

(19) World Intellectual Property Organization  
International Bureau



(43) International Publication Date  
13 November 2008 (13.11.2008)

PCT

(10) International Publication Number  
**WO 2008/137285 A1**

(51) International Patent Classification:  
**B65D 65/38** (2006.01)

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(21) International Application Number:  
PCT/US2008/060769

(81) Designated States (*unless otherwise indicated, for every kind of national protection available*): AE, AG, AL, AM, AO, AT, AU, AZ, BA, BB, BG, BH, BR, BW, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DO, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, GT, HN, HR, HU, ID, IL, IN, IS, JP, KE, KG, KM, KN, KP, KR, KZ, LA, LC, LK, LR, LS, LT, LU, LY, MA, MD, ME, MG, MK, MN, MW, MX, MY, MZ, NA, NG, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RS, RU, SC, SD, SE, SG, SK, SL, SM, SV, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, ZA, ZM, ZW.

(22) International Filing Date: 18 April 2008 (18.04.2008)

(25) Filing Language: English

(26) Publication Language: English

(30) Priority Data:  
60/915,507 2 May 2007 (02.05.2007) US

(84) Designated States (*unless otherwise indicated, for every kind of regional protection available*): ARIPO (BW, GH, GM, KE, LS, MW, MZ, NA, SD, SL, SZ, TZ, UG, ZM, ZW), Eurasian (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European (AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HR, HU, IE, IS, IT, LT, LU, LV, MC, MT, NL, NO, PL, PT, RO, SE, SI, SK, TR), OAPI (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG).

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**Published:**

— with international search report

(54) Title: BATCH INCLUSION PACKAGES

(57) Abstract: Batch inclusion packages made from films and film structures are disclosed. The films and film structures have at least one layer comprising a polyolefin plastomer or elastomer together with relatively high levels of inorganic filler. The batch inclusion packages are useful in wrapping or containing various materials, such as asphalt or bitumen, as the packages are very miscible in such materials.



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**BATCH INCLUSION PACKAGES**Cross-Reference to Related Applications

This application is a non-provisional application claiming priority from the U.S. Provisional Patent Application No. 60/915,507, filed on May 2, 2007, entitled "BATCH INCLUSION PACKAGES," the teachings of which are incorporated by reference herein, as if reproduced in full hereinbelow.

Field of the Invention

This invention relates to batch inclusion packages made from compositions comprising polyolefin plastomers together with an inorganic, filler particularly calcium carbonate. The packages are made from films and are used to contain and protect powders, pellets and flowable materials, wherein the entire package (that is, film and contents) can be added to a mixture during production of a product, for example, placing the package into the extruder/mixer simultaneously with the contained product.

Background and Summary of the Invention

Batch inclusion packages are widely used to contain materials such as carbon black, titanium dioxide powder, elastomers (rubbers), polystyrene pellets and other chemicals. The batch inclusion packages minimize dust health hazards and possible fire hazards, as well as waste, since the entire package and its contents are processed during use of the contents. It is desired to also be able to package bitumen or asphalt in a batch inclusion bag. The batch inclusion package has numerous requirements when used in such a manner. These include compatibility with the material to be contained, low melting

5 temperature, a narrow softening range, and extrudability. In the case of packaging bitumen the bags must be able to withstand the higher temperatures of these materials, typically around 165°C.

USP 4,248,348 (Butler et al.) discloses a package comprising an article (such as an unvulcanized rubber) packaged in a film. The film  
10 is made from an ethylene/vinyl acetate copolymer containing an antiblock agent. USP '348 teaches that polyethylene film is not suitable for overwrapping bales of rubber, because when the bale (including the film wrapper) is charged into a suitable mixer, the polyethylene film is not sufficiently dispersed and causes defects in the end product.

15 Similarly, USP 5,120,787 (Drasner) discloses a method of compounding a rubber by using a bag or liner made from an ethylene/vinyl acetate copolymer. The bag or liner is directly compounded into the mixer and becomes part of the rubber compound.

USP 4,394,473 (Winter et al.) discloses packages comprising  
20 an article (for example, unvulcanized or vulcanized rubber) packaged in a bag made from syndiotactic 1,2-polybutadiene containing an antiblock additive. USP '473 also teaches that the use of polyethylene bags results in incompatibility problems.

EPA 0 270 902 discloses the use of bags made from  
25 copolymers of at least one olefin monomer (for example, ethylene) and acrylic or methacrylic acid, or ionomers thereof, used to package particulate elastomer materials.

Branched, high pressure low density polyethylene (LDPE) and blends of LDPE with small amounts of traditional (that is, Ziegler  
30 polymerized) linear low density polyethylene (LLDPE), typically less than 10 percent LLDPE, are sometimes used to make batch inclusion bags which are used to contain carbon black. The carbon black is then

5 used to make tires. However, the melting and softening characteristics of LDPE limits its use as a batch inclusion packaging material.

Use of traditional LLDPE in batch inclusion is limited because of its relatively high melting point and broad softening range. US 5,525,659 discloses batch inclusion bags made from a particular class  
10 of LLDPE, namely substantially linear ethylene polymers.

It would be desirable to have batch inclusion bags which maintain their durability during hot filling and yet are even more miscible with the contents when used.

It has been discovered that the addition of relatively high  
15 amounts of filler, preferably inorganic filler and most preferably calcium carbonate, leads to improved miscibility of polyolefin based plastomers and/or elastomers with bitumen, asphalt or similar materials. The inorganic is added in an amount of from 15 to 60 percent by weight of the film layer. Calcium carbonate is the preferred filler.

20 The resulting compounded resin can be made into blown films which can be used as preformed bags or tube stock to be converted into bags via a form-fill-seal process at the producer. Bags made from such resin are particularly suited for packaging bitumen or similar materials as they can be hot filled, have adequate hot tack strength and are particularly  
25 miscible with molten bitumen.

#### Detailed Description of the Invention

In a first aspect, the present invention relates to a package which comprises:

- (A) an ingredient;
- 30 (B) a protective film comprising at least one layer comprising
  - i) at least one polyolefin plastomer or elastomer or segmented ethylene-alpha-olefin block copolymer, and

- 5                                    ii) an inorganic filler in an amount of from 15 to 60 percent by weight of the layer, wherein the protective film (B) is miscible with the ingredient under conditions in which the ingredient is used.

                                         The protective film for use in the present invention comprises at least one polyolefin plastomer or elastomer. Elastomers are known in the art and mean a material which at room temperature can be stretched under  
10                                    low stress to at least twice its original length and upon immediate release of the stress, will return with force to its approximate original length. Plastomers are also generally known in the art. For polyethylenes, plastomers will generally contain 20 percent or less of the comonomer  
15                                    (preferably 1-octene) and will have a density of 0.90 g/cm<sup>3</sup> or more, and for elastomers the level of comonomer content is generally over 20 percent and the density is less than 0.90 g/cm<sup>3</sup>.

                                         The preferred polyolefin based plastomers and/or elastomers include polyethylene plastomers and elastomers, polypropylene plastomers  
20                                    and elastomers, and segmented ethylene-alpha-olefin block copolymers. Polyethylene based elastomers and plastomers include homogeneously branched linear ethylene polymers such as those in U.S. Patent No. 3,645,992 and substantially linear ethylene polymers such as those described in U.S. Patent 5,272,236, U.S. Patent 5,278,272, U.S. Patent 5,582,923 and US  
25                                    Patent 5,733,155 and/or blends thereof (such as those disclosed in US 3,914,342 or US 5,854,045). Each of these references is hereby incorporated by reference in their entirety. Polyethylene based polymers also include high pressure copolymers of ethylene such as ethylene vinyl acetate  
interpolymer, ethylene acrylic acid interpolymer, ethylene ethyl acetate  
30                                    interpolymer, ethylene methacrylic acid interpolymer, ethylene methacrylic acid ionomer, and the like. The substantially linear ethylene polymers are preferred. Substantially linear ethylene polymers are commercially

5 available from The Dow Chemical Company under the trade name  
AFFINITY™. Propylene based elastomers and plastomers include the  
propylene based plastomers and elastomers described in WO03/040442,  
and US application 60/709688 filed August 19, 2005 (each of which is  
hereby incorporated by reference in its entirety - some of these materials are  
10 commercially available from The Dow Chemical Company under the trade  
name VERSIFY™), and the propylene based plastomers and elastomers sold  
by ExxonMobil Chemical company under the trade name of  
VISTAMAXX™. The propylene based elastomers and plastomers are most  
preferred. Segmented ethylene-alpha-olefin block copolymers include those  
15 discussed for example in WO 2005/090427, WO 2005/090425 and WO  
2005/090426, each of which are hereby incorporated by reference in their  
entirety.

The polyolefin plastomer or elastomer for use in a batch  
inclusion package application is generally selected based on the  
20 processability of the polymer into batch inclusion packaging material and  
through manufacturing process in which the batch inclusion package is  
used. For example, a polymer having a density low enough to ensure  
adequate melting during use is required, but not too low so that the  
resultant film has too much stretch during the hot fill process or subsequent  
25 palletization and transportation of the filled bags. The film also must have  
enough strength and must not soften too much prior to or during the  
manufacturing process. In addition, a polyolefin plastomer or elastomer  
polymer can have a higher I<sub>10</sub>/I<sub>2</sub> value, which can provide a more stable  
bubble during blown film production of batch inclusion film. The melt  
30 index of the polymer also must be selected based on the bubble stability  
and/or film strength. Selection of a polymer for use in a particular batch

5 inclusion package is thus based on a variety of factors and the foregoing parameters are intended to be illustrative and not limiting.

In some embodiments the melting range of the polyolefin plastomer or elastomer is preferably relatively narrow, so that the polymer melts quickly when it reaches the appropriate temperature. Generally, the  
10 narrow melting range of the polymer is indicated by the difference between the melting point and the softening point. For the preferred polyolefin plastomers or elastomers used in the present invention, the melting point is relatively close to the Vicat Softening point (typically within 9-25°C) of the polymer, such that the bag does not soften too much during transport and  
15 to prevent "strings" of poorly blended/unmelted polymer from forming during use of the bag in the end-user's manufacturing process.

Vicat Softening point is measured herein in accordance with ASTM D 1525-87 using 0.125 inch (thick) plaques having a diameter of 1 inch prepared according to ASTM D-1928-90-C. Heating rate B of the  
20 ASTM test is used ( $120 \pm 12$  °C/hour). The films of the present invention preferably have a Vicat Softening point in the range of from 60°C to 103°C

Preferred polyethylene based resins are will have a melt index ("MI" or "I<sub>2</sub>") between 0.3 and 5 g/10min (190°C, 2.16kg). Preferred polypropylene based resins will have a melt flow rate (MFR) between 1 and  
25 10 g/10min (230°C, 2.16kg).

In one preferred embodiment, the polyolefin plastomer or elastomer is a substantially linear polyethylene. The preferred density for substantially linear ethylene/alpha-olefin polymers (as measured in accordance with ASTM D-792) for use in the present invention is generally  
30 less than about 0.94 g/cm<sup>3</sup>, preferably from 0.87 g/cm<sup>3</sup> to 0.92 g/cm<sup>3</sup>. The density of the preferred substantially linear ethylene/alpha-olefin polymers used in the present invention is generally dependent upon the

5 processability of the substantially linear ethylene/alpha-olefin polymer into batch inclusion packaging materials and packages, the strength required of the batch inclusion packaging material, and the manufacturing process in which the batch inclusion package is used. Generally for elastomers, the density of the substantially linear ethylene/alpha-olefin polymer used to  
10 make the batch inclusion package is from 0.87 g/cm<sup>3</sup> to 0.917 g/cm<sup>3</sup>.

The molecular weight of the preferred substantially linear ethylene/alpha-olefin polymers for use in the present invention is conveniently indicated using a melt index measurement according to ASTM D-1238, Condition 190°C/2.16 kg (formerly known as "Condition (E)" and  
15 also known as I<sub>2</sub>). Melt index is inversely proportional to the molecular weight of the polymer. Thus, the higher the molecular weight, the lower the melt index, although the relationship is not linear. The melt index for the preferred substantially linear ethylene/alpha-olefin polymers useful herein is generally that useful in forming film in conventional film forming  
20 manufacturing operations, such as cast film, blown film and extrusion coating techniques. The melt index will generally be from 0.1 grams/10 minutes (g/10 min) to 20 g/10 min, preferably from 0.3 g/10 min to 5 g/10 min.

Another measurement useful in characterizing the molecular  
25 weight of the preferred substantially linear olefin polymers is conveniently indicated using a melt index measurement according to ASTM D-1238, Condition 190°C/10 kg (formerly known as "Condition (N)" and also known as I<sub>10</sub>). The ratio of these two melt index terms is the melt flow ratio and is designated as I<sub>10</sub>/I<sub>2</sub>. Generally, the I<sub>10</sub>/I<sub>2</sub> ratio for the linear is at  
30 least about 5.63, preferably at least about 7, especially at least about 8 or above. For the substantially linear ethylene/alpha-olefin polymers used in the batch inclusion packages of the invention, the I<sub>10</sub>/I<sub>2</sub> ratio indicates the

5 degree of long chain branching, that is, the higher the  $I_{10}/I_2$  ratio, the more long chain branching in the polymer. Generally, the  $I_{10}/I_2$  ratio of the preferred substantially linear ethylene/ $\alpha$ -olefin polymers is at least about 5.63, preferably at least about 7, especially at least about 8. Generally, the upper limit of  $I_{10}/I_2$  ratio for the preferred homogeneously branched  
10 substantially linear ethylene/ $\alpha$ -olefin polymers is about 50 or less, preferably about 30 or less, and especially about 20 or less.

In another preferred embodiment, the polyolefin plastomer or elastomer is a propylene based plastomer or elastomer or "PBPE". These materials comprise at least one copolymer with at least about 50 weight  
15 percent of units derived from propylene and at least about 5 weight percent of units derived from a comonomer other than propylene. Suitable propylene based elastomers and/or plastomers are taught in WO03/040442, and US application 60/709688 (filed August 19, 2005), each of which is hereby incorporated by reference in its entirety

20 The preferred density for PBPE (as measured in accordance with ASTM D-792) for use in the present invention is generally less than about  $0.888 \text{ g/cm}^3$ , preferably from  $0.867 \text{ g/cm}^3$  to  $0.888 \text{ g/cm}^3$ . The density of the preferred PBPE used in the present invention is generally dependent upon the processability of the PBPE into batch inclusion  
25 packaging materials and packages, the strength required of the batch inclusion packaging material, and the manufacturing process in which the batch inclusion package is used. Generally for elastomers, the density of the PBPE used to make the batch inclusion package is from  $0.867 \text{ g/cm}^3$  to  $0.888 \text{ g/cm}^3$ .

30 The molecular weight of the preferred PBPE polymers for use in the present invention is conveniently indicated using a melt flow rate measurement according to ASTM D-1238, Condition 230°C/2.16 kg and also

5 known as I<sub>2</sub>). Melt flow rate is inversely proportional to the molecular weight of the polymer. Thus, the higher the molecular weight, the lower the melt index, although the relationship is not linear. The melt flow rate for the preferred substantially PBPE polymers useful herein is generally that useful in forming film in conventional film forming manufacturing  
10 operations, such as cast film, blown film and extrusion coating techniques. The melt flow rate will generally be from 1 grams/10 minutes (g/10 min) to 10 g/10 min, preferably from 2 g/10 min to 5 g/10 min.

The polyolefin plastomers or elastomers used in the film of the batch inclusion packages are preferably used as the only polymer  
15 components. However, other polymers, including two or more different polyolefin plastomers or elastomers can be blended and/or multilayered extruded and/or multilayered laminated to modify the film processing, film stiffness, film barrier properties, film strength, film melting characteristics, or other desirable film characteristics. Batch inclusion films made with  
20 appropriate blends of the polyolefin plastomer or elastomer and other polymer components would still maintain enhanced performance. Some useful polymer blend components include, for example, high pressure low density polyethylene (LDPE), ethylene/vinyl acetate copolymer (EVA), polybutylene (PB), linear high density polyethylene (HDPE) having a  
25 density from 0.941 to 0.965 g/cm<sup>3</sup> and heterogeneously branched linear low density polyethylene (LLDPE) having a density from 0.87 to 0.94 g/cm<sup>3</sup>. Preferably the polyolefin plastomers or elastomers comprise at least about 50 percent of the polymer blend composition, more preferably at least about 80 percent of the polyolefin plastomer or elastomer blend composition.  
30 LDPE, and PP based bitumen bags are known, but have very poor miscibility in the heated bitumen. Nevertheless, small amounts of these materials may be blended with the plastomers/elastomers, but at levels

5 greater than about 20 percent these materials may have a deleterious effect on the solubility of the bags.

Preferably the polyolefin plastomer or elastomer will comprise from 20 to 80 percent by weight of the film, more preferably from 20 to 65 percent, and even more preferably from 35 to 50 percent.

10 The second component in the at least one film layer used in the packages of the present invention is an inorganic filler. The inorganic filler is added in an amount of from 15 to 60 percent by weight of the layer, preferably, 20 to 55 percent by weight of the layer, or 30 to 55 percent by weight, and more preferably from 40 to 50 percent by weight of the layer. It  
15 is believed that any inorganic filler can be used, including materials such as titanium dioxide, talc, silica, barium sulfate, diatomaceous earth, aluminosilicates, carbon black, and calcium carbonate, with calcium carbonate generally being most preferred. Preferably when calcium carbonate is used, it has an average particle size of less than 3 microns with  
20 a top cut of 11 microns.

The second component can be added via any means known in the art. Preferred methods include adding as a masterbatch in a dryblending operation. Alternative methods include compounding (via single screw or twin screw extruder) the inorganic filler into the second  
25 component (resin) to form a compound.

The films used in the present invention may also contain other additives such as plasticizers, antioxidants (for example, hindered phenolics (for example, Irganox<sup>®</sup> 1010 made by Ciba Geigy Corp.), phosphites (for example, Irgafos<sup>®</sup> 168)), cling additives (for example, polyisobutylene  
30 (PIB)), heat stabilizers, pigments, light stabilizers (for example, Cyasorb<sup>™</sup> UV 531 benzophenone made by Cyanamid and Tinuvin<sup>™</sup> 622 hindered amine light stabilizer made by Ciba Geigy Corp.), processing aids (for

5 example, polyethylene glycols, fluoropolymers, fluoroelastomers, waxes),  
flame retardants (for example, Amgard™ CPC 102 phosphorous based  
flame retardants made by Albright and Wilson Americas), lubricants (for  
example, waxes, stearates, mineral oils), slip agents (for example,  
erucamide, oleamide), antiblock additives (for example, silicon dioxide),  
10 cross-linking agents (for example, peroxides, (for example, Booster™ made  
by DuPont)), antifogging agents (for example, Atmer™ 100 sorbitan ester  
made by ICI), impact modifiers (for example, Paxon™ Pax Plus rubber  
modified film resin made by Allied Corp.), antistatic agents (for example,  
Armostat 410 ethoxylated tertiary amine made by Akzo Chemicals, Inc.),  
15 and the like, to the extent that they do not interfere with the function of the  
batch inclusion package. The list of additives is merely illustrative and is  
not all inclusive or limiting.

#### Suitable Films and Film Structures

Films and film structures having the novel properties  
20 described herein can be made using conventional hot blown film or cast  
film fabrication techniques. Conventional hot blown film processes are  
described, for example, in The Encyclopedia of Chemical Technology, Kirk-  
Othmer, Third Edition, John Wiley & Sons, New York, 1981, Vol. 16, pp.  
416-417 and Vol. 18, pp. 191-192, the disclosures of which are incorporated  
25 herein by reference.

The films may be monolayer or multilayer films, but at least  
one layer of the film structure comprises the modified polyolefin elastomer  
or plastomer previously described. Preferably the inner layer will comprise  
the modified polyolefin plastomer or elastomer. The inner layer is the  
30 layer adjacent to the material contained in the package. The inner layer can  
be coextruded with the other layer(s) or the inner layer can be laminated  
onto another layer(s) in a secondary operation, such as that described in

5 "Coextrusion For Barrier Packaging" by W.J. Schrenk and C.R. Finch,  
Society of Plastics Engineers RETEC Proceedings, June 15-17 (1981), pp.  
211-229, the disclosure of which is incorporated herein by reference. A  
monolayer film can also be produced via tubular film (that is, blown film  
techniques) or flat die (that is, cast film) as described by K.R. Osborn and  
10 W.A. Jenkins in "Plastic Films, Technology and Packaging Applications"  
(Technomic Publishing Co., Inc. (1992)), the disclosure of which is  
incorporated herein by reference, and, optionally, the film can go through  
an additional post-extrusion step of adhesive or extrusion lamination to  
other packaging material layers to form a multilayer structure. If the film is  
15 a coextrusion of two or more layers (also described by Osborn and Jenkins),  
the film may still be laminated to additional layers of packaging materials,  
depending on the other physical requirements of the final packaging film.  
"Laminations Vs. Coextrusion" by D. Dumbleton (Converting Magazine  
(September 1992), the disclosure of which is incorporated herein by  
20 reference, also discusses lamination versus coextrusion. Monolayer and  
coextruded films can also go through other post extrusion techniques, such  
as a biaxial orientation process.

Extrusion coating is yet another technique for producing  
multilayer packaging materials. Similarly to cast film, extrusion coating is  
25 a flat die technique. A film layer can be extrusion coated onto a substrate  
either in the form of a monolayer or a coextruded extrudate.

Generally for a polymer blend and/or a multilayer film  
structure, the modified polyolefin plastomer or elastomer described herein  
comprises at least one layer of the total multilayer film structure, preferably  
30 the inner layer. Other layers of the multilayer structure include but are not  
limited to barrier layers, and/or tie layers, and/or structural layers.  
Various materials can be used for these layers, with some of them being

5 used as more than one layer in the same film structure. Some of these materials include: high density polyethylene (HDPE), , ethylene/vinyl acetate (EVA) copolymers, ethylene/acrylic acid (EAA) copolymers, ethylene/methacrylic acid (EMAA) copolymers, ionomers, LLDPE, HDPE, LDPE, polypropylene (including homopolymer, random co polymers and  
10 impact copolymers) and graft adhesive polymers (for example, maleic anhydride grafted polyethylene). Generally, the multilayer film structures comprise from 2 to 7 layers.

The thickness of the film comprising the modified polyolefin plastomer or elastomer (whether as a monolayer film or layer in a  
15 multilayer film structure) is typically from 60 micrometers to 250 micrometers, preferably from 100 micrometers to 225 micrometers, with 150 to 200 micrometers being most preferred.

The films of the present invention can also be characterized in terms of their physical properties. In general the preferred films of the  
20 present invention will have a Peak Load of at least 30 N as measured by ASTM 5747-95 (modified by using a probe diameter of 0.50mm instead of 0.75 mm as specified in the ASTM method), more preferably a Peak Load greater than 45N. The elongation at Peak Load for the preferred films will be greater than 30mm, more preferably greater than 35mm Puncture  
25 resistance for the films can be determined using ASTM D 5748-95 modified by using a probe diameter of 0.50mm instead of 0.75 mm as specified in the ASTM method. The puncture resistance for the preferred films will be greater than 30mm more preferably greater than 35mm. The total energy for the preferred films will be greater than 1.0J more preferably greater  
30 than 1.5J, as determined by ASTM 5748-95. The Dart impact for the films can be determined according to ASTM D 1709-01, method B. The Dart

- 5 impact for the preferred films will be greater than 900g, more preferably greater than 1200g.

#### Suitable Batch Inclusion Structures

The films and film structures made from the modified polyolefin plastomer or elastomer are preferably made into bags either in-  
10 line via the form-fill-seal process or off line in the form of preformed bags (also known as valve bags). The bags of the present invention can be hot filled with the desired product (for example bitumen) above the melting point of the bag, using cooling means such as by suspending the bags in a water bath or through the use of sprayed water. Alternatively chilled air or  
15 other gas could be used to keep the bags from prematurely melting during hot-filling. After filling, the bags are sealed, thereby reducing noxious vapors from the solidifying bitumen.

Use of the modified polyolefin plastomers and elastomers in batch inclusion bags and films affords numerous advantages. The modified  
20 polymers have excellent processability in making blown film and also have low melting points and softening ranges, relative to other materials. The modified plastomers and elastomers are also highly miscible with bitumen allowing them to become fully integrated with the bitumen when used, for example, in asphalt applications. It should be noted that the filler used in  
25 the bag will often also be a desirable additive in the bitumen, thereby offering additional advantages.

#### Experimental: Blown Film Examples

In order to demonstrate the beneficial nature of the present invention a series of films were made and their miscibility with bitumen  
30 evaluated.

Table 1 summarizes the modified polymers used to make films for use in these Examples. When present, the filler was added via a

- 5 masterbatch comprising 60 percent by weight  $\text{CaCO}_3$  in a polymer carrier resin such as LDPE resin.

5 **Table 1**

| Example | Polymer                                                               | Melt Index<br>(g/10 min) | Melt Flow Rate | Density<br>(g/cm <sup>3</sup> ) | I <sub>10</sub> /I <sub>2</sub> | % CaCO <sub>3</sub> |
|---------|-----------------------------------------------------------------------|--------------------------|----------------|---------------------------------|---------------------------------|---------------------|
| 1       | Substantially linear ethylene/1-octene copolymer                      | 1.0                      | NA             | 0.902                           | 9                               | 17%                 |
| 1C      | Substantially linear ethylene/1-octene copolymer                      | 1.0                      | NA             | 0.902                           | 9                               | 0                   |
| 2       | Propylene Based Plastomer or elastomer with 5% ethylene as co-monomer | NA                       | 2.0            | 0.888                           | NA                              | 17%                 |
| 2C      | Propylene Based Plastomer or elastomer with 5% ethylene as co-monomer | NA                       | 2              | 0.888                           | NA                              | 0                   |
| 3C      | Low density polyethylene                                              | 0.22                     | NA             | 0.921                           | NA                              | 17%                 |
| 4       | Substantially linear ethylene/1-octene copolymer                      | 1.1                      | NA             | 0.902                           | NA                              | 17%                 |
| 4C      | Substantially linear ethylene/1-octene copolymer                      | 1.1                      | NA             | 0.902                           | NA                              | 0                   |
| 5       | Ethylene vinyl acetate based polymer (VA content 18%)                 | 1.5                      | NA             | 0.940                           | NA                              | 17%                 |
| 6C      | High melt strength polypropylene                                      | NA                       | 0.5            | 0.900                           | NA                              | 17%                 |

- 5                      Blown film is made from the materials described in Table 1. The films are made as monolayer blown films (A/A/A) on a three layer (A/B/C) coextrusion blown film line (Reifenhauser), with a layer ratio of 1:2:1. The blown film line details are as follows: Die Diameter-250mm; Die Gap-2.3mm; Screw Diameter- A - 60 mm, B -70mm, and C - 60 mm.
- 10    Resins are dry blended with masterbatches via the gravimetric blending stations on the extruders. Processing parameters for various resins and their combination are given below. The films produced have a thickness of 165 microns.

Table 2

|                               | PROCESSING PARAMETERS OF EXTRUDER B |              |              |              |              |              |              |              |              |
|-------------------------------|-------------------------------------|--------------|--------------|--------------|--------------|--------------|--------------|--------------|--------------|
| MATERIAL/<br>EXAMPLE          | 1                                   | 1C           | 2            | 2C           | 3C           | 4            | 4C           | 5            | 6C           |
| Temperature profile in °C     | Set / Actual                        | Set / Actual | Set / Actual | Set / Actual | Set / Actual | Set / Actual | Set / Actual | Set / Actual | Set / Actual |
| Barrel Zone 1.1               | 185/184                             | 185/184      | 150/150      | 150/150      | 180/180      | 175/175      | 175/175      | 155/155      | 180/180      |
| Barrel Zone 1.2               | 185/186                             | 185/186      | 160/161      | 160/162      | 180/181      | 175/175      | 175/175      | 155/155      | 180/180      |
| Barrel Zone 1.3               | 195/195                             | 195/196      | 180/181      | 180/180      | 180/180      | 180/180      | 180/180      | 185/185      | 210/210      |
| Barrel Zone 1.4               | 195/195                             | 195/195      | 220/219      | 220/220      | 220/221      | 180/179      | 180/185      | 195/195      | 220/220      |
| Barrel Zone 2.2               | 195/195                             | 195/195      | 225/226      | 225/226      | 225/227      | 180/180      | 180/181      | 200/200      | 225/225      |
| Barrel Zone 2.3               | 195/197                             | 195/195      | 230/226      | 230/226      | 230/226      | 180/180      | 180/180      | 205/204      | 230/231      |
| Barrel Zone 3.1               | 195/194                             | 195/194      | 230/237      | 230/237      | 230/237      | 180/180      | 180/180      | 205/205      | 230/230      |
| Melt Temperature              | 221                                 | 218          | 225          | 226          | 209          | 207          | 217          | 211          | 230          |
| Die head Zone 3.3             | 210/210                             | 210/210      | 225/221      | 225/220      | 225/220      | 215/215      | 215/215      | 200/199      | 230/228      |
| Die head Zone 3.4             | 210/211                             | 210/211      | 225/223      | 225/223      | 225/223      | 215/215      | 215/215      | 200/200      | 230/230      |
| Die head Zone 3.5             | 210/209                             | 210/209      | 225/216      | 225/216      | 225/216      | 215/215      | 215/216      | 200/198      | 230/227      |
| Die head Zone 3.6             | 210/210                             | 210/210      | 225/220      | 225/220      | 225/220      | 215/215      | 215/215      | 200/201      | 230/229      |
| Die head Zone 3.7             | 205                                 | 206          | 203          | 204          | 204          | 206          | 209          | 206          | 218          |
| Die head Zone 4.11            | 210/210                             | 210/210      | 225/217      | 225/217      | 225/218      | 215/215      | 215/215      | 200/200      | 230/228      |
| Die head Zone 4.12            | 210/208                             | 210/208      | 225/216      | 225/216      | 225/217      | 215/216      | 215/215      | 200/197      | 230/227      |
| Extruder B current, Amps      | 101                                 | 101          | 68           | 67           | 69           | 71           | 68           | 38           | 89           |
| Extruder B Screw speed, rpm   | 44                                  | 44           | 29           | 29           | 30           | 48           | 60           | 41           | 30           |
| Extruder B Melt pressure, bar | 262                                 | 262          | 144          | 145          | 140          | 224          | 226          | 92           | 185          |
| Lay flat width, mm            | 1000                                | 1000         | 1000         | 1000         | 1000         | 1000         | 1000         | 1000         | 1000         |
| Haul off speed, mtrs/ min     | 10                                  | 10           | 6            | 6            | 7            | 7            | 7            | 6            | 7            |

5                    In order to evaluate the miscibility of these films in bitumen, 250 ml of bitumen is added to a beaker and heated using a mantle heater having temperature controller to reach the desired temperatures (150°C, 200°C or 250°C). A sample of each film having the dimensions of 25mm x 25mm is obtained and introduced to the molten bitumen. Temperature is  
10                    monitored with a mercury thermometer to ensure there was no fluctuation before sample was introduced. The film samples are introduced with tweezers slowly into the molten bitumen and a stopwatch is used to monitor the changes over time. At 15 minutes intervals, sample is picked up using tweezers for visual inspection and a qualitative record of the  
15                    amount of material remaining is made. Each time before sample is picked up, the bitumen is gently stirred using a glass rod to ensure a uniform temperature of the molten bitumen. At the end of 1 hour, the testing is stopped and the molten bitumen is sieved to inspect the sample. The quantity of film remaining in the bitumen is not able to be quantified due  
20                    to the contamination with bitumen; therefore this test relied on qualitative visual observation. The result is recorded as 'Miscible' or 'Immiscible'. "Immiscible" means there was visible film remaining on the sieve surface, whereas "miscible" means that there was no visible trace of the film. The resulting observations are presented in Table 3

5    **Table 3**

| Example | 150°C      | 200°C      | 250°C      |
|---------|------------|------------|------------|
| 1       | Miscible   | Miscible   | Miscible   |
| 1C      | Immiscible | Immiscible | Immiscible |
| 2       | Immiscible | Miscible   | Miscible   |
| 2C      | Immiscible | Immiscible | Immiscible |
| 3C      | Immiscible | Immiscible | Immiscible |
| 4       | Miscible   | Miscible   | Miscible   |
| 4C      | Immiscible | Immiscible | Immiscible |
| 5       | Immiscible | Immiscible | Miscible   |
| 6C      | Immiscible | Immiscible | Immiscible |

The results show that the polyolefin elastomers and  
plastomers with the inorganic filler (Examples 1, 2, and 4) are more miscible  
10    in the bitumen.

5

We Claim:

1. A package which comprises:
  - (A) an ingredient;
  - 10 (B) a protective film comprising at least one layer comprising
    - i) at least one polyolefin plastomer or elastomer or segmented ethylene-alpha-olefin block copolymer, and
    - ii) an inorganic filler in an amount of from 15 to 60 percent by weight of the layer,
- 15 wherein the protective film (B) is miscible with the ingredient under conditions in which the ingredient is used.
2. The package of claim 1 wherein the package has been hot filled at a temperature above approximately 100°C.
3. The package of claim 1 wherein the package has been hot filled at a temperature above  
20 approximately 100°C and promptly cooled using a fluid cooling medium.
3. The package of claim 1 wherein the polyolefin plastomer or elastomer is selected from the group consisting of polyethylene plastomers  
25 and elastomers and polypropylene plastomers and elastomers.
4. The package of Claim 4 wherein the polyolefin plastomer or elastomer is selected from the group consisting of substantially linear polyethylene, metallocene linear polyethylenes (mLLDPE) and PBPEs.  
30
5. The package of Claim 1 wherein the inorganic filler is calcium carbonate.

5

6. The package of Claim 6 wherein the calcium carbonate is present in an amount of from 20 to 55 percent by weight of the layer.

7. The package of Claim 7 wherein the calcium carbonate is present in an amount of from 40 to 50 percent by weight of the layer.

8. The package of Claim 1 wherein the ingredient is bitumen, asphalt or modified bitumen or asphalt.

9. A method of incorporating an ingredient into a finished article comprising

(A) filling a package with the ingredient

(B) sealing the package

(C) heating the package to a temperature which exceeds the melting temperature of the package

(D) mixing the melted package with the ingredient

wherein the package comprises i) a polyolefin plastomer or elastomer and ii) an inorganic filler, and

wherein the package is fully miscible with the ingredient.

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10. The method of Claim 10 wherein step A occurs while cooling the package.

11. The method of Claim 11 wherein the package is cooled by immersing the package in a cooling medium.

5                   12. The method of Claim 12 wherein the cooling medium is water.

                  13. The method of Claim 11 wherein the package is cooled by blowing air or other inert gas.

10                   14. The method of Claim 10 wherein the polyolefin plastomer or elastomer is selected from the group consisting of polyethylene plastomers and elastomers and polypropylene plastomers and elastomers.

15                   15. The method of Claim 15 wherein the polyolefin plastomer or elastomer is selected from the group consisting of substantially linear polyethylene, metallocene catalyzed polyethylenes and PBPEs.

                  16. The method of Claim 10 wherein the inorganic filler is  
20 calcium carbonate.

                  17. The method of Claim 17 wherein the calcium carbonate is present in an amount of from 20 to 55 percent by weight of the layer.

25                   18. The method of Claim 18 wherein the calcium carbonate is present in an amount of from 40 to 50 percent by weight of the layer.

                  19. The method of Claim 10 wherein the ingredient is bitumen, asphalt or modified bitumen or asphalt.

30                   20. The method of Claim 10 wherein step (A) occurs at a temperature greater than or equal to 100°C.

# INTERNATIONAL SEARCH REPORT

International application No

PCT/US2008/060769

## A. CLASSIFICATION OF SUBJECT MATTER

INV. B65D65/38

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)

B65D

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 practical, search terms used)

EPO-Internal

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☒ Further documents are listed in the continuation of Box C.

☒ See patent family annex.

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\*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

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\*8\* document member of the same patent family

Date of the actual completion of the international search

18 July 2008

Date of mailing of the international search report

29/07/2008

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# INTERNATIONAL SEARCH REPORT

International application No

PCT/US2008/060769

C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

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Information on patent family members

International application No

PCT/US2008/060769

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