Preparation of the HIPS/MA Graft Copolymer and Its Compatibilization in HIPS/PA1010 Blends

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ABSTRACT: The graft copolymer of high-impact polystyrene (HIPS) grafted with maleic anhydride (MA) (HIPS-g-MA) was prepared with melt mixing in the presence of a free-radical initiator. The grafting reaction was confirmed by infrared analyses, and the amount of MA grafted on HIPS was evaluated by a titration method. 1–5% of MA can be grafted on HIPS. HIPS-g-MA is miscible with HIPS. Its anhydride group can react with polyamide 1010 (PA1010) during melt mixing of the two components. The compatibility of HIPS-g-MA in the HIPS/PA1010 blends was evident. Evidence of reactions in the blends was confirmed in the morphology and mechanical behavior of the blends. A significant reduction in domain size was observed because of the compatibilization of HIPS-g-MA in the blends of HIPS and PA1010. The tensile mechanical properties of the prepared blends were investigated, and the fracture surfaces of the blends were examined by means of the scanning electron microscope. The improved adhesion in a 15% HIPS/75% PA1010 blend with 10% HIPS-g-MA copolymer was detected. The morphology of fibrillar ligaments formed by PA1010 connecting HIPS particles was observed. © 1999 John Wiley & Sons, Inc. J Appl Polym Sci 71: 2017–2025, 1999

Key words: PA1010; HIPS; graft; compatibilization

INTRODUCTION

Polymer alloys consisting of two or more components have been used in many advanced materials for obtaining unique physical properties that cannot be obtained from the constituent polymers alone.¹ Polymer immiscibility arises from the very small entropy gained by mixing different kinds of long chains. In fact, it will be shown that, in the limit of high molecular weight, only polymer pairs with zero or negative heats of mixing can form one phase. It should be pointed that the polymer blends with superior mechanical properties in a two-component polymer system cannot be manufactured, if the components are immisci-

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ble completely. Usually sharp interfaces and poor adhesion between the components bring about the materials failure easy. Most of the presently important systems are miscible to the extent that a slight degree of mixing takes place, or interfacial bonding is developed directly, as in grafts or blocks.²

To make polymer alloys with high performance from an immiscible polymer mixture, a compatibilizer must be used to improve interfacial adhesion. There are generally two methods of compatibilization: physical compatibilization and chemical compatibilization. The former technique uses a premade block or graft copolymer whose constituent component is compatible with each component in the blend. In this method, a block or graft copolymer can be located at the interface between two immiscible phases, since the enthalpic contribution becomes dominant over entropy loss.^{3–6}

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Many theories^{7–11} suggest that in order to reduce significantly the interfacial tension between the phases, a block copolymer having larger molecular weight is more efficient than one having smaller molecular weight. But, as the molecular weight of a block copolymer becomes larger, only part of it can go to the interface and the rest stays in the bulk phase owing to the higher possibility of micelle formation of a block copolymer.¹²⁻¹⁴ Another difficulty in physical compatibilization is that a block copolymer cannot reach the interface easily owing to its high viscosity and rather short processing times, because polymer alloys are usually prepared by extruder or compounding machine. Thus, although much work $^{3-6,15-17}$ has been published in this area, physical compatibilization is not widely used for commercial production of polymer alloys.

The chemical compatibilization technique uses the reaction between reactive groups, making in situ graft or block copolymer during melt blending by extruder or internal mixer.^{18–28} This copolymer acts as a compatibilizer between two immiscible polymer components, and it stays near the interface, since the reaction between functional groups occurs easily near the interface. Polymer alloys prepared by this technique have a very fine morphology and broader interface. Moreover, this fine morphology can persist under the very high shear stress found during the injection molding press. To obtain this kind of alloy, each polymer must have a specific reactive group, and both must react within 2-3 min if the extrusion or compounding time is considered. Reactive compatibilization, involving maleic anhydride (MA) block or grafted polyolefins, either used as a toughening agent for polyamides or as a compatibilizer for the polyamide/olefine blends, are probably the must intensively investigated subjects reported in the literature. Other than MA functional group, copolymers containing carboxylic acid, sulfonic acid, oxazoline, and epoxy functional groups have also been used.

Both polyamide 1010 (PA1010) and high-impact polystyrene (HIPS) are commodity polymers that possess unique properties individually. PA1010 has been classified as a major engineering thermoplastic with excellent properties, such as solvent resistance, easy processability, and good mechanical characteristics. HIPS is a low price, but tough thermoplastic with relatively poorer solvent resistance. A combination of PA1010 and HIPS would be an ideal pair to produce a property-balanced blend product. In practice, however, it is difficult to obtain good performance because PA1010 and HIPS are immiscible. Although many studies on the compatibility of immiscible blends have been reported, such as the system of PA6/PS,^{18–21} poly(butylene terephthalate) (PBT)/HIPS,²² polypropylene (PP)/HIPS,⁶ PA6/ABS,^{23–27} and PA6/PP,²⁸ studies on the preparation of functionalized HIPS via melt grafting of MA on to HIPS and its effects on the compatibility of PA1010 and HIPS blends have not been reported up to present.

HIPS has been selected as the excellent liner materials for the refrigerators and freezers. The market for domestic refrigerators and freezers has now reached an annual level ~ 60 million units, which consume ~ 340 kte of polyurethane foam and 400 kte of thermoplastic liner materials.

Before 1989, the polyurethane foams were blown by using freon-11, chlorofluocarbons-11 (CFC-11). It possessed many desirable properties, not the least of which was its very low thermal conductivity in the vapor state giving the polyurethane foams excellent insulation properties, and its low aggressive nature which meant that the risk of attack of liners and other materials of construction was minimal. Unfortunately, CFC-11 and CFC-12 are very potent ozone depleters for the ozone in the stratosphere, and their potential release to atmosphere thus became environmentally unacceptable. The complete phase-out of CFC's was agreed internationally. HCFC-141b, the predominant CFC-11 replacement in polyurethane and polyisocyanurate insulation foams, was selected on the basis of its environmental and physical properties.³¹ However, the HIPS liners were attacked by HCFC-141b. At an early stage of the work to develop appliance foam systems blown with HCFC-141b, it was recognized that the greater solvent power of this material compared with CFC-11 could create problems in terms of liner attack, the liner exhibited multiple crazes, soft and blistered. Studying foam/liner interactions and ultimately developing a HCFC-141b compatible liner are necessary. It was considered that the most convenient and cost-effective solution would be to improve the HCFC-141b resistance of current materials through modification of the chemical composition, morphology, and additives, whereas another would be to laminate with a barrier layer on the foam contact surface. The big advantage of the last approach would be that 90% of the existing HIPS liner plate could be used if the barrier layer material was selected successfully. Usually,

the thickness of the barrier layer is $\sim 10\%$ of the total lamina.

Nylon is an excellent barrier potential material to most chemicals and solvents. We choose the blend of Nylon 1010 and HIPS as the barrier potential materials used to manufacture the liners in refrigerators and freezers. The big advantage of Nylon 1010 is the most suitable member blended with HIPS in the nylon family, because its melting point is closed to the processing temperature of HIPS. HIPS component in the Nylon 1010/HIPS blend could improve the adhesion between the barrier lamina and the basic HIPS plate effectively.

In this article, we attempt to graft MA on HIPS through reactive processing and the effect of in situ compatibilizer of HIPS-g-MA on the final morphology and mechanical property of the immiscible polymer blends of PA1010 and HIPS. Note that the anhydride group in grafted with MA (HIPS-g-MA) reacts easily with the amine group at the chain in PA1010, and that HIPSg-MA has good compatibility with HIPS due to the presence of structurally similar HIPS units in the polymer backbone; thus, the physical and chemical interactions across the phase boundaries will control the overall performance of the polymer blends. A series of blends of HIPS/ PA1010 with excellent performance can be conducted by using the compatibilizer.

EXPERIMENTAL

Materials

HIPS used in this study was a commercial product (492-J) manufactured by Yanshan Petrochemical Co. (Beijing, China), and its melting flow index is 3.1 g/10 min. The content of polybutadiene is 7% in HIPS. PA1010 was supplied by Shijinggou Union Chemical Co. (Jilin, China). Its melting flow rate is 10 g/10 min. The chemical structure of PA1010 is shown as follows:

Dicumyl peroxide (DCP), obtained from Xizhong Chemical plant (Beijing, China) was used as an organic peroxide initiator. Its half-life is ~ 15 s at 190°C.

Maleation of HIPS

The graft reaction of HIPS with MA was conducted in the molten state by using a Brabender mixer processed at 50 rpm for 5 min at 180°C. HIPS was introduced into the mixing chamber first, then MA and DCP were added simultaneously. The product obtained in this way was dissolved in toluene, then the solution was precipitated in methanol to extract the unreacted MA and DCP from the product. The precipitate was collected and dried in vacuum for 24 h at 60°C. The polymer was dissolved in toluene again and the quantitative ethanolic KOH solution was added. The solution was violently stirred to make the anhydride group hydrolyze completely. The MA content was determined by back-titration with an isopropyl alcoholic HCl solution using phenolphthalein as an indicator. The grafting degree (GD) of MA was calculated from the following equation:

$$GD = rac{\left[V_{KOH} C_{KOH} - V_{HCL} C_{HCL}
ight] imes M_{MA}}{2 imes W_S} imes 100\%$$

where V, C, M, and W_S are the volume, concentration, molecular weight, and the weight of the sample, respectively.

Spectroscopic Analysis

The presence of MA grafted onto HIPS was assessed using a FTS-7 Fourier transform infrared (FTIR; Bio-Rad, Richmond, CA) spectrometer and an ESCA CAB MK-II (VG Co., England) electron spectrometer. The thin films, made from solution casting, were used for infrared measurements.

Blend Preparation

PA1010 was dried for 24 h at 90°C before melt blending. The polymers were melt-mixed using a Brabender twin-screw extruder operated at a rotation speed of 20 rpm and a temperature range of 210° -230°C.

The weight ratio of PA1010 to HIPS blends was fixed at 75/25. The amount of HIPS-g-MA in the blends (the GD of which is 4.7%) was 0, 5, and 10 wt % of the total blend weight.

Morphology Observation

Morphology of the blends was observed with a scanning electron microscope (SEM; JXA-840) at an accelerating voltage of 25 kV. Blend samples



Figure 1 FTIR spectra of (a) HIPS and (b) HIPS-g-MA.

were fractured at liquid nitrogen temperature, and the fractured surface was coated with gold.

Specimens for transmission electron microscopy (TEM) observation were obtained by using a cryoultramicrotomy technique at liquid nitrogen temperature. Ultrathin sections were stained with osmium tetroxide to enhance the contrast for the microscopy observations. A Jeol 1200EX TEM operated at 200 kV was used to examine the morphology of these blends.

Tensile Property Measurement

Dumbbell-shaped specimens were prepared at 230°C with hot press molding. Tensile tests were conducted on an Instron 1121 machine at room temperature with a crosshead speed of 5 mm min⁻¹. Seven specimens of each blend were tested, and average values were taken as experimental data.

RESULTS AND DISCUSSION

Characterization of HIPS-g-MA

FTIR spectra of the HIPS and HIPS-g-MA are shown in Figure 1. In the spectrum of HIPS-g-MA

[Figure 1(b)], the new absorption bands at 1218, 1780, and 1857 cm⁻¹ appeared. However, those bands were not observed in the spectrum of HIPS. These characteristic bands are assigned to a C—O single bond (1218 cm⁻¹) and a C=O double bond (1789 and 1857 cm⁻¹) stretching in MA units that are introduced on the molecular chains of HIPS via the reactive extrusion process, because the unreacted MA has been removed from the sample. The most reliable evidence of the grafting reaction between HIPS and MA was conducted by the titration measurement. The grafting degree of MA on the HIPS is 4.7 wt %. It means that the content of the oxygen element in the graft copolymer of HIPS and MA is 2.3%.

Grafting was also confirmed using X-ray photoelectron spectroscopy (XPS) spectra. Figure 2 shows the XPS spectra of HIPS and HIPS-g-MA. The binding energy of $O_{\rm ls}$ is ~ 532.8 ev. The intensity of the oxygen peak of HIPS-g-MA is significantly larger than that of the HIPS. The ratio of the intensity between HIPS-g-MA and HIPS is ~ 2.2 : 1. This can be tentatively explained as follows: the weak $O_{\rm ls}$ peak of the HIPS is very difficult to identify, and it originates from the absorbed oxygen from the air on the surface of the HIPS sample. On the other hand, the larger peak of $O_{\rm ls}$ related to the oxygen atom of the HIPS-g-MA graft copolymer was observed obviously on the XPS spectrum.

Morphology

The electron photomicrographs of the fracture surface of blends are shown in Figure 3. The SEM



Figure 2 XPS spectra of (a) HIPS and (b) HIPS-g-MA.



PA1010/HIPS/HIPS-g-MA = 75/25/0



PA1010/HIPS/HIPS-g-MA = 75/20/5



PA1010/HIPS/HIPS-g-MA = 75/15/10

Figure 3 SEM photomicrographs of the PA1010/HIPS/HIPS-g-MA blends (×3000).

photomicrograph is the most convenient approach to differentiate the morphologies between a compatibilized blend and an uncompatibilized counterpart. An immiscible and incompatible blend usually results in coarser morphology than the compatibilized one, and the finer phase domains are an indication of better compatibilization of the blend. The presence of a compatibilizer, usually distributed at interface, is able to reduce the interfacial tension and breakup the phase easier in a typical melt blending process, and the resultant separated phases are therefore more stable and have less tendency to recombine. In the SEM 75PA1010/25HIPS photomicrographs of the blends, the white spheres are HIPS particles and the dark holes are the HIPS phase that was removed from PA1010 matrix. HIPS domains, which diameter ranged between 0.8 and 8 μ m, have a well-defined spherical shape and a very broad size distribution. The large particle size, with no evidence of adhesion between the matrix and dispersed phase, confirmed the immiscibility of the two components. Also, surfaces of the domains appear to be very smooth. This is a typical morphology of an immiscible blend. Some different features are observed for the ternary blends of PA1010/HIPS/HIPS-g-MA. The presence of HIPS-

g-MA significantly reduced the domain size of the dispersed phase and its distribution. Interfacial adhesion seemed to be improved with forming *in situ* the compatibilizer during melt mixing. Rough fracture surfaces in compatibilized blends were observed because the HIPS particles had adhered to the matrix material.

Interfacial tension, the shear rate of mixing and the viscosity ratio of the blending components are key parameters governing the degree of dispersion.²⁹ When the shear rate of mixing and the viscosity ratio of the blended polymers are constant, the interfacial tension of the blend plays an important role in reducing the domain size. Therefore, better dispersion and the improved interfacial adhesion should be attributed to formation of the grafting copolymer at the interface between PA1010 and HIPS via reaction of MA in HIPS-g-MA with the terminal amine groups of PA1010 during melt extrusion.

However, the SEM technique does not make it possible to distinguish the finer morphological features, in particular, the character of HIPS dispersion in blends with 5 and 10 wt % of the graft copolymer, HIPS-g-MA. Majumdar and colleagues²⁴ had examined the morphologies of Nylon6/ABS blend compatibilized with SMA25 by



Figure 4 TEM photomicrographs of the HIPS material. The sample was stained with O_sO_4 .

TEM using several different staining techniques. To examine the morphology features of compatibilized blends of HIPS/PA1010, the ultrathin sections of the blends were studied by using TEM. Figure 4 shows the TEM photomicrograph for the HIPS sample, 492-J, used in this work. The complex cellular structure was observed. The domain size has a broad distribution. Now, the morphologies of PA1010/HIPS blends compatibilized with HIPS-g-MA are examined by TEM. Figure 5 shows photomicrographs of the series of blends containing 75% PA1010 with varying amounts of HIPS-g-MA. For the blends without any HIPSg-MA compatibilizer, the cellular structure rubber particles of the HIPS exit in certain regions where the biggest diameter is $\sim 8 \,\mu m$. Comparing this with the SEM photomicrograph, we can confirm that the regions are HIPS phases. Cellular structure rubber particles of the HIPS are entirely absent from other regions, which must consist primarily of PA1010 [Fig. 5(a)]. For the uncompatibilized blends, the sharp boundaries and clear gaps between the HIPS dispersed phase and the PA1010 matrix were observed. The biggest domain size is $\sim 8 \ \mu m$ in diameter. In these domains, the continuous phase consists of polystyrene, whereas cellular structure shown in HIPS was observed in 75PA1010/25HIPS blend as the secondary dispersed phase. The domain

size and its distribution are similar to that in HIPS. These results show that the cellular structure features and the morphological characteristic in HIPS are not broken, and they are kept in the PA1010/HIPS blends totally. The worse miscibility between PA1010 and HIPS can be conducted from these observations. O_SO₄ staining techniques reveal the features and the distribution of polybutadiene chains in the domains. As is well known, polybutadiene consists of the matrix in cellular structure. They should be assigned as third-phase morphology in PA1010/HIPS blends. For compatibilized blends, morphologies shown in Figure 5(b,c) reveals that the sharp boundaries and clear gaps between the dispersed phase and the matrix have disappeared. Due to the interfacial reaction between PA1010 and HIPS-g-MA, the interfacial situation between PA1010 and HIPS is absolutely changed. Namely, the interface between PA1010 and HIPS is diffused and no clear layer of PS, as could be seen in the blend of PA1010/HIPS, can be identified. The site of the





(a) PA1010/HIPS/HIPS-g-MA=75/25/0

(b) PA1010/HIPS/HIPS-g-MA=75/20/5



(c) PA1010/HIPS/HIPS-g-MA=75/15/10

Figure 5 TEM photomicrographs for 75% PA1010 blends containing (a) 0%, (b) 5%, and (c) 10% HIPSg-MA (\times 6000). The samples were stained with O_sO₄.

reaction on HIPS is the polybutadiene segment; hence, the PB chains should face toward the PA1010 matrix through the reaction. It means that the molecular architecture will affect the morphology of the blends and that the compatibilizer located at the interface broadens the interfacial region and that the molecular chains of the HIPS-MA-PA1010 copolymer have penetrated into the adjacent phases of PA1010 and HIPS. In fact, the secondary dispersed phase, identified clear in the uncompatibilized PA1010/HIPS blend, cannot be observed. However, third-phase features are still maintained. After we examine carefully the morphological features of the third dispersed phase, different characteristics among the compatibilized and uncompatibilized samples were confirmed. First, the regular spherical cellular structure shown in the uncompatibilized blends was detracted in compatibilized blends. Second, domains with cellular structure were distributed in the whole TEM images for compatibilized blends; on the contrary, they are concentrated in the PS domains in the uncompatibilized blend (i.e., a relatively high number of the third order of domains are visible in some regions, whereas almost no domains are seen in other regions).

As is well known, the graft copolymer of styrene and butadiene formed during the polymerization of HIPS located preferably on the interfaces between PS matrix and the domains with cellular structures, and it is an effective compatibilizer for PS and polybutadiene in HIPS. However, the double bonds on molecular chains of PS-g-polybutadiene (PB) will react preferably with MA to form the HIPS-g-MA copolymers, because the soluble bonds have higher reactive activity during the grafting copolymerization initiated by free radicals. Then, they are transformed into new grafting copolymers, maleated HIPS-g-PA1010 that will migrate to the interfaces between HIPS and PA1010 from the interfaces between PS and cellular domains to accompany the grafting reaction between HIPS-g-MA and PA1010. Thus, regular spherical domains were shown in an uncompatibilized blend of HIPS and PA1010, whereas the irregular ones were created in the compatibilized blends of PA1010/HIPS-g-MA. On the other hand, the reactive activity of double bonds of PB in HIPS inside the domains with the cellular structure was reduced because the slight crosslinks of their molecular chains will restrain the grafting reaction between PB and MA.

PA1010/HIPS/ HIPS-g-MA	Strength (MP)	Energy (J)	Elongation (%)
100/0/0	44	8.1	279
0/100/0	28	0.2	11
75/25/0	41	0.4	15
75/20/5	43	0.9	31
75/15/10	39	6.7	247

Tensile Properties

Interfacial adhesion is an important parameter for controlling the mechanical properties of multiphase systems: good adhesion between the matrix and a dispersed phase may be essential for proper stress transfer without interfacial debonding. From the morphological observation, we realized that the PA1010-g-HIPS copolymer, which is formed during melt mixing, acts as a compatibilizer of the PA1010/HIPS blends. The mechanical properties of binary and ternary blends are summarized in Table I; an increase in the tensile properties is observed when HIPS-g-MAs is introduced into blends of PA1010/HIPS. The low tensile properties of the PA1010/HIPS blend can be related essentially to the larger size of the HIPS domains with poor adhesion to the matrix. These domains act as gross material defects, causing premature rupture of the specimen soon after the beginning of the yield. On the other hand, in PA1010/HIPS/HIPS-g-MA blends, all particle size reduction and an improvement of interfacial adhesion, caused by the formation of PA1010-g-HIPS copolymer at the interface between PA1010 and HIPS seem to lead to the improvement of mechanical properties.

Because the tensile yield strength of the HIPS material is lower than that of the PA1010 matrix, an indication of the interfacial strength is derived from the extent to which the HIPS particles are deformed when the blend fractures. SEM images of the fracture surface are given in Figure 6 for compatibilized and uncompatibilized blends of PA1010/HIPS. The distinct phase-separated domains, having a size of $0.5-8 \mu m$, are clearly seen for the uncompatibilized blends, whereas there are hardly any signs of a phase-separated morphology in the compatibilized blends of PA1010/ HIPS/HIPS-g-PA1010 after a tensile test. Some other changes in the fracture surfaces were observed on the SEM photomicrographs as the amount of compatibilizer increases. On the frac-



(a) PA1010/HIPS/HIPS-g-MA=75/25/0



(b) PA1010/HIPS/HIPS-g-MA=75/20/5



(c) PA1010/HIPS/HIPS-g-MA=75/15/10

Figure 6 SEM photomicrographs of the tensile fracture surfaces containing (a) 0%, (b) 5%, and (c) 10% HIPS-g-MA (\times 1000).

ture of the PA1010/HIPS blends, undeformed HIPS particles with smooth surfaces debonded from the surrounding PA, which was pulled out and fabricated. This indicates that the interfacial adhesion is very poor between PA1010 and HIPS. However, in PA1010/HIPS/HIPS-g-MA blends, numerous thin ligaments connected HIPS particles to the deformed matrix and incorporated the particles into the fibrous PA1010 area. This evidence that adhesion between HIPS and PA1010 was good enough to prevent debonding during the drawing indicated that interfacial matrix strength was higher than the fracture strength of the matrix. The ribbonlike features on the drawn ligaments were the result of recoil after they fractured. The better adhesion provided by 10% HIPS-g-MA was manifest primarily in the ultimate elongation. The tensile energy and elongation at the break of the PA1010/HIPS/HIPS-g-MA blends were improved >10 times with adding HIPS-g-MA from 0 to 10 wt %. It was presumed that the interfacial strength in the blends was determined primarily by interaction of the compatibilizer with PA, because adhesion to HIPS was provided by chemical linkages. The interaction of HIPS-g-MA with PA1010 was strong enough to sustain particle-matrix adhesion because the compatibilizer was drawn along with the PA. Interfacial agents generated in situ during the process of melt mixing through reaction between chemical functionalities available in the polymer chains have been reported in the literature. Both experimental observations and theoretical prediction indicate a reduction in the dispersed phase domain size. In addition, the presence of the compatibilizer at the interface broadens the interfacial region through penetration of the copolymer chains into the adjacent phase.³⁰ These factors described herein translated on the macroscale into higher fracture elongation for the blends.

CONCLUSIONS

HIPS was grafted with MA in a mixer. The results of FTIR and XPS confirmed that MA units are incorporated in the HIPS. The introduction of MA units in HIPS was very effective for controlling the morphology of blends of PA1010/HIPS/ HIPS-g-MA. The domain size of the dispersed phase of ternary blends decreased with increasing HIPS-g-MA content in blends, whereas PA1010/ HIPS binary blends showed typical morphology of an incompatible blend.

The effects of the HIPS-g-MA concentration on the tensile properties of blends have been examined. The improved mechanical properties should be attributed to the interaction between HIPSg-MA and PA1010. For the tensile fracture of the compatibilized blends, no HIPS particles are visible on the fracture surface. This behavior indicates that the HIPS particles are firmly attached to the PA1010 matrix; otherwise, the HIPS particles would have been dislodged from the matrix during the tensile fracture. With increasing HIPS-g-MA content from 0 to 10%, the microscale deformation mode progressed from debonding to particle drawing and fibril fracture. Interfacial adhesion was postulated to increasing by increasing the HIPS-g-MA content in the blend.

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