[5] US Patent No. 7,622,529 relates to olefin interpolymers as compatiblizers, which are prepared by polymerizing one or more monomers or mixtures of monomers, such as ethylene and one or more comonomers, to form an interpolymer product having unique physical properties. The olefin interpolymers contain two or more differing regions or segments (blocks), resulting in unique processing and physical properties.

[6] WO/2011/041696 relates to block composites and their use as impact modifiers; WO/2011/041698 relates to block composites in soft compounds; and WO/2011/041699 relates to block composites in thermoplastic vulcanisates.

SUMMARY

[7] ~~In a first aspect, t~~The disclosure provides a composition comprising a functionalized olefin-based polymer formed from at least (A) and (B):

(A) a block composite comprising:
  i) a block copolymer comprising a crystalline propylene-based block and an ethylene/α-olefin block;
  ii) a crystalline propylene-based polymer; and,
  iii) an ethylene/α-olefin polymer; and
(B) at least one functionalization agent.

~~In a second aspect, t~~The disclosure also provides a composition comprising a functionalized olefin-based polymer formed from at least (A) and (B):

(A) a crystalline block composite comprising:
  i) a block copolymer comprising a crystalline propylene-based block and an crystalline ethylene-based block;
  ii) a crystalline propylene-based polymer; and,
  iii) an crystalline ethylene-based polymer; and
(B) at least one functionalization agent.

[7a] In one aspect, there is provided a composition comprising a functionalized olefin-based polymer formed from at least (A) and (B):

(A) a block composite comprising:
  a) a block copolymer comprising a crystalline propylene-based block and an ethylene/α-olefin block;
  ii) a crystalline propylene-based polymer; and,
  iii) an ethylene/α-olefin polymer; and
(B) at least one functionalization agent selected from the group consisting of silane, maleic anhydride and amine, wherein the weight percent of polypropylene from HTLC separation

before functionalization and the weight percent of polypropylene from HTLC separation after functionalization comprises a difference not exceeding 20 weight percent.

BRIEF DESCRIPTION OF THE DRAWINGS

Figure 1 shows the DSC profile for CBC2.

Figure 2 shows the TREF analysis of CBC2

Figure 3 shows HTLC analysis of CBC2.

Figure 4 shows the IR spectrum for MAH-g-CBC2-4.

Figure 5 shows the DSC profile for MAH-g-CBC2-4.

Figure 6 shows HTLC analysis of MAH-g-CBC2-4.

Figure 7 shows TEM for CBC5, MAH-g-CBC2 (Ex. 5) and MAH-g-(EP1/iPP1 50/50 Blend) (Ex. C).

atmosphere. The films of 3 mil thickness are cooled by transferring the plates to the lower level platens set at ambient temperature. IR spectra are collected using Nicolet 6700 FTIR. FTIR spectra were used for determining the level of g-MAH in each sample using a method that has been calibrated against the tetrabutylammonium Hydroxide (TBAOH) titration. The wt% of g-MAH was determined from the ratio of the height of the peak at ca. 1790 cm^{-1} corresponding to the carbonyl stretch of the anhydride, to the height of the 2751 cm^{-1}, as follows

$$\text{wt\% MAH} = 0.0344 \times \frac{(\text{peak height @ ca. } 1790\text{cm}^{-1})}{(\text{peak height @ ca. } 2751\text{cm}^{-1})} + 0.1925 \times \frac{(\text{peak height @ ca. } 1790\text{cm}^{-1})}{(\text{peak height @ ca. } 2751\text{cm}^{-1})} + 0.0767$$

[81] As for the TBAOH titration, 1 - 2 g of the dried resin was dissolved in 150 ml xylene by heating the sample to 100 deg. C on a stirred hot plate. Upon dissolution, the sample was titrated, while hot, with 0.025N TBAOH in 1:1 toluene/methanol using 10 drops of Bromothymol blue as indicator. The endpoint is recorded when solution turns blue.

Silane grafting level analysis

[82] Duplicate samples were prepared by transferring approximately 3.0 grams of the pellets into pre-cleaned 2-dram polyethylene vials. Samples are vacuum stripped at 140 °C for 20 minutes in a vacuum oven to remove any residual, volatile, or surface silane. Duplicate Si standards are prepared from their NIST traceable standard solution into similar vials. The standards are diluted to a similar volume as the samples using pure water. A blank sample of the water is also prepared. The samples, standards and a blank are then analyzed for Si. Specifically, irradiation is done for 3 minutes at 250kW reactor power. The waiting time is 9 minutes and the counting time was 270 seconds using an HPGe detector set. The Si concentrations are calculated in ppm using Canberra software and comparative technique. Typical uncertainty ranges from 2% to 5% relative and the detection limit is less than 100ppm. The vinyltrimethoxysilane content is back calculated using stoichiometry, assuming that the grafting is done using vinyltrimethoxysilane only.

Gel fraction analysis

[83] Gel fractions of crosslinked silane grafted samples are measured using a Soxhlet extractor and xylene as solvent. Crosslinked samples are extracted under reflux for a minimum of 19 hours. The extracted samples are dried in a vacuum oven at 90°C for 3 hours. The samples are crosslinked at 85% relative humidity and 85°C for 3 weeks on a 4 mil compression molded film. Triple specimens are tested and the average was reported.

Tensile properties

[84] For tensile property measurements, samples are compression molded into 70 mil thick plaque (5 inch X 5 inch (12.7 cm x 12.7 cm)) with a Carver hydraulic press at 190°C for 6 min at 6000 lb (2721.6 kg) at ambient

atmosphere. The plaques are then cooled to 50°C at 15°C/min in the press under 30000 lb force (133.4 N). Stress-strain behavior in uniaxial tension is measured using ASTM D1708 microtensile specimens. Specimens are die-cut from the plaques in conformation of the dimensions specified in ASTM D1708. The gauge length of samples is 22 mm and samples are stretched with an Instron at 554% of (initial gauge length) min^{-1} at 23°C. Tensile properties are reported from an average of 5 specimens.

Transmission Electron Microscopy

[85] The compression molded plaques used for microtensile test are examined by TEM. The samples are trimmed so that sections could be collected near the core of thickness of the sample. The trimmed samples are cryopolished at -60°C to prevent smearing using a diamond knife on a Leica UCT microtome prior to staining. The cryo-polished blocks are stained with the vapor phase of a 2% aqueous ruthenium tetraoxide solution for 3hrs at ambient temperature. The staining solution is prepared by weighing 0.2gm of ruthenium (III) chloride hydrate (RuCl3 x H2O) into a glass bottle with a screw lid and adding 10ml of 5.25% aqueous sodium hypochlorite to the jar. The samples are attached to a glass slide using double sided tape. The slide is placed in the jar in order to suspend the blocks about 1 inch above the staining solution for 3 hr. Sections of approximately 100 nanometers in thickness are collected from the stained sample at ambient temperature using a diamond knife on a Leica EM UC6 microtome and placed on 600 mesh virgin TEM grids for observation. TEM images are collected using a JEOL JEM-1230 operated at 100kV accelerating voltage and photographed using a Gatan-791 and 794 digital cameras.

^{13}C NMR spectroscopy method for ^{13}C-labeled maleic anhydride grafted polyolefin

[86] The sample was prepared by adding approximately 2.7g of TCE-d_2 with 0.025M chromium acetylacetonate (relaxation agent) to 0.2 g sample in a 10mm NMR tube, and then purging in a N_2 box for 2 h. The sample was dissolved and homogenized by heating the tube and its contents at 140-150°C.

[87] For ^{13}C NMR, the data were collected using a Bruker 400 MHz spectrometer equipped with a Bruker Dual DUL high-temperature CryoProbe. This method is described in, for example, Z. Zhou, R. Kuemmerle, J. C. Stevens, D. Redwine, Y. He, X. Qiu, R. Cong, J. Klosin, N. Montanez, G. Roof, Journal of Magnetic Resonance, 2009, 200, 328-333 and Z. Zhou, J. C. Stevens, J. Klosin, R. Kümmerle, X. Qiu, D. Redwine, R. Cong, A. Taha, J. Mason, B. Winniford, P. Chauvel and N. Montañez, Macromolecules, 2009, 42, 2291-2294.

[88] The data were acquired using 320 transients per data file, a 7.3 sec pulse repetition delay (6 sec delay + 1.3 sec acq. time), 90 degree flip angles, and a modified inverse gated decoupling with a sample temperature of 120°C. All measurements were made on non spinning samples in locked mode.

Table 2. Reactor process conditions to produce crystalline block composites CBC1-CBC6

Material	CBC1		CBC2		CBC3		CBC4		CBC5		CBC6	
Reactor	1st Reactor	2nd Reactor	1st Reactor	2nd Reactor	1st Reactor	2nd Reactor	1st Reactor	2nd Reactor	1st Reactor	2nd Reactor	1st Reactor	2nd Reactor
Reactor Control Temp.(°C)	130	115	118	110	120	115	96	116	141	135	140	115
Solvent Feed (lb/hr)/(kg/hr)	228 (103.4)	185 (83.9)	145 (65.8)	145 (65.8)	130 (59)	292 (132.4)	167 (75.7)	253 (114.8)	242 (109.8)	245 (111.1)	144 (65.3)	360 (163.3)
Propylene Feed (lb/hr)/(kg/hr)	4.34 (1.97)	34.88 (15.8)	1.98 (0.898)	31.03 (14.07)	2.92 (1.32)	58.66 (26.6)	3.48 (1.58)	50.99 (23.1)	5.44 (2.47)	48.76 (22.1)	3.42 (1.55)	69.17 (31.4)
Ethylene Feed (lb/hr)/(kg/hr)	32.9 (14.9)	0.0 (0)	27.8 (12.6)	0.0 (0)	24.5 (11.1)	0.0 (0)	28.2 (12.8)	0.0 (0)	47.0 (21.3)	0.0 (0)	28.0 (12.7)	0.0 (0)
Hydrogen Feed (SCCM)	9.7	9.3	9.6	9.2	9.7	9.8	9.7	9.6	9.5	0.0	9.6	9.6
Reactor Propylene Conc. (g/L)	1.64	2.27	1.06	2.00	1.98	1.93	2.20	2.67	3.57	2.26	1.99	2.03
Catalyst Efficiency (gPoly/gM) *1.0E6	0.877	0.238	5.166	0.167	0.738	0.081	2.434	0.028	0.706	0.075	0.581	0.146
Catalyst Flow (lb/hr)/(kg/hr)	0.68 (0.308)	0.38 (0.172)	1.16 (0.526)	0.95 (0.431)	0.89 (0.403)	1.79 (0.812)	0.42 (0.191)	3.00 (1.36)	0.47 (0.213)	1.78 (0.807)	0.36 (0.163)	0.93 (0.421)
Catalyst Conc. (ppm)	60	385	5	196	40	400	30	600	150	500	150	500
Cocatalyst-1 Flow (lb/hr)/(kg/hr)	0.91 (0.413)	0.20 (0.09)	1.48 (0.671)	0.93 (0.421)	0.80 (0.362)	0.96 (0.435)	0.57 (0.259)	2.08 (0.943)	1.41 (0.64)	1.12 (0.508)	0.36 (0.163)	0.58 (0.263)
Cocatalyst-1 Conc. (ppm)	600	6000	50	2000	600	6000	300	7800	500	8000	2000	8000
Cocat.-2 Flow (lb/hr)/(kg/hr)	0.57 (0.259)	0.00 (0)	0.91 (0.412)	0.00 (0)	0.51 (0.231)	0.00 (0)	0.60 (0.272)	0.00 (0)	1.18 (0.535)	9.98 (4.53)	0.75 (0.34)	15.02 (6.81)
Cocat.-2 Conc. (ppm)	3995	0	1494	0	3995	0	1993	0	1993	1993	1993	1797
DEZ Flow (lb/hr)/(kg/hr)	1.24 (0.56)	0.00 (0)	1.10 (0.499)	0.00 (0)	1.65 (0.748)	0.00 (0)	0.97 (0.44)	0.00 (0)	1.89 (0.857)	0.00 (0)	1.20 (0.544)	0.00 (0)
DEZ Conc. (ppm)	50000	0	30000	0	30000	0	30000	0	30000	0	30000	0
Production Rate (lb/hr)/(kg/hr)	35.6 (16.1)	35.0 (15.88)	29.2 (13.24)	31.1 (14.1)	26.3 (11.93)	58.4 (26.5)	30.9 (14.02)	50.4 (22.9)	49.5 (22.5)	56.1 (25.4)	31.0 (14.1)	68.1 (30.89)

Synthesis of Block Composites

[131] The Block Composite samples are synthesized in dual reactors in series in a similar fashion to the Crystaline Block Composite samples. The first reactor in each case was approximately 12 gallons (45.4 litre) in volume while the second reactor was approximately 26 gallons (98.4 litre). The process conditions are contained in Table 5.

Table 5. Reactor process conditions to produce block composites

Material	BC1		BC2	
Reactor	1st reactor	2nd reactor	1st reactor	2nd reactor
Reactor Control Temp. (°C)	95	105	96	109
Solvent Feed (lb/hr)/(kg/hr)	145 (65.78)	145 (65.78)	145 (65.78)	145 (65.78)
Propylene Feed (lb/hr)/(kg/hr)	11 (4.99)	30 (13.61)	19 (8.62)	29 (13.15)
Ethylene Feed (lb/hr)/(kg/hr)	20 (9.07)	0 (0)	12 (5.44)	0 (0)
Reactor Propylene Conc. (g/L)	1.72	2.08	2.14	2.08
Hydrogen Feed (SCCM)	15	19	10	11
Catalyst Efficiency (gPoly/gM)*106	4.55	0.20	5.42	0.15
Catalyst Flow (lb/hr)/(kg/hr)	0.15 (0.068)	0.74 (0.336)	0.38 (0.172)	1.02 (0.462)
Catalyst Conc. (ppm)	45	196	15	196
Cocatalyst-1 Flow (lb/hr)/(kg/hr)	0.3	0.73	0.38	1.99
Cocatalyst-1 Conc. (ppm)	300	2000	199	1000
Cocat.-2 Flow (lb/hr)/(kg/hr)	1.01	0	0.61	0
Cocat.-2 Conc. (ppm)	1494	0	1494	0
DEZ Flow (lb/hr)/(kg/hr)	0.81 (0.367)	0 (0)	0.72 (0.327)	0 (0)
DEZ Concentration (ppm)	30000	0	30000	0
Production Rate (lb/hr)/(kg/hr)	30 (13.61)	30 (13.61)	31 (14.06)	29 (13.15)

Table 6 –Block composite physical properties

Example	MFR (230°C/2.16 kg)	Wt% PP from HTLC Separation	Mw Kg/mol	Mw/ Mn	Total Wt% C_2	Tm (°C) Peak 1 (Peak 2)	Tc (°C)	Melt Enthalpy (J/g)
BC1	4.1	21.5	133	2.35	33.8	139 (40)	93	66
BC2	6.0	21.6	176	3.69	20.5	138 (na)	96	56

Table 7 Block Composite Index Estimations

Sample	Wt% iPP	Wt% EP	Wt% C2 in EP	(Crystalline) Block Composite Index
BC1	49	51	65	0.413
BC2	50	50	40	0.368

Synthesis of EP1 and iPP1

[132] Samples EP1 and iPP1 were produced using the same catalyst and cocatalysts as the Crystaline Block Composites and Block Composites but each were produced in a single reactor. For sample EP1 the final product MFR was regulated by manipulating the DEZ flow. Sample iPP1 was produced at higher propylene concentration in the reactor and hydrogen was used to regulate the final product MFR rather than DEZ. Sample EP1 was produced in a 12 gallon reactor and iPP1 was produced in a 52 gallon (196.8 litre) reactor.

Table 8. Reactor process conditions to produce EP1 and iPP1

Material	EP1	iPP1
Reactor Control Temp. (°C)	146	155
Solvent Feed (lb/hr)/(kg/hr)	290 (131.54)	1208.3 (548.08)
Propylene Feed (lb/hr)/(kg/hr)	7.4 (3.36)	322.2 (146.1)
Ethylene Feed (lb/hr)/(kg/hr)	57.8 (26.2)	2.5 (1.13)
Hydrogen Feed (SCCM)	0	171
Reactor Propylene Conc. (g/L)	4.13	46.56
Catalyst Efficiency (gPoly/gM)*106	1.12	0.794
Catalyst Flow (lb/hr)/(kg/hr)	0.85 (0.386)	2.45 (1.11)
Catalyst Conc. (ppm)/(kg/hr)	64.9 (29.4)	120 (54.4)
Cocatalyst-1 Flow (lb/hr)/(kg/hr)	0.55 (0.249)	1.55 (0.704)
Cocatalyst-1 Conc. (ppm)	1000	1397
Cocat.-2 Flow (lb/hr)/(kg/hr)	1.40 (0.635)	1.11 (0.503)
Cocat.-2 Conc. (ppm)	3995	1397
DEZ Flow (lb Zn/hr)/(kg/hr)	1.68 (0.762)	0 (0)
DEZ Concentration (ppm)	40,000	0
Production Rate (lb/hr)/(kg/hr)	63.4 (28.76)	233 (105.69)

Table 9 –Single reactor product physical properties

Example	MFR (230°C/2.16 kg)	Mw Kg/mol	Mw/ Mn	Total Wt% C_2	Tm (°C)	Tc (°C)	Melt Enthalpy (J/g)
EP1	6.2	70.6	2.06	92	108	92	115
iPP1	7.7	248	2.64	0.9	131	99	75

Preparation of MAH grafted OBC1 (MAH-g-OBC1)

[133] A ZSK-92 Megacompounder with 11 barrels (45 L/D) is used to graft MAH on INFUSE™ 9807 (OBC1). The OBC1resin is fed with K-Tron T-60 feeder. MAH is injected at barrel 3, port 1 using a Lewa pump. The peroxide/oil mixture (50/50 wt/wt) was injected at barrel 3, port 2 using a Prominent Sigma plunger pump. The barrel temperature is set at 80°C for zone 1 and 225°C for zone 2 to 11. A vacuum pump system is used for devolatilization at barrel 9. A minimum of 20 inches Hg (67.7 KPa) is used. The screw RPM is at 200 to 230, the torque ranges from 56% to 61%. The feed rate for OBC1 is set at 1500 lb/hr (680.4 kg/hr). The feed formulation is 1.70% MAH, 0.20% peroxide/mineral oil (50/50, wt/wt) mixture. The final MAH grafting level is 0.95% and melt index of MAH-g-OBC1 is 8.0 (2.16kg, 190°C).

Preparation of MAH grafted polymers (MAH grafted CBC, EP1, iPP1 and blends of EP1 and iPP1)

[134] A Coperion ZSK-25MC twin screw extruder is used for the reactive extrusion process to functionalize the base plastic resins. The 25-mm diameter extruder was equipped with 12 barrel segments for a total length to diameter ratio of 48. There are 11 independently controlled barrel sections with electric heating and water cooling. Polymer pellets are fed to the main extruder hopper in barrel 1 using a K-tron Model KCLQX3 loss-in-weight feeder. The liquid reactants were injected into the third barrel section of the extruder using a HPLC precision metering pump (Alltech Model 627 HPLC pump). A dialkyl organic peroxide (Luperox 101, CAS# 78-63-7) is used as the initiator. The liquid feed to the extruder included a solvent blend containing both the maleic anhydride monomer and the organic peroxide. In order to improve the accuracy of the feeding, both the peroxide and maleic anhydride powder were dissolved in methylethylketone (MEK) solvent. 300 grams of maleic anhydride crystals were weighted out and placed into a 2-liter plastic container with a sealable lid. An equal weight of MEK solvent was weighted out on a scale, and added to the container with the maleic anhydride. The blend was left in the laboratory hood with a magnetic stir bar to assist in dissolving the maleic anhydride into the solvent. The ratio of maleic anhydride to peroxide was held constant for the experimental samples at 0.02 parts peroxide and 0.9 parts maleic anhydride. In order to minimize the time when the peroxide and monomer are mixed together, the peroxide were dissolved in the previously made MEK/maleic anhydride solution immediately before moving the blend to the inlet of the positive displacement pump. The HPLC

injection rate was 9.07 g/min. Typical injection pressures for the liquids were 115 – 160 psi (792.9 – 1103.2 KPa). The MEK solvent, unreacted maleic anhydride and volatile compounds were removed via the vacuum line-trap system at the devolatilization port in barrel 11. The vacuum system was operated at 508 mm Hg (67.7 KPa) vacuum. The melt was pelletized using a GALA LPU underwater pelletization system. A two-hole die was used with 7.2°C water temperature and a cutter speed of 1700 rpm. The polymer feed rate to the process was 6.80 kg/h with a fixed extruder screw speed of 500 rpm and a motor torque load of 55 – 65%.

[135] The formulations used for grafting maleic anhydride (MAH) to CBC1-CBC4 are shown in Table 10. Process conditions for production of MAH grafted CBC2 are shown in Table 11. Similar conditions were used for CBC1 and CBC3 – CBC6.

[136] An IR spectrum for MAH-g-CBC2-4 is shown in Figure 4. The anhydride peak can be seen at 1785 cm^{-1}. A typical thermogram for MAH-g-CBC2-4 is shown in Figure 5. The DSC profile shows a melting peak at 131°C which is representative of the CAOP and CAOB and 113°C which corresponds to the CEP and CEB. The observed melt enthalpy is 103 J/g. The thermal properties of the grafted CBC2 are comparable to the original CBC2 before grafting. A typical HTLC for MAH-g-CBC2-4 is shown in Figure 6. The elution profile of CBC2 by HTLC shows that 29 wt% of an early eluting peak between 1.6-2.2 mL and 71 wt% of a later eluting peak between 3-5 mL is eluted. From the concentration and composition measurement, it is determined that the early eluting peak was isolated PP which is CAOP and representative of the CAOB. This is shown by the composition profile of the wt% of C_3 present. The second peak and later eluting peak is rich in C_2 and shows a gradient of C_3. It can be interpreted that this peak is the PE phase and contains the block copolymer and CEP. The composition gradient shows that the block copolymer is eluted earlier and the CEP is eluted last.

[137] The characterizations of MAH-g-CBC and MAH-g-OBC1 are shown in Table 12. Compared to Example A, inventive Examples 1, 2 and 5-8 have higher melting temperatures. All inventive examples in Table 12 have higher melt enthalpy than Example A, which translates to a high crystallinity. The tensile properties of MAH-g-CBC and MAH-g-OBC1 are shown in Table 13. The inventive examples in Table 12 have higher tensile modulus, toughness and ultimate tensile strength.

Table 12. Characterizations of MAH-g-CBC

Example	Description	MFR (2.16kg, 230°C)	Grafting level MAH wt%	Wt% PP From HTLC Separation	Tm (°C) Peak 1 (Peak 2)	Tc (°C)	Melt Enthalpy (J/g)
1	MAH-g-CBC2-2	2.7	0.28	24.0	131 (113)	97	105
2	MAH-g-CBC2-4	0.71	0.64	29.0	131 (113)	95	103
3	MAH-g-CBC1-1	13.0	0.47	23.9	120 (104)	87	98
4	MAH-g-CBC1-2	8.8	0.76	26.8	119 (103)	86	95
5	MAH-g-CBC3-1	87.6	0.46	38.3	136 (103)	94	105
6	MAH-g-CBC3-2	87.1	0.88	41.3	137 (102)	95	96
7	MAH-g-CBC4-1	24.1	0.54	49.6	135 (97)	96	95
8	MAH-g-CBC4-2	31.9	0.87	56.3	136 (97)	98	83
A	MAH-g-OBC1	8.0*	0.98	n/a	121	111	32

* Measured at 2.16kg, 190°C

Table 13. Tensile properties of MAH-g-CBC and MAH-g-OBC1

Example	Description	2% Secant Modulus (psi)/(MPa)	Ult. Elongation (%)	Toughness (in*lbf)/ (Nm)	Ult Tensile (psi)/(MPa)
1	MAH-g-CBC2-2	34057 (234.81)	660	170 (19.21)	3135 (21.62)
2	MAH-g-CBC2-4	35700 (246.14)	697	188 (21.24)	3259 (22.47)
3	MAH-g-CBC1-1	32600 (224.77)	619	111 (12.54)	2226 (15.34)
4	MAH-g-CBC1-2	30550 (210.63)	645	125 (14.12)	2341 (16.14)
5	MAH-g-CBC3-1	43602 (300.63)	357	76 (8.59)	2880 (21.62)
6	MAH-g-CBC3-2	45819 (315.91)	391	85 (9.6)	2886 (19.86)
7	MAH-g-CBC4-1	40924 (282.16)	601	156 (17.63)	3646 (25.14)
8	MAH-g-CBC4-2	41958 (289.29)	601	158 (17.85)	3646 (25.14)
A	MAH-g-OBC1	1735 (11.96)	1456	104 (11.75)	1073 (7.40)

Comparison of MAH-g-CBC and MAH-g-iPP1/EP1 blends

[138] The process of grafting iPP1/EP1 blends is the same as described for the CBCs above. The iPP1 and EP1 are dry blended prior to grafting. The formulations used for grafting MAH to iPP1/EP1 blends and CBC5 and CBC6 are shown in Table 14. Characterizations of MAH grafted iPP1/EP1 blends and CBC5 and CBC6 are shown in Table 15. Tensile properties of MAH grafted iPP1/EP1 blends and CBC5 and CBC6 are shown in Table 16.

[139] At total C2 content of 46-48 wt%, compared to Example D [MAH-g-(EP1/iPP1 50/50 blend)], Example 9 (MAH-g-CBC5) has higher grafting level. Similarly at total C2 content of 28-29 wt%, Example 10 (MAH-g-CBC6) has higher grafting level than Example E [MAH-g-(EP1/iPP1 30/70

Table 16. Tensile properties of MAH-g-CBC and MAH-g-iPP1/EP1 blends

Example	Description	2% Secant Modulus (psi)/(MPa)	Ult. Elongation (%)	Toughness (in*lbf)/(Nm)	Ult Tensile (psi)/(MPa)
9	MAH-g-CBC5	30672 (211.4)	604	119 (13.45)	2157 (14.87)
10	MAH-g-CBC6	47848 (329.9)	495	130 (14.69)	3007 (20.7)
B	MAH-g-EP1	28413 (195.9)	588	121 (13.67)	1906 (13.14)
C	MAH-g-iPP1	79019 (544.8)	184	49 (5.54)	4149 (28.6)
D	MAH-g-(EP1/iPP1 50/50 blend)	45068 (310.7)	58	15 (1.69)	2405 (16.58)
E	MAH-g-(EP1/iPP1 30/70 blend)	50693 (349.5)	17	5 (0.56)	3008 (20.74)

Preparation of MAH-g-BC

[141] The process of grafting iPP1/EP1 blends is the same as described for the CBCs above. The formulations used for grafting MAH to BC are shown in Table 17. Characterizations of MAH-g-BC are shown in Table 18. Compared to Example A, inventive Examples 11 and 12 have higher melting temperatures and higher melt enthalpy than Example A.

Table 17. MAH grafting formulation and resin properties

Example	ID	Description	Feed (wt%)			
			Polymer	MAH	POX	MEK
11	12022-38587	MAH-g-BC1	95.8	2.00	0.20	2.00
12	12022-38588	MAH-g-BC2	95.8	2.00	0.20	2.00

Table 18. Characterizations of MAH-g-CBC and MAH-g-iPP1/EP1 blends

Example	Description	MFR (2.16kg, 230°C)	Grafting level MAH wt%	Wt% PP From HTLC Separation	Total Wt% C_2	Tm (°C) Peak 1 (Peak 2)	Tc (°C)	Melt Enthalpy (J/g)	Oligomer (ppm)
11	MAH-g-BC1	15.9	1.01	34.9	33.8	140 (42)	98	62	198
12	MAH-g-BC2	52.6	0.78	33.0	20.5	138	98	43	196
A	MAH-g-OBC1	8.0*	0.98	n/a	57.4	121	111	32	n/m

the jars. Specifically, 6.3 ml of DEDA (5.23 g) is added. Jars are shaken to distribute the amine, capped lightly with Teflon lined lids and returned to the oven for 1 hr. The jars are removed, shaken again, lids secured with electrical tape and placed inside a 1-gallon (3.78 litre) wide mouthed HDPE jug as a secondary container. The glass bottles are padded with brown crepe paper to keep them from rolling within the secondary jars. Lids of the secondary containers are sealed with electrical tape then put on a Stovall low profile powered roller unit set to maximum rolling speed. Jars are rolled for 2 days. When checked the next day there was still some visible "loose" liquid on the sides so returned the jars to the 50°C oven for 2 hours. Samples are again put in 50 °C oven for at least 3 hours before running.

[147] Haake unit is set to 170°C, allowed to equilibrate and the rotors are calibrated at 100 RPM rotor speed. Imbibed pellets are added to mix bowl and ran for 10 minutes after feed ram secured in closed position. The bowl is opened; sample removed and allowed to cool on Teflon sheet. Films of raw samples (about 3 mil thick) are pressed at 160°C using Carver hydraulic press and checked by FTIR (Perkin-Elmer Spectrum One) for conversion. Samples are then dried under vacuum at 120°C for overnight. An IR spectrum for Imide-g-CBC2-4 is shown in Figure 7. The anhydride (1785-1790 cm^{-1}) band is transformed to the imide band at 1700-1710 cm^{-1} after reaction with the diamine. The disappearance of the anhydride band and formation of imide band indicate the reaction is complete.

Converting MAH-g-OBC1 to Imide-g-OBC1

[148] For converting MAH-g-OBC1 to Imide-g-OBC1, 190g of MAH-g-OBC is weighed into 16 oz., wide mouthed glass jars then warmed in an oven at 50°C temperature for 30 minutes. 6.1 ml of DEDA (5.12 g) is added by gastight syringe into the jars. The rest follows the same procedure as converting MAH-g-CBC2 to Imide-g-CBC2.

[149] The characterizations of Imide-g-CBC and Imide-g-OBC1 are shown in Table 20. Compared to Example J (Imide-g-OBC1), inventive Examples 15and 16 have higher melting temperatures and higher melt enthalpy. The inventive examples in Table 20 have higher tensile modulus, toughness and ultimate tensile strength.

Table 20. Characterizations and tensile properties of imide grafted polymers

Example	Description	MFR (2.16kg, 230°C)	Tm (°C) Peak 1 (Peak 2)	Tc (°C)	Melt Enthalpy (J/g)	2% Secant Modulus (psi)/(MPa)	Ult. Elongation (%)	Toughness (in*lbf)/ (Nm)	Ult Tensile (psi)/ (MPa)
15	Imide-g-CBC2-2	0.79	132 (114)	101	102	48156 (332)	436	113 (0.779)	2893 (19.94)
16	Imide-g-CBC2-4	n/m	133 (113)	99	92	43580 (300)	663	172 (1.19)	3433 (23.67)
J	Imide-g-OBC1	0.33	121	111	29	1214 (8.37)	964	62 (0.427)	565 (3.89)

Preparation of Silane grated OBC2 (VTMOS-g-OBC2)

[150] A ZSK-92 Megacompounder with 11 barrels (45 L/D) is used to graft VTMOS on INFUSE™ 9500 (OBC2). The OBC2 resin is fed with K-Tron T-60 feeder. The barrel temperature is set at 25°C for zone 1 and zone 2 and 190°C for zone 3 and zone 4, 230°C for zone 5 to 8, and 190°C for zone 9 to 11. The die temperature is set at 190°C. A vacuum pump system is used for devolatilization at barrel 9. A minimum of 20 inches Hg (67.7 KPa) is used. The VTMOS/peroxide mixture (20/1, wt/wt) is injected using an American Lewa, Inc. type EH-1 pump with a micromotion flow meter model CMP025 to measure flow. The injection point is at barrel #4. A vacuum pump system is used for devolatilization at barrel 9 and a vacuum of 20 inches Hg (67.7 KPa) is used on order to minimize the concentration of volatile components and residual silane in the melt. The screw RPM is at 250RPM and feed rate of OBC2 is 1800 lb/hr (816.4 kg/hr). The feed formulation is 1.90% VTMOS and 0.095% peroxide. The final VTMOS grafting level is 1.47% and melt index of VTMOS-g-OBC2 is 4.9 (2.16kg, 190°C).

Silane grafting of CBC and BC

[151] The same equipment was used for the preparation of the silane grafted polymer samples as discussed previously for the maleic anhydride grafting examples (1-8). A Coperion ZSK-25MC twin screw extruder is used for the reactive extrusion process to functionalize the base plastic resins. Polymer pellets are fed to the main extruder hopper in barrel 1 using a K-tron Model KCLQX3 loss-in-weight feeder. The liquid reactants were injected into the third barrel section of the extruder using a HPLC precision metering pump (Alltech Model 627 HPLC pump). A dialkyl organic peroxide (Luperox 101, CAS# 78-63-7) is used as the initiator. The liquid feed to the extruder included a blend containing both the vinyltrimethoxysilane monomer and the organic peroxide. In order to minimize the time when the peroxide and monomer are mixed together, the peroxide was dissolved in the vinyltrimethoxysilane immediately before moving the blend to the inlet of the positive displacement pump. The HPLC injection rate was 4.0 ml/min. Typical injection pressures for the liquids were 2.56 – 2.78 MPa. The unreacted vinyltrimethoxysilane and volatile compounds were removed via the vacuum line-trap system at the devolatilization port in barrel 11. The vacuum system was operated at 508 mm Hg vacuum. The melt was pelletized using a GALA LPU underwater pelletization system. A two-hole die was used with 7.2°C water temperature and a cutter speed of 3400 rpm. The polymer feed rate to the process was 11.36 kg/h with a fixed extruder screw speed of 450 rpm and a motor torque load of 63 – 73%.

[152] The formulations used for grafting VTMOS to CBC and BC are shown in Table 21. The process conditions are shown in Table 22. Characterizations of Silane grafted CBC abd BC are shown in Table 23. Gel fraction of crosslinked VTMS-g-CBC, VTMS-g-BC and VTMS-g-OBC2 is shown in Table 24. Compared to Example K (VTMOS-g-OBC2), inventive Examples 17 to 19 have higher melting temperatures and higher melt enthalpy. The inventive examples in Table 23 also have

Table 23. Characterizations of VTMOS-g-CBC, VTMOS-g-BC and VTMOS-g-OBC2

Example	Description	MFR (2.16kg, 230°C)	Grafting level VTMOS wt%	Tm (°C) Peak 1 (Peak 2)	Tc (°C)	Melt Enthalpy (J/g)	2% Secant Modulus (psi)/(MPa)	Ult. Elongation (%)	Toughness (in*lbf)/(Nm)	Ult Tensile (psi) /(MPa)
17	VTMOS-g-CBC5	11.7	1.19	128 (107)	93	98	38698 (266.8)	554	100 (11.3)	2103 (13.88)
18	VTMOS-g-CBC6	35.0	1.06	139 (102)	101	97	69214 (477)	329	73 (8.25)	3097 (21.35)
19	VTMOS-g-BC1	8.5	1.27	141 (44)	100	50	25687 (177)	186	27 (3.05)	1449 (9.99)
K	VTMOS-g-OBC2	4.9*	1.47	122**	102**	52**	2606** (17.97)	1767**	146** (16.5)	1281** (8.83)

* Measured at 2.16kg, 190°C

** Measured on ungrafted OBC2

Table 24. Gel fraction of crosslinked VTMS-g-CBC, VTMS-g-BC and VTMS-g-OBC2

Example	Description	Gel fraction (%)
17	VTMS-g-CBC5	49
18	VTMS-g-CBC6	39
19	VTMS-g-BC1	16
K	VTMS-g-OBC2	>60

5

CLAIMS:

1. A composition comprising a functionalized olefin-based polymer formed from at least (A) and (B):

(A) a block composite comprising:

i) a block copolymer comprising a crystalline propylene-based block and an ethylene/α-olefin block;

ii) a crystalline propylene-based polymer; and,

iii) an ethylene/α-olefin polymer; and

(B) at least one functionalization agent selected from the group consisting of silane, maleic anhydride and amine, wherein the weight percent of polypropylene from HTLC separation before functionalization and the weight percent of polypropylene from HTLC separation after functionalization comprises a difference not exceeding 20 weight percent.

~~2. A composition comprising a functionalized olefin-based polymer formed from at least (A) and (B):~~

~~(A) a crystalline block composite comprising:~~

~~i) a block copolymer comprising a crystalline propylene-based block and an crystalline ethylene-based block;~~

~~ii) a crystalline propylene-based polymer; and,~~

~~iii) an crystalline ethylene-based polymer; and~~

~~(B) at least one functionalization agent.~~

~~3. The composition of Claim 1 wherein the functionalization agent is selected from silane, maleic anhydride, peroxide, and amine.~~

~~4. The composition of Claim 2 wherein the functionalization agent is selected from silane, maleic anhydride, peroxide, and amine.~~