cloisite20A), creating a masterbatch. In the second dilution step, each masterbatch sample was dry mixed with predetermined amounts of the compatibilizer and PP, and subsequently compounded in the mixer. The amounts of the masterbatch, compatibilizer, and PP were 45, 40, and 15 (by weight), respectively. Therefore, the percentages of clay, compatibilizer, and PP in the nanocomposites were 9, 40, and 51, respectively.

Characterizations

Wide angle X-ray data were collected on a Rigaku D/MAX-IIIC X-ray diffractometer (Cu Kα radiation, wavelength = 1.5418) with accelerating voltage

of 40 kV. Diffraction spectra were obtained over a 2θ range of 1.28–10°. Fourier transform infrared (FTIR) spectra were recorded on a PerkinElmer FTIR spectrometer. Films were obtained by hot-plate pressing, and then dried at 80 °C under vacuum for 6 h before analysis. FTIR spectra were recorded on a PerkinElmer FTIR spectrometer. Films were obtained by hot-plate pressing, and then dried at 80 °C under vacuum for 6 h before analysis.

Blended samples were dried and injection-molded into a dog-bone shaped tensile bar and subjected to an Izod impact test. Tensile properties were tested using a universal mechanical testing machine (Model Hounsfield H25KS) at a crosshead speed of 20 mm/min (ASTM-D638). Notched Izod impact tests were carried out at room temperature according to the ASTM-D256 standard method with specimens of 3.2 mm thickness. Melt flow index (MFI) was measured at 230 °C and 2.16 kg load (ASTM D1238).

Results and discussion

The grafting reaction of MAH onto PP was confirmed by FTIR spectra. Figure 1 shows the FTIR spectra of PP-*g*-MAH and PP-*g*-MAH-St fabricated in this study. Characteristic peaks of MAH are clearly seen at ca. 1782 cm^{-1} in all PP-*g*-MAH and PP-*g*-MAH-St. We removed unreacted MAH, styrene monmer, ungrafted polystyrene (PS), and poly(styrene-co-maleic anhydride) (SMA), which are formed during the melt grafting process, via use of chloroform in a soxhlet extraction device. When the physical blend of PP with MAH, styrene, PS, and SMA was processed in the soxhlet device, no residual MAH was detected in the FTIR spectrum. Therefore, the peak at ca. 1782 cm^{-1} confirms that the MAH group is successfully grafted onto the PP backbone. It is also noted that the intensity of the MAH peaks increases with the amount of MAH added, which implies that the grafting yield increases with the amount of MAH added.

It is also noteworthy that the intensity of the MAH peaks increases with the amount of styrene comonomer at a given MAH content. MAH has very low reactivity with MAH itself, and thus MAH grafted onto polyolefins is present in the form of a single MAH or succinic anhydride [24, 25]. As a result, the grafting yield of MAH onto a polymer is limited. When styrene is incorporated as a comonomer, styrene is copolymerized with MAH and can form a chain structure composed of MAH and St (most likely in an alternating sequence) in a grafted side chain. Therefore, incorporation of styrene is regarded as an effective means of increasing the grafting yield of MAH. It was also observed that the grafting yield continuously increases with the St/MAH ratio when the amount of MAH added is relatively low (1.0 and 2.0 phr). However, the grafting yields increase and level off at a St/MAH ratio = 1.0 when the amount of MAH added is 4.0 phr.

There have been contradictory findings regarding the effect of the St/MAH ratio. Chen et al. [26] reported that the grafting yield is the highest at a ratio of St/MAH = 1 and decreases with a further increase of the St/MAH ratio in the maleation of poly(ethylene-*co*-octene). Li et al. reported the same results when the concentration of the initiator (dicumyl peroxide) is high in the maleation of PP, but

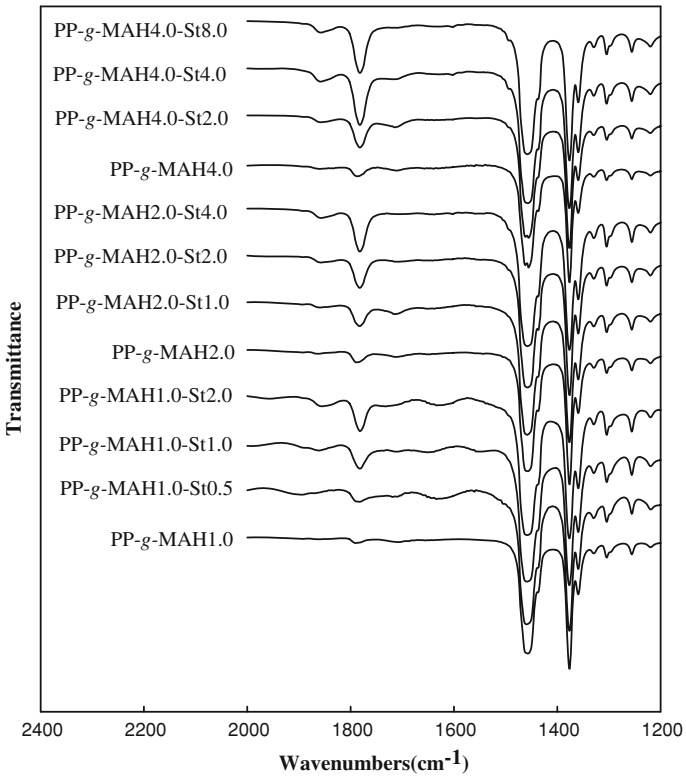

Fig. 1 FTIR spectra of PP-*g*-MAH and PP-*g*-MAH-St

they observed that the grafting yield continuously increases with the St/MAH ratio when the concentration of the initiator is low (less than 0.2 phr) [22]. In the present study, the concentrations of DCP were higher than 0.2 phr for all compositions. Numerous factors affect the grafting reaction of MAH onto the polyolefin (e.g., concentration of MAH, concentration of the initiator, initiator/MAH concentration, extrusion temperature, extrusion rate, etc.). Thus, it is inferred that the different experimental conditions as compared with other studies, excluding the St/MAH ratio and the concentration of initiator, account for the different results.

Homogeneous dispersion of silicate layers within a polymer matrix is a critical factor to achieve good physical properties of polymer/clay nanocomposites. Since PP is one of the most non-polar polymers and clay is a relatively polar material, it is difficult to obtain a well-dispersed nanostructure in PP/clay nanocomposites unless a proper compatibilizer is incorporated. Thus, using XRD, we investigated the efficiency of PP-*g*-MAH-St in the dispersion of silicate layers. Figure 2 shows the XRD patterns for the PP/clay nanocomposites compatibilized by PP-*g*-MAH and PP-*g*-MAH-St. The vertical line in Fig. 2 represents the position of the d_{001} peak of the organoclay (Cloisite 20A) itself. Based on the XRD data, the d_{001} spacings (clay interlayer distance) were calculated and the values are shown in each curve. As can

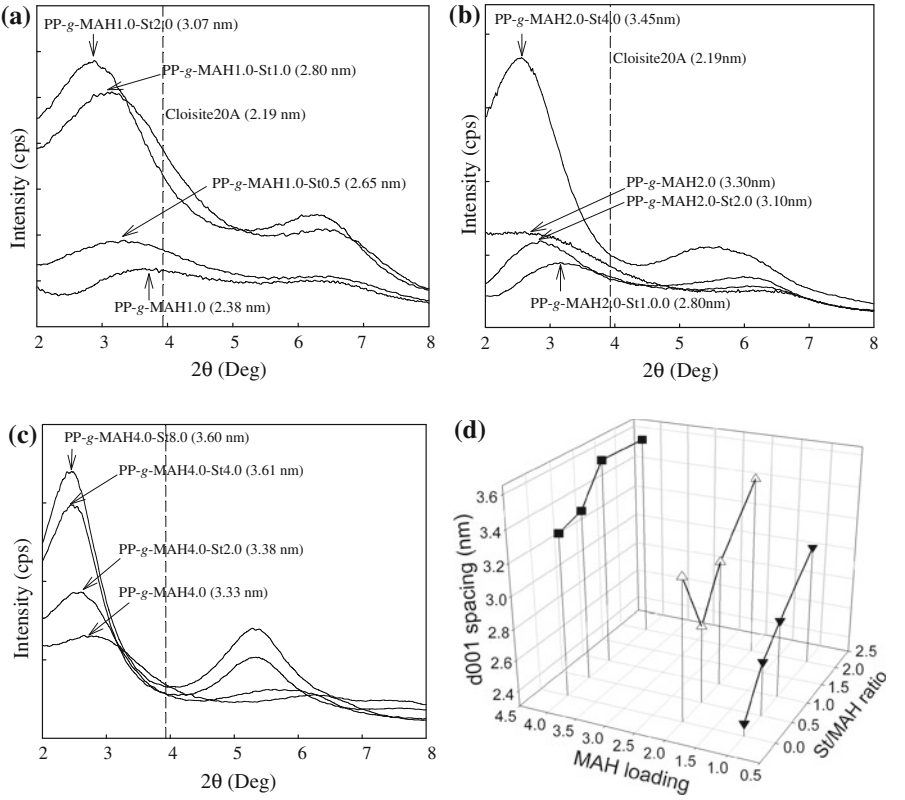

Fig. 2 X-ray diffraction patterns of PP/clay nanocomposites compatibilized by PP-*g*-MAH and PP-*g*-MAH-St. The *vertical line* represents the position of the d_{001} peak of the organoclay (Cloisite 20A). *Numbers* shown in each peak represent the clay interlayer distances of the composites. **a** 1.0 phr of MAH, **b** 2.0 phr of MAH, **c** 4.0 phr of MAH, and **d** 3D plot of interlayer distance as fuction of MAH loading and St/MAH ratio

be seen, all composites show the intercalated structures. The d_{001} peaks of the nanocomposites are observed to shift to lower angles compared with the peak of the organoclay, indicating that the clay interlayer distances are increased. It is seen that the interlayer spacing increases with the grafting degree of MAH and levels off at a higher graft degree (The interlayer spacing of the nanocomposites containing PP-*g*-MAH2.0St0.0 ~4.0 is much greater than that of the nanocomposites containing PP-*g*-MAH1.0St0.0 ~2.0, while the interlayer spacing of the nanocomposites containing PP-*g*-MAH2.0St0.0 ~4.0 and PP-*g*-MAH4.0St0.0 ~8.0 does not show a large increase with MAH content or St/MAH ratios). It is known that PP-*g*-MAH having higher MAH content provides better dispersion of the silicate layers due to its increased polarity. However, when styrene is incorporated as a comonomer, the sizes of grafted functional groups become too large for easy insertion into the clay platelets. Thus, it is inferred that there is an optimum level of MAH content for the largest interlayer spacing of the clay, and it is estimated that 1.0–2.0 phr of MAH is the best level of loading.

Improved dispersion of nano silicates by PP-*g*-MAH and PP-*g*-MAH-St is reflected in improved physical properties. Figures 3 and 4 show stress–strain curves and Izod impact strengths of the PP/clay nanocomposites investigated in this study. It is seen that the stiffness of the composites (tensile modulus and tensile stress at a yield point) slightly increase with the St/MAH ratio at given MAH content. However, when looking at the effect of MAH content on the stiffness at the same St/MAH ratio, it is seen that the stiffness is not significantly affected by MAH content. It was reported that the stiffness of PP/clay nanocomposites compatibilized by PP-*g*-MAH increases with the grafting degree of MAH, reaches a maximum value at MAH content of about 0.5–1.0 wt%, and thereafter decreases to a constant value [13, 14]. In our study, we could not measure the grafting degree of MAH directly; however, most likely it is much higher than the critical value at which the stiffness is maximal, because the MAH characteristic peaks in the FTIR spectra are very strong compared with a previous study where the grafting degree of MAH in PP-*g*-MAH was well over 1.0 wt% [23]. Moreover, the amount of compatibilizers used in this study is higher than in previous studies [13, 14]. Therefore, the stiffness of the nanocomposites is already in a saturated region and is not affected by the MAH content.

Previous studies [7, 13, 14] reported that PP/clay nanocomposites compatibilized by PP-*g*-MAH become more brittle as the grafting degree of MAH increases, and this is due to the following reason. In order to obtain PP-*g*-MAH with higher MAH content, both the radical initiator and MAH must be added in increased amounts. Since the radical initiator used for maleation of PP not only provides radical formation, but also causes chain scission of the polymer chains, the molecular weight of PP-*g*-MAH produced by the melt grafting method is very low compared with a pure PP. The molecular weight of PP-*g*-MAH decreases and PP-*g*-MAH becomes more brittle as the grafting degree of MAH increases. In addition, the miscibility between PP and PP-*g*-MAH decreases with MAH content, since the higher level of polar MAH group prevents good mixing of PP-*g*-MAH with the non-polar PP, which deteriorates the toughness of the nanocomposite. Therefore, it is impossible to obtain both high stiffness and toughness when the nanocomposite is compatibilized by PP-*g*-MAH.

In this study, we evaluated the efficiency of PP-*g*-MAH-St as a compatibilizer in a PP/clay nanocomposite for the first time. As can be seen in Figs. 3 and 4, the toughness (elongation at break and Izod impact strength) of the nanocomposite compatibilized by PP-*g*-MAH decreases with MAH content but increases rapidly without sacrificing stiffness when styrene is incorporated as a comonomer. Both higher toughness and stiffness are achieved when PP-*g*-MAH-St is used as a compatibilizer. This result could never be achieved when PP-*g*-MAH is used as a compatibilizer. The high level of toughness at a higher grafting degree of MAH can be achieved because the incorporation of styrene as a comonomer helps prevent chain scission, as explained previously.

Figure 5 shows the melt flow indices (MFI) of PP-*g*-MAH and PP-*g*-MAH-St prepared with various St/MAH ratios. It is seen that the MFI (inverse of viscosity and molecular weight) is significantly decreased with the incorporation of styrene. When PP-*g*-MAH is used as a compatibilizer in PP/clay nanocomposites, the

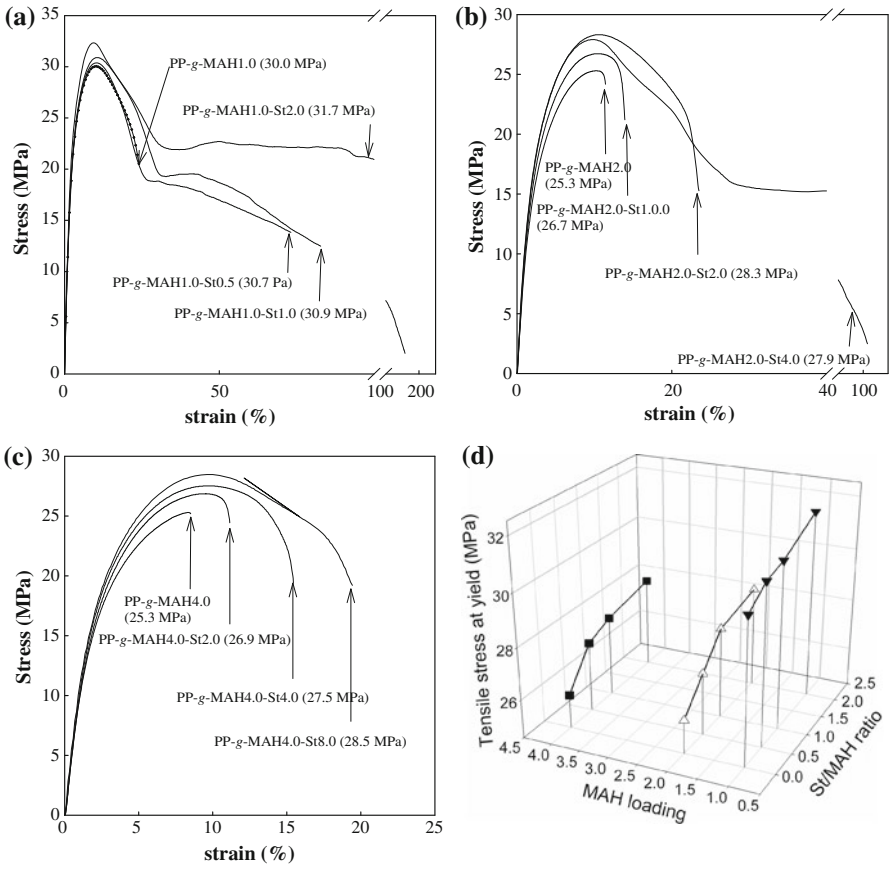

Fig. 3 Stress–strain curve of PP/clay nanocomposites compatibilized by PP-*g*-MAH and PP-*g*-MAH-St. *Numbers* shown in each curve represent the tensile stress at the yield point. **a** 1.0 phr of MAH, **b** 2.0 phr of MAH, **c** 4.0 phr of MAH, and **d** 3D plot of tensile stress at the yield point as function of MAH loading and St/MAH ratio. The standard uncertainty (standard deviation divided by tensile stress at the yield point) is less than 1.5%

miscibility between PP and PP-*g*-MAH is low upon a high grafting degree of MAH, since the high level of polar MAH groups prevents good mixing of PP-*g*-MAH with the non-polar PP, which deteriorates the toughness of the nanocomposite [27]. It is inferred that such a decrease of miscibility with higher grafting of MAH does not occur when styrene is incorporated, since the toughness increases continuously with the grafting level of MAH groups. It was reported that MAH is grafted onto the main chain of iPP in the form of a single MAH [25]. Thus, PP-*g*-MAH more closely resembles a linear copolymer having many single MAH pendants, as depicted in Fig. 6. On the contrary, PP-*g*-MAH-St is a graft copolymer having several long chains composed of MAs and styrene (SMA). Upon the same grafting degree of MAH, PP-*g*-MAH-St has a smaller number of branching points than that of PP-*g*-MAH. As a consequence, the miscibility of PP-*g*-MAH-St with PP is higher

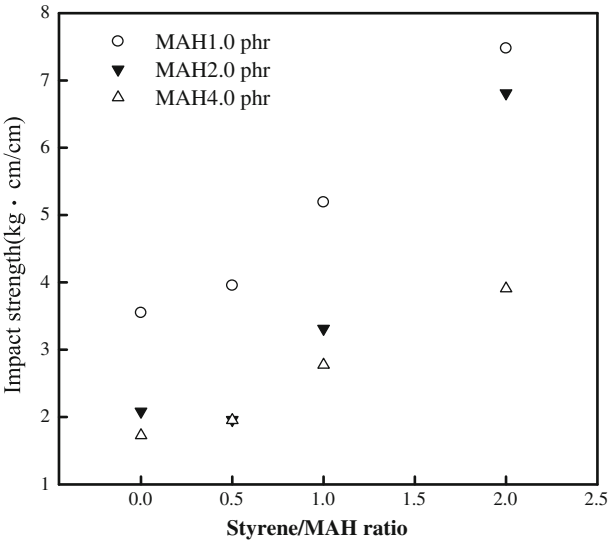

Fig. 4 Izod impact strength of PP/clay nanocomposites compatibilized by PP-*g*-MAH and PP-*g*-MAH-St

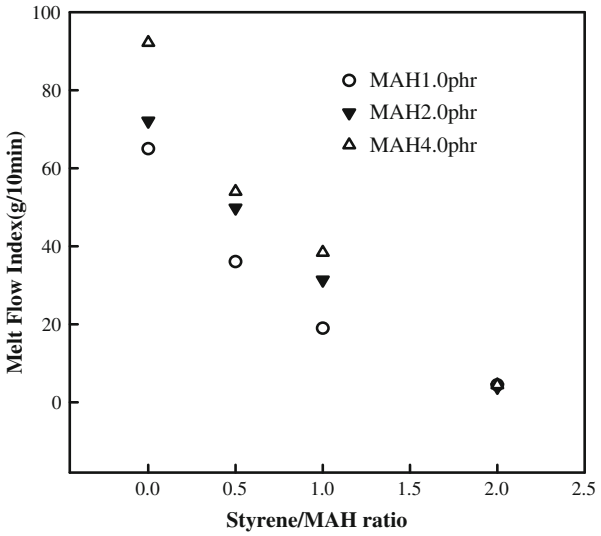

Fig. 5 Melt flow index of the PP-*g*-MAH and the PP-*g*-MAH-St investigated in this study

than that between PP-*g*-MAH and PP at the same grafting degree of MAH. Moreover, SMA branches in the PP-*g*-MAH-St can exist between the clay tactoids for intercalation of the PP-*g*-MAH-St into the clay, whereas part or all of the main chains of the PP-*g*-MAH must lie between the clay tactoids for intercalation, as depicted in Fig. 6. As a consequence, the remaining part of the PP-*g*-MAH-St located outside of the clay layers is longer and has greater likelihood of becoming

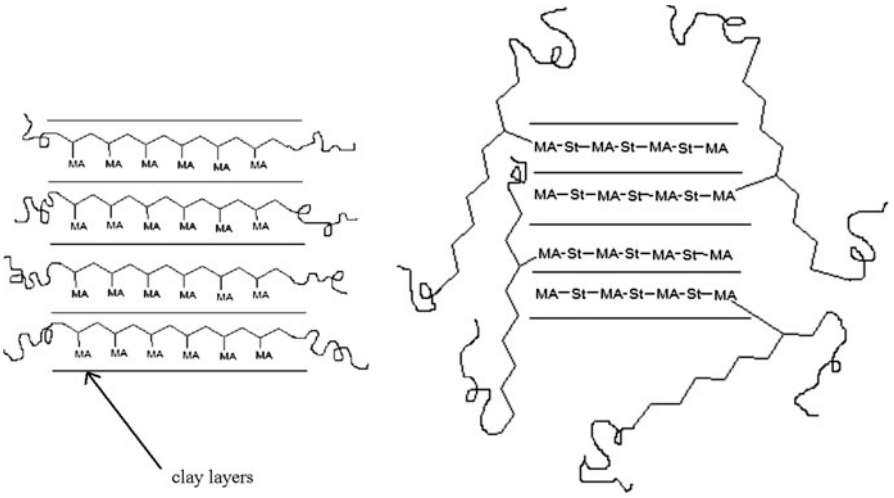

Fig. 6 Schematic representation for intercalation of PP-*g*-MAH and PP-*g*-MAH-St into clay layers

entangled with matrix polymers than does PP-*g*-MAH, leading to better physical properties.

Conclusion

We evaluated performance of PP-*g*-MAH-St as a compatibilizer in PP/clay nanocomposites. It was observed that PP-*g*-MAH-St show better compatibilizing effect than the PP-*g*-MAH. Upon higher graft degree of the MAH group, PP-*g*-MAH-St enables PP/clay nanocomposites to have the higher stiffness as well as the higher toughness, whereas the PP-*g*-MAH provides only higher stiffness but the lower toughness in the nanocomposites. It was verified that the incorporation of styrene as a comonomer in the melt grafting reaction of the MAH onto PP reduces β-scission of the PP main chain. The PP-*g*-MAH-St that is thus formed is less brittle, leading to higher toughness of the PP/clay nanocomposite.

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