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(54) Title: ETHYLENE/PROPYLENE/NONCONJUGATED DIENE INTERPOLYMER COMPOSITION

(57) Abstract: The present disclosure provides a composition. The composition can be formed into a foam composition. In an embodiment, the composition includes a neat ethylene/propylene/nonconjugated polyene interpolpolymer. The neat ethylene/propylene/nonconjugated polyene interpolpolymer includes from greater than 6.0 wt% to 15.0 wt% nonconjugated polyene. The neat ethylene/propylene/nonconjugated polyene interpolpolymer has the following properties: (i) a molecular weight (Mw) from 240,000 to 270,000; (ii) a Mooney viscosity (ML (1+4), 125°C) from 85 to 95; (iii) a rheology ratio (RR) from 35 to 65; (iv) a Mw/Mn from 2.2 to 3.5; and (v) a Mw < 1389.6 [g/mole] MV + 140,000 g/mol wherein Mw is the weight average molecular weight and MV is the Mooney Viscosity (ML 1+4, 125°C), MV is the Mooney Viscosity (ML 1+4, 125°C), and Mw is the weight average molecular weight, as determined by conventional GPC.



ETHYLENE/PROPYLENE/NONCONJUGATED DIENE INTERPOLYMER COMPOSITION

BACKGROUND

[0001] Ethylene-propylene-nonconjugated polyene terpolymer (also known as "EPDM") is widely used in many automotive and industrial applications. EPDM foam, for example, is used for automotive sealing applications. Conventional EPDM foam, however, is limited because of low foam expansion and rough foam surface quality.

[0002] The art recognizes the ongoing need for new EPDM compositions having higher molecular weight in order to provide improved foam expansion, extrudate surface smoothness, high physical properties and shape retention during extrusion. The art further recognizes the need for new foam compositions made from such EPDM compositions.

SUMMARY

[0003] The present disclosure provides an EPDM composition. The present EPDM composition exhibits high molecular weight, high Mooney viscosity for improved foaming characteristics and foam physical properties. The present disclosure also provides a foam composition made from the present EPDM composition.

[0004] The present disclosure provides a composition. In an embodiment, the composition includes a neat ethylene/propylene/nonconjugated polyene interpolymer. The neat ethylene/propylene/nonconjugated polyene interpolymer includes from greater than 6.0 wt% to 15.0 wt% nonconjugated polyene. The neat ethylene/propylene/nonconjugated polyene interpolymer has the following properties:

- (i) a molecular weight (Mw) from 240,000 to 270,000;
- (ii) a Mooney viscosity (ML (1+4), 125°C) from 85 to 95;
- (iii) a rheology ratio (RR) from 35 to 65;
- (iv) a Mw/Mn from 2.2 to 3.5; and
- (v) a $Mw < 1389.6 \text{ [g/mole]} MV + 140,000 \text{ g/mol}$ wherein Mw is the weight average molecular weight and MV is the Mooney Viscosity (ML 1+4, 125°C). MV is the Mooney Viscosity (ML 1+4, 125°C), and Mw is the weight average molecular weight, as determined by conventional GPC.

[0005] The present disclosure provides a foam composition. In an embodiment, the foam composition includes a neat ethylene/propylene/nonconjugated polyene interpolpolymer. The neat ethylene/propylene/nonconjugated polyene interpolpolymer includes from greater than 6.0 wt% to 15.0 wt% nonconjugated polyene. The neat ethylene/propylene/nonconjugated polyene interpolpolymer has the following properties:

- (i) a molecular weight (Mw) from 240,000 to 270,000;
- (ii) a Mooney viscosity (ML (1+4), 125°C) from 85 to 95;
- (iii) a rheology ratio (RR) from 35 to 65;
- (iv) a Mw/Mn from 2.2 to 3.5; and
- (v) a $Mw < 1389.6 \text{ [g/mole]} MV + 140,000 \text{ g/mol}$ wherein Mw is the weight average molecular weight and MV is the Mooney Viscosity (ML 1+4, 125°C). MV is the Mooney Viscosity (ML 1+4, 125°C), and Mw is the weight average molecular weight, as determined by conventional GPC.

DEFINITIONS

[0005] Any reference to the Periodic Table of Elements is that as published by CRC Press, Inc., 1990–1991. Reference to a group of elements in this table is by the new notation for numbering groups.

[0006] For purposes of United States patent practice, the contents of any referenced patent, patent application or publication are incorporated by reference in their entirety (or its equivalent U.S. version is so incorporated by reference) especially with respect to the disclosure of definitions (to the extent not inconsistent with any definitions specifically provided in this disclosure) and general knowledge in the art.

[0007] The numerical ranges disclosed herein include all values from, and including, the lower and upper value. For ranges containing explicit values (e.g., 1 or 2, or 3 to 5, or 6, or 7), any subrange between any two explicit values is included (e.g., 1 to 2; 2 to 6; 5 to 7; 3 to 7; 5 to 6; etc.).

[0008] Unless stated to the contrary, implicit from the context, or customary in the art, all parts and percentages are based on weight and all test methods are current as of the filing date of this disclosure.

[0009] The terms "blend" or "polymer blend," as used herein, is a blend of two or more

polymers. Such a blend may or may not be miscible (phase separated). Such a blend may or may not be phase separated. Such a blend may or may not contain one or more domain configurations, as determined from transmission electron spectroscopy, light scattering, x-ray scattering, and other methods known in the art.

[0010] The term "composition" refers to a mixture of materials which comprise the composition, as well as reaction products and decomposition products formed from the materials of the composition.

[0011] The terms "comprising," "including," "having" and their derivatives, are not intended to exclude the presence of any additional component, step or procedure, whether or not the same is specifically disclosed. In order to avoid any doubt, all compositions claimed through use of the term "comprising" may include any additional additive, adjuvant, or compound, whether polymeric or otherwise, unless stated to the contrary. In contrast, the term "consisting essentially of" excludes from the scope of any succeeding recitation any other component, step, or procedure, excepting those that are not essential to operability. The term "consisting of" excludes any component, step, or procedure not specifically delineated or listed. The term "or," unless stated otherwise, refers to the listed members individually as well as in any combination. Use of the singular includes use of the plural and vice versa.

[0012] An "ethylene-based polymer" is a polymer containing, in polymerized form, a majority weight percent of units derived from ethylene based on the total weight of the polymer. Nonlimiting examples of ethylene-based polymers include low density polyethylene (LDPE), linear low density polyethylene (LLDPE), very low density polyethylene (VLDPE), ultra low density polyethylene (ULDPE), medium density polyethylene (MDPE), high density polyethylene (HDPE), and functionalized polyethylene, e.g., ethylene vinyl acetate (EVA), ethylene ethyl acrylate (EEA), and the like.

[0013] An "interpolymer" is a polymer prepared by the polymerization of at least two different monomers. This generic term includes copolymers, usually employed to refer to polymers prepared from two different monomers, and polymers prepared from more than two different monomers, e.g., terpolymers, tetrapolymers, etc.

[0014] An "olefin-based polymer" or "polyolefin" is a polymer that contains more than 50

weight percent polymerized olefin monomer (based on total amount of polymerizable monomers), and optionally, may contain at least one comonomer. Nonlimiting examples of an olefin-based polymer include ethylene-based polymer or propylene-based polymer.

[0015] A "polymer" is a compound prepared by polymerizing monomers, whether of the same or a different type, that in polymerized form provide the multiple and/or repeating "units" or "mer units" that make up a polymer. The generic term polymer thus embraces the term homopolymer, usually employed to refer to polymers prepared from only one type of monomer, and the term copolymer, usually employed to refer to polymers prepared from at least two types of monomers. It also embraces all forms of copolymer, e.g., random, block, etc. The terms "ethylene/ α -olefin polymer" and "propylene/ α -olefin polymer" are indicative of copolymer as described above prepared from polymerizing ethylene or propylene respectively and one or more additional, polymerizable α -olefin monomer. It is noted that although a polymer is often referred to as being "made of" one or more specified monomers, "based on" a specified monomer or monomer type, "containing" a specified monomer content, or the like, in this context the term "monomer" is understood to be referring to the polymerized remnant of the specified monomer and not to the unpolymerized species. In general, polymers herein are referred to as being based on "units" that are the polymerized form of a corresponding monomer.

TEST METHODS

[0016] *Absolute tan delta slope.* Absolute value of tan delta slope is defined by Equation (A) below.

$$m = \left| \frac{[\tan\delta@0.1\text{rad/s} - \tan\delta@100\text{rad/s}] * 1000}{0.1\text{rad/s} - 100\text{rad/s}} \right|,$$

[0017] ¹³C NMR Method for EPDM Composition Analysis. The samples were prepared by adding approximately "2.6g" of a "50/50 mixture of tetrachloroethane-d₂/orthodichlorobenzene" that is "0.025M" in chromium acetylacetonate (relaxation agent) to "0.2 g sample" in a 10 mm NMR tube. The samples were dissolved, and homogenized, by heating the tube and its contents to 150°C. The data were collected using a Bruker 400 MHz spectrometer, equipped with a Bruker Dual DUL high-temperature CryoProbe. The data was acquired using "160 scans per data file," a six second pulse repetition delay, with a sample

temperature of 120°C. The acquisition was carried out using a spectral width of 25,000Hz and a file size of 32K data points.

[0018] NMR spectral analysis of the composition of the examples was carried out using the following analysis method. Quantitation of monomers present in EPDM can be calculated using the following equations (1 through 9).

[0019] The calculation of moles ethylene normalizes the spectral range from 55.0-5.0 ppm to 1000 integral units. The contribution under the normalized integral area only accounts for 7 of the ENB carbons. The ENB diene peaks at 111 and 147 ppm are excluded from the calculation due to concerns that double bonds may react at high temperature.

$$\text{Equation 1} \quad \text{molesEth} = \frac{(1000 - 3 * \text{molesP} - 7 * \text{molesENB})}{2}$$

$$\text{Equation 2} \quad \text{molesENB} = \text{CH3}(13.6-14.7\text{ppm})$$

$$\text{Equation 3} \quad \text{molesP} = \text{CH3}(19.5-22.0\text{ppm})$$

$$\text{Equation 4} \quad \text{mole\%ethylene} = \frac{100 * \text{molesE}}{\text{molesE} + \text{molesP} + \text{molesENB}}$$

$$\text{Equation 5} \quad \text{moles\%propylene} = \frac{100 * \text{molesP}}{\text{molesE} + \text{molesP} + \text{molesENB}}$$

$$\text{Equation 6} \quad \text{moles\%ENB} = \frac{100 * \text{molesENB}}{\text{molesE} + \text{molesP} + \text{molesENB}}$$

$$\text{Equation 7} \quad \text{Wt\%ethylene} = \frac{100 * \text{moles\%E} * 28}{\text{mole\%E} * 28 + \text{mole\%P} * 42 + \text{mole\%ENB} * 120}$$

$$\text{Equation 8} \quad \text{Wt\%propylene} = \frac{100 * \text{moles\%P} * 42}{\text{mole\%E} * 28 + \text{mole\%P} * 42 + \text{mole\%ENB} * 120}$$

$$\text{Equation 9} \quad \text{Wt\%ENB} = \frac{100 * \text{moles\%ENB} * 120}{\text{mole\%E} * 28 + \text{mole\%P} * 42 + \text{mole\%ENB} * 120}$$

[0020] *Intermediate Chain Branching %Area* ¹³C NMR. “Intermediate chain branching” (or “iCB”) is the phenomenon wherein oligomers produced *in situ* are rapidly incorporated into the polymer backbone. The branches resulting from incorporation of these oligomers do not increase the viscosity at low shear rates and therefore the resultant polymer exhibits similar behavior to that of linear molecules. Incorporation of the oligomers is highly efficient such that oligomer content is undetectable in the final polymer. It is believed that iCB polymer exhibits a

combination of high density of branches (estimated by NMR) and their intermediate length (30 to 75 carbon units) extend the backbone chain length required for entanglement in the molten state. This behavior is virtually similar to that of linear molecules.

[0021] ^{13}C NMR spectral analysis of the EPDMs to quantitate the level of intermediate chain branching was performed using tetrachloroethane- d_2 with 0.025 M chromium acetylacetonate, to improve the spectral resolution in the 34.6-34.4 ppm region. The samples were prepared by adding approximately "2.6 g" of a "tetrachloroethane- d_2 with 0.025 M chromium acetylacetonate" to "0.3 g sample" in a 10 mm NMR tube. The samples were dissolved, and homogenized, by heating the tube and its contents to 140°C. The data were collected using a Bruker 400 MHz spectrometer with inverse gated NMR pulse, equipped with a Bruker Dual DUL high-temperature CryoProbe. The data was acquired using "8000 scans per data file," a 7.3 second pulse repetition delay, with a sample temperature of 120°C. Spectral data were referenced to the EEE backbone at 30 ppm.

[0022] EPDM polymer with iCB exhibits a methylene peak in the region from 34.4 ppm to 34.6 ppm which is greater than 0.01% of the total integral area from 160.0 to 100.0 ppm plus 60.0 to 0.000 ppm (whole spectra excluding tetrachloroethane solvent) as disclosed in WO2018/005922. ^{13}C NMR spectral analysis of the inventive EPDM displayed no methylene peak in the region from 34.4ppm to 34.6 ppm. The inventive EPDM interpolymer of the present disclosure is void of intermediate chain branching.

[0023] *Density* is measured in accordance with ASTM D792, Method B. The result is recorded in grams per cubic centimeter (g/cc).

[0024] *Differential Scanning Calorimetry (DSC).* Differential Scanning Calorimetry (DSC) is used to measure crystallinity in ethylene-based (PE) samples (including EPDM) and propylene-based (PP) samples. Sample (0.5 g) sample was compression molded into a film, at 5000 psi, 190°C, for two minutes. About 5 to 8 mg of film sample is weighed and placed in a DSC pan. The lid is crimped on the pan to ensure a closed atmosphere. The sample pan is placed in a DSC cell, and then heated, at a rate of approximately 10°C/min, to a temperature of 180°C for PE (230°C for PP). The sample is kept at this temperature for three minutes. Then the sample is cooled at a rate of 10°C/min to -90°C for PE (-90°C for PP), and kept isothermally at that

temperature for three minutes. The sample is next heated at a rate of 10°C/min, until complete melting (second heat). The percent crystallinity is calculated by dividing the heat of fusion (H_f), determined from the second heat curve, by a theoretical heat of fusion of 292 J/g for PE (165 J/g, for PP), and multiplying this quantity by 100 (for example, % cryst. = $(H_f / 292 \text{ J/g}) \times 100$ (for PE)).

[0025] Unless otherwise stated, melting point(s) (T_m) of each polymer is determined from the second heat curve, and the crystallization temperature (T_c) is determined from the first cooling curve.

[0026] *Dynamic Mechanical Spectroscopy (DMS).* Small angle oscillatory shear (melt DMS) was performed using a TA Instruments ARES, equipped with "25 mm parallel plates," under a nitrogen purge. The time between sample loading, and the beginning of the test, was set to five minutes for all samples. The experiments were performed at 190°C, over a frequency range of 0.1 rad/s to 100 rad/s. The strain amplitude was adjusted, based upon the response of the samples from 1 to 3%. The stress response was analyzed in terms of amplitude and phase, from which, the storage modulus (G'), loss modulus (G''), dynamic viscosity η^* , and tan delta were calculated. Specimens for Dynamic Mechanical Spectroscopy were "25 mm diameter x 3.3 mm thick" compression molded discs, formed at 180°C, and 10 MPa molding pressure, for five minutes, and then quenched between chilled platens (15-20°C) for two minutes. The rheology ratio ($V_{0.1}/V_{100}$ at 190°C; also referred to as "RR"), tan delta (0.1rad/s, 190°C) and tan delta (100rad/s, 190°C) were recorded.

[0027] *FTIR Method for EPDM Composition Analysis.* The terpolymers containing ethylene, propylene, and 5-ethylidene- 2-norbornene were analyzed using ASTM D9300 for its ethylene content, and ASTM D6047 for its ethylidene- norbornene or dicyclopentadiene content.

[0028] *Gel Permeation Chromatography.* The chromatographic system consisted of either a Polymer Laboratories Model PL-210 or a Polymer Laboratories Model PL-220. The column and carousel compartments were operated at 140°C. The columns were three Polymer Laboratories, 10-micron Mixed-B columns. The solvent used was 1,2,4 trichlorobenzene. The samples were prepared at a concentration of "0.1 gram of polymer in 50 milliliters of solvent." The solvent used to prepare the samples contained "200 ppm of butylated hydroxytoluene

(BHT).” Samples were prepared by agitating lightly for two hours at 160°C. The injection volume was 100 microliters, and the flow rate was 1.0 milliliters/minute.

[0029] Calibration of the GPC column set was performed with 21 “narrow molecular weight distribution polystyrene standards,” with molecular weights ranging from 580 to 8,400,000 g/mole, arranged in six “cocktail” mixtures, with at least a decade of separation between individual molecular weights. The standards were purchased from Polymer Laboratories (Shropshire, UK). The polystyrene standards were prepared at “0.025 grams in 50 milliliters of solvent” for molecular weights equal to, or greater than, 1,000 kg/mol, and “0.05 grams in 50 milliliters of solvent” for molecular weights less than 1,000 kg/mol. The polystyrene standards were dissolved at 80 degrees Celsius, with gentle agitation, for 30 minutes. The narrow standards mixtures were run first, and in order of decreasing “highest molecular weight” component to minimize degradation. The polystyrene standard peak molecular weights were converted to polyethylene molecular weights using the following equation: $M_{\text{polyethylene}} = A \times (M_{\text{polystyrene}})^B$, where M is the molecular weight, A has a value of 0.431 and B is equal to 1.0 (as described in Williams and Ward, *J. Polym. Sci., Polym. Let.*, 6, 621 (1968)). Polyethylene equivalent molecular weight calculations were performed using Viscotek TriSEC software Version 3.0.

[0030] *Mooney Viscosity.* Mooney Viscosity (ML1+4 at 125°C) was measured in accordance with ASTM D1646, with a one minute preheat time and a four minute rotor operation time. The instrument is an Alpha Technologies Mooney Viscometer 2000. The viscosity of each formulated compositions was measured using an uncured blanket (see experimental section), so that the viscosity of the uncured composition could be examined. Samples were conditioned for 24 hours at room temperature, prior to testing.

[0031] *Tan delta ratio.* Tan delta ratio is defined tan delta (0.1rad/s, 190°C) for the first polymer component produced in the first reactor/tan delta (0.1rad/s, 190°C) of the final composition in the second reactor.

BRIEF DESCRIPTION OF THE DRAWING

[0032] FIGS. 1A and 1B are ^{13}C NMR spectra showing the presence of a peak at 34.4 ppm to 34.6 ppm for a prior art EPDM of comparative sample 3 (CS3) (FIG. 1A) compared to an ^{13}C NMR

spectrum showing no peak at 34.4 ppm to 34.6 ppm for the EPDM of inventive Example 1 (FIG. 1B) in accordance with an embodiment of the present disclosure.

DETAILED DESCRIPTION

[0033] The present disclosure provides a composition. In an embodiment, the composition includes a neat ethylene/propylene/nonconjugated polyene interpolpolymer. The neat ethylene/propylene/nonconjugated polyene interpolpolymer includes from greater than 6.0 wt% to 15.0 wt% nonconjugated polyene. The neat ethylene/propylene/nonconjugated polyene interpolpolymer has the following properties:

- (i) a molecular weight (Mw) from 240,000 to 270,000;
- (ii) a Mooney viscosity (ML (1+4), 125°C) from 85 to 95;
- (iii) a rheology ratio (RR) from 35 to 65;
- (iv) a Mw/Mn from 2.2 to 3.5; and
- (v) a $Mw < 1389.6 \text{ [g/mole]} MV + 140,000 \text{ g/mol}$ wherein Mw is the weight average molecular weight and MV is the Mooney Viscosity (ML 1+4, 125°C).

[0034] MV is the Mooney Viscosity (ML 1+4, 125°C), and Mw is the weight average molecular weight, as determined by conventional GPC.

[0035] The ethylene/propylene/nonconjugated polyene interpolpolymer is neat. A “neat” ethylene/propylene/nonconjugated polyene interpolpolymer contains no added oil, otherwise is void of oil, or otherwise is “oil-free.” It is therefore understood that the Mooney viscosity value and the rheology ratio value are for the neat ethylene/propylene/nonconjugated polyene interpolpolymer that is void of oil.

[0036] The neat ethylene/propylene/nonconjugated polyene interpolpolymer includes ethylene monomer, propylene comonomer, and nonconjugated polyene terpolymer. Although propylene is the copolymer, it is understood that other α -olefins may be used as the comonomer. Nonlimiting examples of suitable comonomers other than propylene include C_4 - C_8 α -olefins such as 1-butene, 1-hexene, and 1-octene.

[0037] The nonconjugated polyene is a C_6 - C_{15} straight chain, branched chain, or cyclic hydrocarbon diene. Nonlimiting examples of suitable straight chain acyclic dienes include 1,4-hexadiene 1,5-heptadiene. Nonlimiting examples of suitable branched chain acyclic dienes

include 5-methyl-1,4-hexadiene, 2-methyl-1,5-hexadiene, 6-methyl-1,5-heptadiene, 7-methyl-1,6-octadiene, 3,7-dimethyl-1,6-octadiene, 3,7-dimethyl-1,7-octadiene, 5,7-dimethyl-1,7-octadiene, 1,9-decadiene, and mixed isomers of dihydromyrcene. Nonlimiting examples of suitable single ring alicyclic dienes include as 1,4-cyclohexadiene, 1,5-cyclooctadiene and 1,5-cyclododecadiene. Nonlimiting examples of multi-ring alicyclic fused and bridged ring dienes include tetrahydroindene, methyl tetrahydroindene, alkenyl, alkylidene, cycloalkenyl and cycloalkylidene norbornenes such as 5-methylene-2-norbornene (MNB), 5-ethylidene-2-norbornene (ENB), 5-vinyl-2-norbornene, 5-propenyl-2-norbornene, 5-isopropylidene-2-norbornene, 5-(4-cyclopentenyl)-2-norbornene, and 5-cyclohexylidene-2-norbornene.

[0038] In an embodiment, the diene is selected from ENB, dicyclopentadiene, 1,4-hexadiene, 7-methyl-1,6-octadiene, dicyclopentadiene and 1,4-hexadiene.

[0039] In an embodiment, the diene is 5-ethylidene-2-norbornene (ENB).

[0040] In an embodiment, the neat ethylene/propylene/nonconjugated polyene interpolmer includes (i) from 50 wt% to 60 wt%, or from 52 wt% to 58 wt%, or from 53 wt% to 56 wt% ethylene monomer, (ii) from 35 wt% to 45 wt%, or from 37 wt% to 43 wt%, or from 38 wt% to 41 wt% propylene comonomer, (iii) from 7 wt% to 10 wt%, or from 8 wt% to 9 wt% ENB, and the neat ethylene/propylene/nonconjugated polyene has the following properties:

- (i) a Mw from 240,000 to 260,000, or from 245,000 to 258,000, or from 248,000 to 256,000,
- (ii) a Mooney viscosity from 90 to 95, or from 92 to 94,
- (iii) a rheology ratio from 35 to 45, or from 37 to 43, or from 39 to 41;
- (iv) a Mw/Mn from 2.8 to 3.2, or from 2.9 to 3.1;
- (v) a $Mw < 1389.6 \text{ [g/mole]} MV + 140,000 \text{ g/mol}$ wherein Mw is the weight average molecular weight and MV is the Mooney Viscosity (ML 1+4, 125°C); and
- (vi) is void of an ^{13}C NMR peak from 34.4 ppm to 34.6 ppm.

Weight percent is based on total weight of the neat ethylene/propylene/nonconjugated polyene interpolmer. The neat ethylene/propylene/nonconjugated polyene interpolmer with the foregoing amounts of ethylene, propylene, and ENB and properties (i)-(vi) is hereafter referred as *composition1*.

[0041] In an embodiment, *composition1* includes (i) from 50 wt% to 60 wt%, or from 52 wt% to 58 wt%, or from 53 wt% to 56 wt% ethylene monomer, (ii) from 35 wt% to 45 wt%, or from 37 wt% to 43 wt%, or from 38 wt% to 41 wt% propylene comonomer, (iii) from 7 wt% to 10 wt%, or from 8 wt% to 9 wt% ENB, and the neat ethylene/propylene/nonconjugated polyene and has the following properties:

- (i) a Mw from 240,000 to 260,000, or from 245,000 to 258,000, or from 248,000 to 256,000,
- (ii) a Mooney viscosity from 90 to 95, or from 92 to 94,
- (iii) and a rheology ratio from 35 to 45, or from 37 to 43, or from 39 to 41.
- iv) a Mw/Mn from 2.8, or 2.9 to 3.1, or 3.2;
- (v) a $Mw < 1389.6 \text{ [g/mole]} MV + 140,000 \text{ g/mol}$ wherein Mw is the weight average molecular weight and MV is the Mooney Viscosity (ML 1+4, 125°C);
- (vi) is void of an ^{13}C NMR peak from 34.4 ppm to 34.6 ppm; and
- (vii) a Mw/VO.1 ratio is less than 1.80 (g/mol) or from 1.5 to 1.7 (g/mol).

[0042] In an embodiment, *composition1* includes (i) from 50 wt% to 60 wt%, or from 52 wt% to 58 wt%, or from 53 wt% to 56 wt% ethylene monomer, (ii) from 35 wt% to 45 wt%, or from 37 wt% to 43 wt%, or from 38 wt% to 41 wt% propylene comonomer, (iii) from 7 wt% to 10 wt%, or from 8 wt% to 9 wt% ENB, and the neat ethylene/propylene/nonconjugated polyene and has the following properties:

- (i) a Mw from 240,000 to 260,000, or from 245,000 to 258,000, or from 248,000 to 256,000,
- (ii) a Mooney viscosity from 90 to 95, or from 92 to 94,
- (iii) and a rheology ratio from 35 to 45, or from 37 to 43, or from 39 to 41.
- iv) a Mw/Mn from 2.8, or 2.9 to 3.1, or 3.2;
- (v) a $Mw < 1389.6 \text{ [g/mole]} MV + 140,000 \text{ g/mol}$ wherein Mw is the weight average molecular weight and MV is the Mooney Viscosity (ML 1+4, 125°C);
- (vi) is void of an ^{13}C NMR peak from 34.4 ppm to 34.6 ppm;
- (vii) a Mw/VO.1 ratio is less than 1.80 (g/mol) or from 1.5 to 1.7 (g/mol); and
- (viii) a Mn from 80,000 to 90,000.

[0043] In an embodiment, the present neat ethylene/propylene/nonconjugated polyene interpolymers are produced in two polymerization reactors that are configured and operated in series, *i.e.* the output of one reactor is fed to the second reactor. The reactors can be the same or different. In an embodiment, each reactor is the same and each reactor is a loop reactor.

[0044] The reactors can be operated in like or different modes. In an embodiment, the first reactor is operated in such a manner that the formation of the high molecular weight fraction of the desired polymer is promoted while the second reactor (*i.e.*, the reactor that receives as a feed the output from the first reactor) is operated in such a manner that the formation of the low molecular weight fraction of the desired polymer is promoted.

[0045] The temperature difference between first reactor and second reactor influences the Mw/Mn of the final product, the greater the temperature difference, the broader the Mw/Mn. While the operating temperature of each reactor will depend upon a host of factors, *e.g.*, the α -olefin(s), the diene, the catalyst, the solvent, equipment and equipment design, pressure, flow rate and turbulence, the relative amounts of the reagents, desired product properties, and the like, a typical operating temperature for the first reactor is from 65°C to 90°C and a typical operating temperature for the second reactor is from 85°C to 120°C. The residence time of the reactants in the individual reactors will also depend upon these factors, but a typical residence time for the reactants in each reactor is from two minutes to 90 minutes.

[0046] Ethylene, α -olefin (*i.e.*, propylene), and diene (if any), solvent and, optionally, hydrogen are blended by any convenient means in desired proportion, and then the blend is introduced into the first reactor, *i.e.*, the reactor whose output is used as a feed for the other or second reactor. A first catalyst is typically introduced into the first reactor apart from the other reagents. The first reactor and the second reactor are operated on a continuous basis such that as output from the first reactor is withdrawn as feed for the second reactor, additional reagents are added to the first reactor to maintain the reaction mass in a steady-state, *i.e.* a relatively constant and proportional concentration of ethylene, α -olefin, diene (if any), solvent, catalyst and molecular weight regulator (if any).

[0047] The first reactor and second reactor are in fluid communication with one another, typically connected by one or more conduits. These conduits are typically equipped with one or more mixers (to promote a homogeneous blending of the reaction mass).

[0048] As with the first reactor, ethylene, α -olefin, diene (if any), solvent and, optionally, hydrogen are blended in desired proportion, and then the blend is introduced into the second reactor, *i.e.*, the reactor that receives as a feed the output from the other or first reactor. Likewise, a second catalyst (a different catalyst from the first catalyst introduced into the first reactor) is typically introduced into the second reactor apart from the other reagents. The output from the first reactor typically contains from 1 weight percent to 30 weight percent solids (*i.e.*, polymer), and it either can be fed to the second reactor apart from the other reagents, or it can first be blended with one or more of the other reagents prior to its introduction into the second reactor. The output from the second reactor typically contains between from 8 weight percent to 30 weight percent solids, in which the desired polymer product is separated from the solvent and unreacted monomers, and the latter (*i.e.*, solvent and unreacted monomers) are individually either recycled or disposed in a safe and environmentally acceptable manner.

[0049] In an embodiment, the neat ethylene/propylene/nonconjugated polyene interpolymer is an in-reactor blend of a first polymer component and a second polymer component. The first polymer component is produced in the first reactor and the second polymer component is produced in the second reactor. The first polymer component is composed of ethylene, propylene, and ENB and has a Mw from 400,000 to 450,000, or from 410,000 to 440,000, or from 420,000 to 430,000.

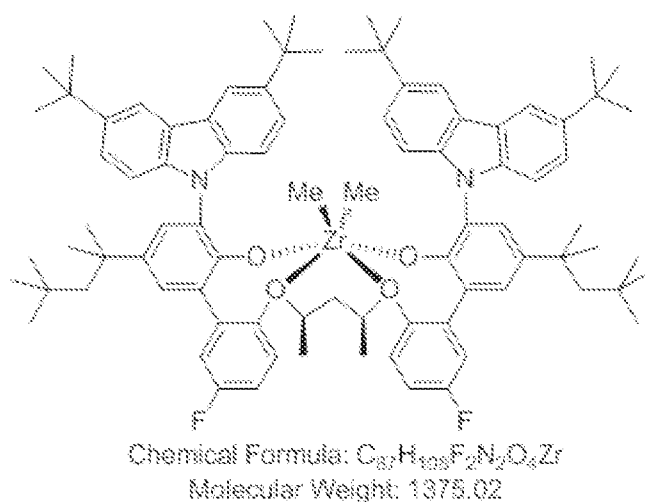
[0050] In an embodiment, the first polymer component is composed of ethylene/propylene/ENB and the ENB content in the first polymer component is less than 80%, or from 60% to less than 80% of the overall ENB content in the final composition.

[0051] A first catalyst can be introduced in the first reactor and a second catalyst can be introduced into the second reactor. The second catalyst can be the same as, or different than, the first catalyst. Alternatively, a single catalyst (first catalyst) is introduced into the first reactor only.

[0052] In an embodiment, a first catalyst is introduced in the first reactor only. The first catalyst (introduced into the first reactor) is a zirconium -based catalyst. The present neat ethylene/propylene/nonconjugated polyene interpolymer contains a residual amount of zirconium.

[0053] In an embodiment, the first catalyst is [[rel-2',2'''-[[[(1R,3S)-1,3-Methyl-1,3-propanediyl]bis(oxy-κO)]bis[3-[3,6-bis(1,1-dimethylethyl)-9H-carbazol-9-yl]-5'-fluoro-5-(1,1,3,3-tetramethylbutyl)][1,1'-biphenyl]-2-olato-κO]](2-)]dimethylzirconium and is hereafter referred to as *catalyst A*. Catalyst A has the structure (A) as provided below.

Structure (A)



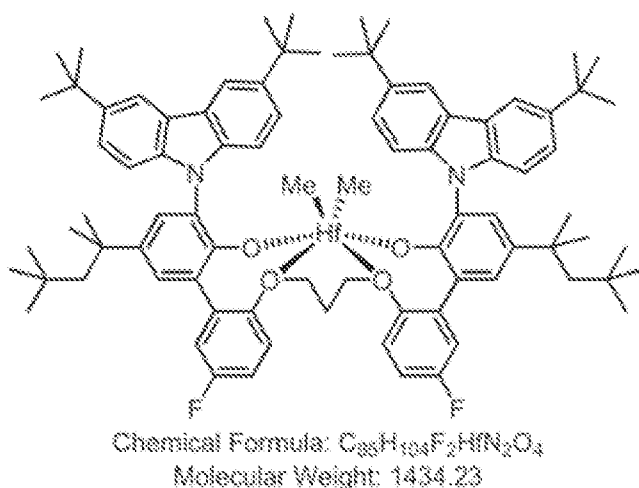
[0054] In an embodiment, a single catalyst is introduced into the first reactor and the single catalyst is a zirconium-based catalyst (catalyst A) and the present neat ethylene/propylene/nonconjugated polyene interpolymer contains a residual amount of zirconium. The term “residual amount,” as used herein is from greater than 0 ppm to 5 ppm.

[0055] In an embodiment, *composition 1* contains from 1 ppm to 5 ppm residual zirconium. In a further embodiment, the residual amount for the zirconium metal is to the exclusion of titanium.

[0056] In an embodiment, a second catalyst is introduced in the second reactor. The second catalyst is [[[2',2'''-[1,3-propanediylbis(oxy-κO)]bis[3-[3,6-bis(1,1-dimethylethyl)-9H-carbazol-9-yl]-5'-fluoro-5-(1,1,3,3-tetramethylbutyl)][1,1'-biphenyl]-2-olato-κO]](2-)]-hafniumdimethyl or OC-6-33)-Dimethyl[[[2',2'''-[1,3-propanediylbis(oxy-κO)]bis[3-[3,6-bis(1,1-

dimethylethyl)-9H-carbazol-9-yl]-5'-fluoro-5-(1,1,3,3-tetramethylbutyl)[1,1'-biphenyl]-2-olato- κ O]](2-)]hafnium and is hereafter referred to as *catalyst B*. *Catalyst B* has the structure (B) provided below.

Structure (B)



2. Additive

[0057] The present neat ethylene/propylene/nonconjugated polyene interpolymer may include optional other additives. Nonlimiting examples of suitable additives include, oil, fillers, antioxidants, UV stabilizers, foaming agents, flame retardants, colorants or pigments, and combinations thereof.

[0058] In an embodiment, oil is added to the neat ethylene/propylene/nonconjugated polyene interpolymer such that the ethylene/propylene/nonconjugated polyene interpolymer is no longer void of oil.

[0059] In an embodiment, the neat ethylene/propylene/nonconjugated polyene interpolymer composition comprises at least one additive selected from fillers, antioxidants, UV stabilizers, foaming agents, or combinations thereof. Fillers include, but are not limited to, carbon black; silicates of aluminum, magnesium, calcium, sodium, potassium and mixtures thereof; carbonates of calcium, magnesium and mixtures thereof; oxides of silicon, calcium, zinc, iron, titanium, and aluminum; sulfates of calcium, barium, and lead; alumina trihydrate; magnesium hydroxide; phenol-formaldehyde, polystyrene, and poly(α -methyl)-styrene resins, natural fibers, synthetic fibers, and the like. In

an embodiment, the filler is carbon black.

[0060] In an embodiment, the neat ethylene/propylene/nonconjugated polyene interpolymer includes an antioxidant. Nonlimiting examples of suitable antioxidant include, hindered phenols, bisphenols, and thiobisphenols; substituted hydroquinones; tris(alkylphenyl)phosphites; dialkylthiodipropionates; phenylnaphthylamines; substituted diphenylamines; dialkyl, alkyl aryl, and diaryl substituted p-phenylene diamines; monomeric and polymeric dihydroquinolines; 2-(4-hydroxy-3,5-t-butylaniline)-4,6-bis(octylthio)l,3,5-triazine, hexahydro-l,3,5-tris-(3,5-di-t-butyl-4-hydroxyphenyl)propionyl-s-triazine, 2,4,6-tris(n-l,4-dimethylpentylphenylene-diamino)-l,3,5-triazine, tris-(3,5-di-t-butyl-4-hydroxybenzyl)isocyanurate, nickel dibutylthiocarbamate, 2-mercaptotolylimidazole and its zinc salt, petroleum waxes, and the like.

3. Foam

[0061] The present disclosure provides a foam composition. In an embodiment, the foam composition includes a neat ethylene/propylene/nonconjugated polyene interpolymer. The neat ethylene/propylene/nonconjugated polyene interpolymer includes from greater than 6.0 wt% to 15.0 wt% nonconjugated polyene. The neat ethylene/propylene/nonconjugated polyene interpolymer has the following properties:

- (i) a molecular weight (Mw) from 240,000 to 270,000;
- (ii) a Mooney viscosity (ML (1+4), 125°C) from 85 to 95;
- (iii) a rheology ratio (RR) from 35 to 65;
- (iv) a Mw/Mn from 2.2 to 3.5; and
- (v) a $Mw < 1389.6 \text{ [g/mole]} MV + 140,000 \text{ g/mol}$ wherein Mw is the weight average molecular weight and MV is the Mooney Viscosity (ML 1+4, 125°C).

[0062] In an embodiment, the foam composition includes *composition1* as previously disclosed above. *Composition1* has the properties as previously disclosed herein.

4. Applications

[0063] The present neat ethylene/propylene/nonconjugated polyene interpolymer composition can be further compounded into a rubber formulation (Banbury mixer or extruder) and further cured into a final article (extrusion, injection molding, calendaring).

[0064] The neat ethylene/propylene/nonconjugated polyene interpolymers and/or final articles may be used in automotive hoses, automotive belts, weatherstrip profiles, architectural profiles, roofing membranes, injection molded articles, general purpose rubber goods, tires, impact modification, TPE compounds, and other traditional and non-traditional rubber elastomer uses.

[0065] By way of example, and not limitation, some embodiments of the present disclosure will now be described in detail in the following Examples.

EXAMPLES

1. Synthesis of Experimental EPDM

[0066] Continuous Polymerization

[0067] The polymerization reaction was performed under steady state conditions, that is, constant reactant concentration and continual input of solvent, monomers, and catalyst, and constant withdrawal of unreacted monomers, solvent and polymer. The reactor system was cooled and pressurized to prevent formation of a vapor phase. Monomers: ethylene (CAS 74-85-1); propylene (CAS 115-07-1); 5-ethylidene-2-norbornene, ENB (CAS 16219-75-3).

[0068] The polymer composition was produced in a solution polymerization process using two reactors, (i) a continuous stirred-tank reactor ("first reactor") followed by (ii) a loop reactor ("second reactor"). Ethylene was introduced in a mixture of a solvent of ISOPAR E (available from ExxonMobil), propylene was introduced and 5-ethylidene-2-norbornene (ENB) was introduced, each forming a reactor feed stream.

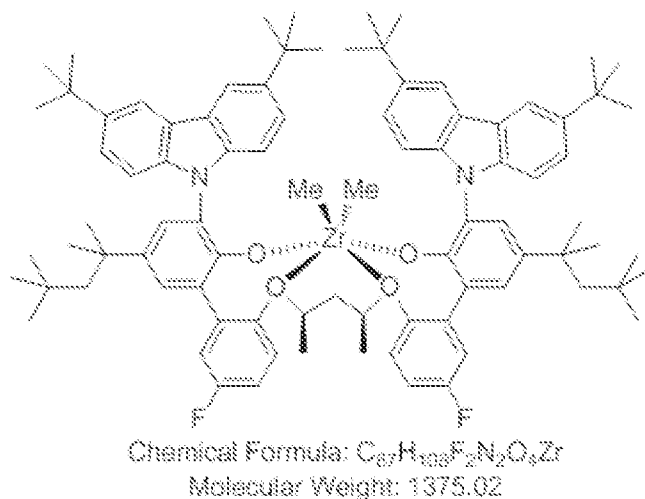
[0069] Catalyst A was fed to the first reactor and catalyst A was activated *in-situ* using co-catalyst 1 and co-catalyst 2.

[0070] Cocatalyst-1 is a mixture of methyl-di(C₁₄₋₁₈ alkyl)ammonium salts of tetrakis(pentafluorophenyl)borate, prepared by reaction of a long chain trialkylamine (ARMEEN M2HT, available from Akzo-Nobel, Inc.), HCl and Li[B(C₆F₅)₄], substantially as disclosed in USP 5,919,988 (Ex. 2). Cocatalyst-1 is from Boulder Scientific, and used without further purification.

[0071] Cocatalyst-2 is modified methylalumoxane (MMAO), from Akzo Nobel, and used without further purification.

[0072] The structure for catalyst A is provided below.

[0073] Catalyst A: [[rel-2',2'''-[[[(1R,3S)-1,3-Methyl-1,3-propanediyl]bis(oxy-κO)]]bis[3-[3,6-bis(1,1-dimethylethyl)-9H-carbazol-9-yl]-5'-fluoro-5-(1,1,3,3-tetramethylbutyl)[1,1'-biphenyl]-2-olato-κO]](2-)]dimethylzirconium. CAS # 1360629-63-5.



[0074] The outlet of each reactor was consequently a mixture of polymer, solvent, and reduced levels of the initial monomers. The outlet of the first reactor was fed directly into the second reactor (unless otherwise sampled). The molecular weight of the polymer was controlled by adjusting each reactor's temperature, monomer conversion and/or the addition of a chain terminating agent such as hydrogen.

[0075] After polymerization, a small amount of water was introduced into the reactor exit stream as a catalyst kill, and the reactor exit stream was introduced into a flash vessel, in which the solids concentration was increased by at least 100 percent. A portion of the unreacted monomers, that is, ENB, ethylene, and propylene, and the unused diluent were then collected, and recycled back to the reactor feeds as appropriate.

[0076] Monomer feed rate and polymerization temperature and other conditions are listed in below in Table 1 and 2 below.

Table 1: Reaction Conditions for Inventive EPDM

EXAMPLE	REACTOR TEMP. [DEGC]	PRESSURE [PSIG]	SOLVENT/ ETHYLENE FEED RATIO [LB/LB]	PROPYLENE/ ETHYLENE FEED RATIO [LB/LB]	ENB/ ETHYLENE FEED RATIO [LB/LB]
EPDM01-R1 (first reactor)	123.08	729.02	12.97	2.06	0.41
EPDM01 (second reactor)	120.0	725	4.51	0.42	0.19

Table 2: Reaction Conditions for Inventive EPDM

Example	H2 Mol %	C2 Concen. [g/L]	Catalyst Efficiency [lb_poly/ lb_metal] *10E6	Borate/ Cat. Metal Ratio [mol/mol]	Aluminum/ Cat. Metal Ratio [mol/mol]
EPDM01 (first reactor)	0.01	23.69	1.93	1.99	29.96
EPDM01(second reactor)	0.11	8.16	0.42	2.00	30.04

Table 3: Property Data for Comparative and Inventive EPDM

	CS1	CS2		IE 1		CS3	CS4	CS5
	R2 (final)	R1	R2	R1	R2 (final)			
%C2	55	48.9	49	57.9	53.3	69.4	70.5	70
%ENB	8.5	4.4	4.9	6.4	8.3	5.1	4.3	4.9
Mooney	65		87		92	70.3	70	70
Mn (g/mol)	71,482		94237	189326	84589	68551	63907	60648
Mw (g/mol)	202680	369661	253846	422692	256733	167265	264574	163750
Mz (g/mol)	478163	688393	597609	831103	582681	337137	863056	366099
Mw/Mn	2.84	2.09	2.7	2.23	3.04	2.44	4.14	2.7
0.1, 190°C (Pa-s)	138442	452095	114000	860444	153457	49392	116420	107012
100, 190°C (Pa-s)	3388	8052	4240	9789	3898	4842	3927	3441
RR V0.1/V100	41	56	27	88	39	10.2	30	31
Tan Delta@0.1 rad/s	1.05	2.19	2.03	1.69	1.40	3.27	2.17	1.45
Tan Delta@100 rad/s	0.56	0.26	0.48	0.18	0.45	0.64	0.59	0.623
Absolute Tan delta slope m	4.91	19.29	15.50	15.03	9.50	26.3	15.8	8.3
Overall (R2)Mw/V0.1	1.5	0.8	2.2	0.5	1.7	3.4	2.3	1.5
Overall Mw/((1389.6*MV+115 000))	0.99		1.08		1.05	0.8	1.3	0.8
Overall Mw/((1389.6*MV+140 000))	0.88		0.97		0.95	0.70	1.1	0.7
V0.1/ 0.5464 [(Pa-s)(mole/g) Mz - 155,000 Pa-s],	0.78	1.55	0.47	2.33	0.66	0.5	0.3	0.9
V0.1/ 0.5464 [(Pa-s)(mole/g) Mz - 200,000 Pa-s],	0.91	1.69	0.52	2.50	0.73	0.7	0.3	1.2

*CS3 is Inv. 1 Table 3, FIG. 3, WO2018/005922

*CS4 is Inv. 2 table 4, WO2018/005852

*CS5 is EPDM B47 Table 2. WO2014/003857

IE—inventive example

[0077] IE1 contains 38 wt% of the first polymer component (EPDM produced in reactor 1) and 62 wt% of the second polymer component (EPDM produced in reactor 2). Weight percent is based on total weight of the final EPDM terpolymer.

[0078] The inventive example (IE1) contains more ENB (8.3 wt%) than CS2 (4.9 wt% ENB), CS3 (5.1 wt% ENB), CS4 (4.3 wt% ENB) and CS5 (4.9 wt% ENB) which is beneficial for the production of foam compositions.

[0079] As shown in FIG. 1B, IE1 has no iCB (no ^{13}C NMR peak at 34.4ppm to 34.6 ppm) whereas CS3 has iCB as evident by the ^{13}C NMR peak at 34.4 ppm to 34.6 ppm in FIG. 1A.

[0080] IE1 has a Mw/Mn of 3.04 which is broader than the Mw/Mn of CS1 (2.84), CS2 (2.09), CS3 (2.44), and CS5 (2.7) and narrower than CS4 (4.14).

[0081] IE1 has Mw less than the Mooney viscosity relationship of $1389.6[\text{g/mole}] \text{ MV} + 140,000 \text{ g/mol}$.

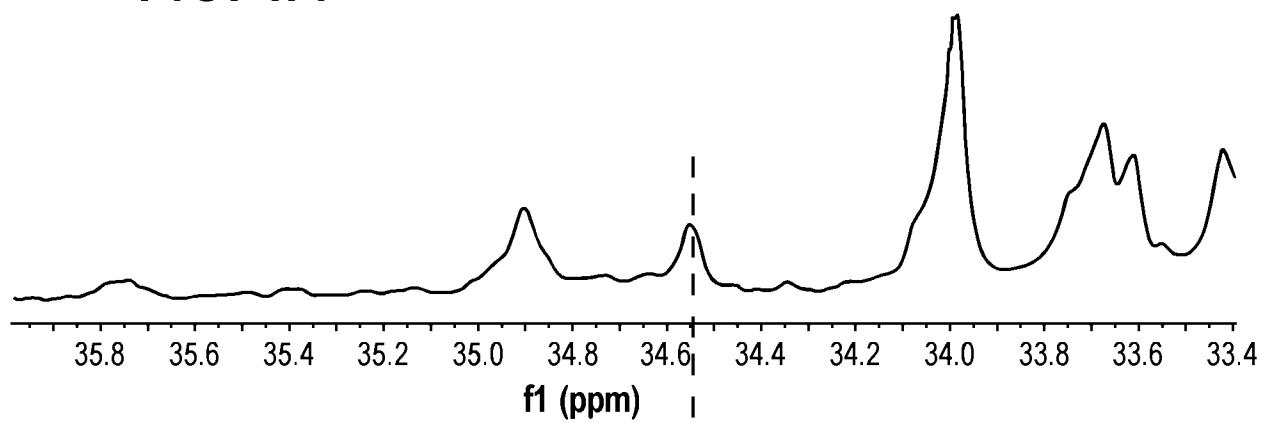
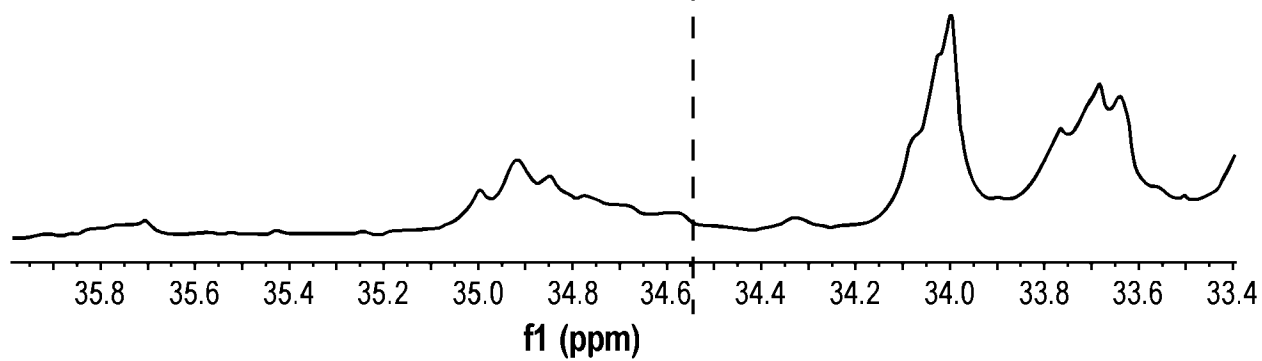
[0082] IE1 exhibits high Mn (80,000-90,000), medium Mw/Mn (2.8-3.5), and high ENB (6-15 wt%) which yields beneficial features of suitable expansion, suitable extrudate surface smoothness and suitable shape retention during extrusion for foam applications.

[0083] It is specifically intended that the present disclosure not be limited to the embodiments and illustrations contained herein, but include modified forms of those embodiments including portions of the embodiments and combinations of elements of different embodiments as come within the scope of the following claims.

CLAIMS

1. A composition comprising:
a neat ethylene/propylene/nonconjugated polyene interpolpolymer comprising from greater than 6.0 wt% to 15 wt% nonconjugated polyene, the interpolpolymer having the following properties
(i) a molecular weight (Mw) from 240,000 to 270,000;
(ii) a Mooney viscosity (ML (1+4), 125°C) from 85 to 95;
(iii) a rheology ratio from 35 to 65;
(iv) a Mw/Mn from 2.2 to 3.5; and
(v) a $Mw < 1389.6 \text{ [g/mole]} MV + 140,000 \text{ g/mol}$ wherein Mw is the weight average molecular weight and MV is the Mooney Viscosity (ML 1+4, 125°C).
2. The composition of claim 1 wherein the neat ethylene/propylene/nonconjugated polyene interpolpolymer has a Mw/V0.1 ratio less than 1.80 (g/mol).
3. The composition of any of claims 1-2 wherein the neat ethylene/propylene/nonconjugated polyene interpolpolymer is void of a ^{13}C NMR peak from 34.4 ppm to 34.6 ppm.
4. The composition of claim any of claims 1-3 wherein the neat ethylene/propylene/nonconjugated polyene interpolpolymer comprises
from 50 wt% to 60 wt% ethylene monomer;
from 35 wt% to 45 wt% propylene comonomer; and
from 7 wt% to 10 wt% 5-ethylidene-2-norbornene termonomer (ENB).
5. The composition of claim 4 wherein the neat ethylene/propylene/nonconjugated polyene interpolpolymer has the following properties
(i) a molecular weight (Mw) from 240,000 to 260,000;
(ii) a Mooney viscosity (ML (1+4), 125°C) from 90 to 95;
(iii) a rheology ratio from 35 to 45;
(iv) a Mw/Mm from 2.8 to 3.2; and
(v) a $Mw < 1389.6 \text{ [g/mole]} MV + 140,000 \text{ g/mol}$.

6. The composition of any of claims 1-5 wherein the neat ethylene/propylene/nonconjugated polyene interpolymer is an in-reactor blend of
- a first polymer component composed of ethylene/propylene/ENB and having a Mw from 400,000 to 450,000; and
 - a second polymer component composed of ethylene/propylene/ENB.
7. The composition of any of claims 1-6 wherein the neat ethylene/propylene/nonconjugated polyene interpolymer comprises from 1 ppm to 5 ppm zirconium.
8. A foam composition comprising:
- a neat ethylene/propylene/nonconjugated polyene interpolymer comprising from greater than 6.0 wt% to 15 wt% nonconjugated polyene, the interpolymer having the following properties
- (i) a molecular weight (Mw) from 240,000 to 270,000;
 - (ii) a Mooney viscosity (ML (1+4), 125°C) from 85 to 95;
 - (iii) a rheology ratio from 35 to 65;
 - (iv) a Mw/Mn from 2.2 to 3.5; and
 - (v) a $Mw < 1389.6 \text{ [g/mole]} MV + 140,000 \text{ g/mol}$ wherein Mw is the weight average molecular weight and MV is the Mooney Viscosity (ML 1+4, 125°C).

FIG. 1A**FIG. 1B**

INTERNATIONAL SEARCH REPORT

International application No.

PCT/CN2019/085137

A. CLASSIFICATION OF SUBJECT MATTER

C08L 23/16(2006.01)i

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

C08L

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

CNABS, VEN, CNKI, CA, ethylene, propylene, polyene, diene, Mooney, rheology

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	WO 2018005852 A1 (DOW GLOBAL TECHNOLOGIES LLC) 04 January 2018 (2018-01-04) the whole document	1-10
A	WO 9530698 A1 (DSM NV et al.) 16 November 1995 (1995-11-16) the whole document	1-10



Further documents are listed in the continuation of Box C.



See patent family annex.

* Special categories of cited documents:

“A” document defining the general state of the art which is not considered to be of particular relevance

“E” earlier application or patent but published on or after the international filing date

“L” document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

“O” document referring to an oral disclosure, use, exhibition or other means

“P” document published prior to the international filing date but later than the priority date claimed

“T” later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

“X” document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

“Y” document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art

“&” document member of the same patent family

Date of the actual completion of the international search

09 January 2020

Date of mailing of the international search report

16 January 2020

Name and mailing address of the ISA/CN

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INTERNATIONAL SEARCH REPORT
Information on patent family members

International application No.

PCT/CN2019/085137

Patent document cited in search report			Publication date (day/month/year)	Patent family member(s)			Publication date (day/month/year)
WO	2018005852	A1	04 January 2018	KR	20190022785	A	06 March 2019
				CN	109563326	A	02 April 2019
				EP	3478762	A1	08 May 2019
				BR	112018077402	A2	09 April 2019
				JP	2019519660	A	11 July 2019
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				AU	2149895	A	29 November 1995
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				CN	1150809	A	28 May 1997
				DE	69503778	T2	15 April 1999
				MX	9605423	A	31 December 1997
				TW	294681	B	01 January 1997