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(54) Title: MOULDED ARTICLE FROM POLYPROPYLENE COMPOSITION

(57) Abstract: There is provided herein a polymer blend composition, that comprises a polymeric component comprising a random polypropylene copolymer or terpolymer having a Tm of from 120°C to 145°C and an elastomeric polymer having a density of from 0.850 to 0.900 g/cm^3; and an organic peroxide, where the organic peroxide is present in an amount of from 0.001 to 1 parts by weight, per 100 parts by weight of the polymeric component (e.g. from 0.005 to 0.5 parts by weight per 100 parts by weight of the polymeric component); the melt flow rate of the polymer blend composition is from 8 to 40 g/10 minutes (e.g. 15 to 39 g/10 minutes); and the falling weight impact of the blend at 23°C after moulding into a 1.4 mm sheet is from 4 to 30 J. Also provided herein is a method to make said polymer composition and a kit of parts suitable for the manufacture of said composition.

Moulded article from Polypropylene Composition

FIELD OF INVENTION

This invention relates to moulded articles with enhanced impact strength prepared using a blended polymer, where the blended polymer comprises a random polypropylene copolymer and/or a random polypropylene terpolymer, an organic peroxide and an elastomeric polymer. This invention also relates to the blended polymer.

BACKGROUND

The listing or discussion of a prior-published document in this specification should not necessarily be taken as an acknowledgement that the document is part of the state of the art or is common general knowledge.

Polypropylene copolymers, either random copolymers or random terpolymers, are widely used in many applications. However, polypropylene copolymers are not suitable for use in all applications without further modification. For example, a moulded article made using polypropylene has lower impact strength than would normally be desired in certain applications, such as transparent containers. In order to enhance the impact strength of the article, the polypropylene copolymer can be blended with an elastomer, such as an ethylene-based elastomer (though other elastomers may be used). The resulting article has greater impact strength, making it more suited to the applications envisaged.

In addition, it is possible to add a range of additive materials to a polypropylene copolymer to improve additional properties (said additives may be combined with the blending of the polypropylene copolymer with an elastomer to improve the properties of the polypropylene copolymer further). For example, nucleating agents can be added to enhance mechanical strength and/or a clarifying agent (e.g. a sorbitol) may be added to reduce haze in moulded articles. In certain cases, the clarifying agent may also increase the stiffness (or flexural modulus) of the resulting polymer composition too.

In order to obtain a transparent, moulded product with good transparency, the preference has been to use a single phase random copolymer or random terpolymer of polypropylene. Polypropylene heterophasic copolymers comprise a polymer matrix with a dispersed polymer phase (e.g. a copolymer, ethylene, propylene or any C_4-C_{10} α-olefin phase). Said

heterophasic polymer blends may be produced in the reactor during polymerisation, in the extruder or during dry blending. When such heterophasic polymer blends are used, the transparency of the moulded article will be significantly affected.

To obtain an impact-resistant polypropylene composition that retains transparency upon moulding, while keeping other desirable properties is quite difficult. For example, the inclusion of an elastomeric polymer results in increased haze and reduced stiffness (or flexural modulus) in the moulded article, which are not desired for certain applications (e.g. for syringes and transparent containers).

For certain applications, moulded articles made from polypropylene are subjected to sterilisation using radiation. For example, gamma ray radiation generated from a cobalt 60 source, or electron beam radiation generated from an electron gun at high voltage. These sterilisation techniques are commonly used in health care industries to provide sterilised articles for use in a clinical setting. Examples of articles subjected to radiation sterilisation include syringes and cups or containers designed to contain any form of liquid and solids that must be kept in a sterile environment. However, the use of radiation as a sterilisation method causes significant degradation of the polymeric materials, resulting in the loss of mechanical properties due to change in molecular weight. In addition, this degradation may also cause a change in the colour/transparency of the moulded article as well. It has been noted that the level of degradation in objects made comprising polypropylene following exposure to radiation is higher than objects made using most other plastic materials. Therefore, given that polypropylene provides many desirable properties, there is still need for compositions of polypropylene that retain mechanical strength after irradiation.

An attempt to provide such a material has been disclosed in Japanese patent application No. 2003-72730A, which described the formation of a polypropylene random copolymer or terpolymer blend with an elastomeric material. This appears to provide materials with a good odour and improved impact strength. Although the blending of an elastomeric polymer with a polypropylene random copolymer or terpolymer improves the impact strength, but the cost of the resulting material is expensive and there remain issues with the clarity and stiffness of the resulting material. Therefore, there remains a need to find new polypropylene materials with enhanced impact strength that can be used to form moulded articles that are intended to be subjected to irradiation.

SUMMARY OF INVENTION

This invention provides polypropylene compositions that contain a random polypropylene copolymer or terpolymer in combination with an organic peroxide and an elastomeric polymer that show increased falling weight impact strength compared to polypropylene compositions where the organic peroxide is absent.

In a first aspect of the invention, there is provided a polymer blend composition, comprising:

(a) a polymeric component comprising a random polypropylene copolymer or terpolymer having a Tm of from 120°C to 145°C and an elastomeric polymer having a density of from 0.850 to 0.900 g/cm^3; and

(b) an organic peroxide, wherein:

the organic peroxide is present in an amount of from 0.001 to 1.0 parts by weight, per 100 parts by weight of the polymeric component (e.g. from 0.005 to 0.5 parts by weight per 100 parts by weight of the polymeric component);

the melt flow rate of the polymer blend composition is from 2 to 40 g/10 minutes (e.g. 8 to 39 g/10 minutes); and

the falling weight impact of the blend at 23°C after moulding into a 1.4 mm sheet is from 4 to 30 J.

In an embodiment of the invention, the random polypropylene copolymer or terpolymer may have a Tm of from 129°C to 144°C (e.g. from 135°C to 144°C).

In a further embodiment of the invention, the random polypropylene copolymer may comprise ethylene or a C_4-C_{10} α-Olefin as a comonomer, or the random polypropylene terpolymer comprises both ethylene and a C_4-C_{10} α-Olefin as comonomers (e.g. the random polypropylene copolymer or terpolymer is a random polypropylene copolymer containing ethylene).

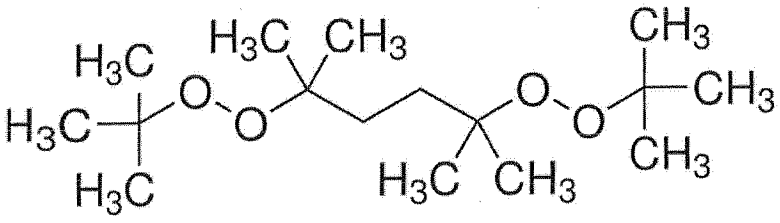
In certain embodiments of the invention, the polymer blend composition may have a melt flow rate of from 20 to 35 g/10 minutes (e.g. 25 to 30 g/10 minutes). Additionally or alternatively, the melt flow rate of the polymer blend composition may be from 8 to 40 g/10 minutes (e.g. from 8 to 39 g/10 minutes or from 10 to 40 g/10 minutes, such as 15 to 39 g/10 minutes, more particularly of from 20 to 35 g/10 minutes such as from 25 to 35 g/10 minutes).

In yet further embodiment of the invention, the elastomeric polymer may have a density of from 0.860 to 0.899 g/cm^3, such as a density of from 0.870 to 0.899 g/cm^3 (e.g. from 0.880 to 0.899 g/cm^3 or from 0.890 to 0.899 g/cm^3).

In still further embodiments, the elastomeric polymer may be a copolymer comprising ethylene and an α-olefin as comonomers (e.g. ethylene and a C_4-C_{10} α-olefin as comonomers). For example, the elastomeric copolymer may comprise more than 55 wt% ethylene (e.g. from 55 to 90 wt% (such as from 55 to 85 wt%) ethylene, with the remainder being an α-olefin).

In yet a still further embodiment, the random polypropylene copolymer or terpolymer may be present in an amount of from 85 wt% to 98 wt% of the polymeric component in the polymer blend composition, and the elastomeric polymer may be present in an amount of from 2 wt% to 15 wt% of the polymeric component in the polymer blend composition (e.g. the random polypropylene copolymer or terpolymer is present in an amount of from 90 wt% to 98 wt% of the polymeric component and the elastomeric polymer is present in an amount of from 2 wt% to 10 wt% of the polymeric component).

In certain embodiments, the organic peroxide may be selected from the group consisting of hydroperoxides, dialkyl peroxides, diacyl peroxides, peroxydicarbonates, peroxyesters, ketone peroxides, peroxyketals, and alkyl peroxy carbonates. In particular embodiments, the organic peroxide may be a dialkyl peroxide, for example the dialkyl peroxide may be 2,5-bis(*tert*-butylperoxy)-2,5-dimethylhexane:



In still further embodiments, the polymer blend may further comprise a neutralising agent. When present, the neutralising agent may be present in an amount of from 0.001 to 1 parts by weight per 100 parts by weight of the polymeric component (e.g. from 0.01 to 0.10 parts by weight, such as 0.05 parts by weight per 100 parts by weight of the polymeric component). The neutralising agent may be a metal salt of a fatty acid and/or a carbonate

mineral (e.g. the neutralising agent may be a metal stearate, such as calcium stearate and/or hydrotalcite).

In yet still further embodiments, the polymer blend may further comprise an anti-oxidant (when present, the anti-oxidant may be present in an amount of from 0.001 to 1 parts by weight per 100 parts by weight of the polymeric component (e.g. from 0.05 to 0.20, such as from 0.10 to 0.15 parts by weight per 100 parts by weight of the polymeric component)). The anti-oxidant may be a phosphite and/or phosphate. For example, the anti-oxidant may be a tris-(2,4-ditert-butyl phenyl) phosphate and/or bis-(2,4-ditert-butyl phenyl) pentaerythritol diphosphite.

In yet still further embodiments, the polymer blend may further comprise a light stabiliser (when present, the light stabiliser may be present in an amount of from 0.001 to 1 parts by weight per 100 parts by weight of the polymeric component (e.g. from 0.05 to 0.15, such as 0.1 parts by weight per 100 parts by weight of the polymeric component)). The light stabiliser may be selected from the group consisting of benzophenones, benzotriazoles, hindered amines, and inorganic light stabilizers. For example, the light stabiliser may be poly-[1-(2'–hydroxyethyl)-2,2,6,6-tetramethyl-4-hydroxy–piperidyl succinate].

In yet still further embodiments, the polymer blend may further comprise an α-nucleating agent (when present, the α-nucleating agent may be present in an amount of from 0.001 to 1 parts by weight per 100 parts by weight of the polymeric component (e.g. from 0.15 to 0.25 parts by weight per 100 parts by weight of the polymeric component)). For example, the α-nucleating agent may be a salt of benzoic acid (e.g. a sodium salt), bis-(T-tert butyl-benozate) aluminium hydroxide, a sorbitol and/or a phosphate ester or a salt/solvate thereof. In certain embodiments, the α-nucleating agent may be one or more selected from the group consisting of the sodium salt of benzoic acid, bis-(T-tert butyl-benozate) aluminium hydroxide, 1,3:2,4-bis-(3,4-dimethyl benzylidene) sorbitol, or, more particularly, 1,2,3-trideoxy-4,6:5,7-bis-O-[(4-propylphenyl)methylene]-nonitol and the sodium salt of 2,4,8,10-Tetra(tert-butyl)-6-hydroxy-12H-dibenzo[d,g][1,3,2]dioxaphosphocin-6-oxide.

In yet further embodiments, the polymer blend where the falling weight impact of the blend at 23°C after moulding into a 1.4 mm sheet may be from 4 to 30 J (e.g. from 6.5 to 25 J, such as from 9.0 to 15.0 J). Additionally or alternatively, the polymer blend where the decrease in the falling weight impact of the blend at 23°C after moulding into a 1.4 mm sheet may be less than 25% following exposure to electron beam radiation at 57 KGy.

In yet further embodiments, the haze of the polymer blend composition may be less than 20% (e.g. from 10% to 18%), optionally wherein the elastomeric polymer is present in an amount of from 2 wt% to 5 wt% of the polymeric component.

In certain embodiments of the invention that may be mentioned herein, the polymer blend composition does not contain an inorganic filler material. In yet further embodiments of the invention that may be mentioned herein, the polymer blend composition does not contain any inorganic material.

In a second aspect of the invention, there is provided a process for preparing a polymer blend composition as described in the first aspect of the invention or any of the embodiments thereof.

In a third aspect of the invention, there is provided a moulded article comprising a polymer blend composition as described in the first aspect of the invention or any of the embodiments thereof. In certain embodiments, the moulded article may have been subjected to irradiation.

In a fourth aspect of the invention, there is provided a process for preparing a moulded article comprising a polymer blend composition as described in the first aspect of the invention or any of the embodiments thereof. In certain embodiments, the process may further comprise the step of irradiating the moulded article.

In a fifth aspect of the invention, there is provided a kit of parts suitable for preparing the polymer blend composition according to the first aspect of the invention or any of the embodiments thereof, said kit comprising:

(a) a random polypropylene copolymer or terpolymer having a Tm of from 120°C to 145°C (e.g. from 135°C to 144°C);

(b) an elastomeric polymer having a density of from 0.850 to 0.900 g/cm^3 (e.g. 0.860 to 0.899 g/cm^3, such as from 0.870 to 0.899 g/cm^3 (e.g. from 0.880 to 0.899 g/cm^3 or from 0.890 to 0.899 g/cm^3)); and

(c) an organic peroxide.

The fifth aspect of the invention may comprise component parts described in the first aspect of the invention and embodiments thereof.

DESCRIPTION

When used herein, the terms "comprises" and "comprising" and equivalents thereof are intended to be non-limiting in nature. Said terms are intended to encompass the terms "consists essentially of" and "consists of" and equivalents thereof. It is intended that the terms "comprises" and "comprising" and equivalents thereof may be replaced by the terms "consists essentially of" and "consists of" and equivalents thereof.

The applicants have surprisingly found that a formulation containing a polypropylene copolymer blend and an organic peroxide has been found to solve the problems identified above. That is, there is provided a polymer blend composition that comprises:

(a) a polymeric component comprising a random polypropylene copolymer or terpolymer having a Tm of from 120°C to 145°C and an elastomeric polymer having a density of from 0.850 to 0.900 g/cm^3; and

(b) an organic peroxide,

wherein the organic peroxide is present in an amount of from 0.001 to 1 parts by weight, per 100 parts by weight of the polymeric component (e.g. from 0.005 to 0.5 parts by weight per 100 parts by weight of the polymeric component),the melt flow rate of the polymer blend composition is from 2 to 40 g/10 minutes (e.g. 8 to 39 g/10 minutes), and the falling weight impact of the blend at 23°C after moulding into a 1.4 mm sheet is from 4 to 30 J.

The random polypropylene copolymer or terpolymer may have a Tm (i.e. melting point or crystalline melting temperature) of from 120°C to 145°C. In certain embodiments, the Tm may be from 129°C to 144°C, such as from 135°C to 144°C. A method for measuring the Tm is provided in the examples section below.

In embodiments mentioned herein, the random polypropylene copolymer may comprise ethylene or a C_4-C_{10} α-Olefin as a comonomer. In alternative embodiments, the random polypropylene terpolymer may comprise both ethylene and a C_4-C_{10} α-Olefin as comonomers. In certain embodiments, the random polypropylene copolymer or terpolymer that may be mentioned herein is a random polypropylene copolymer containing ethylene.

The random polypropylene copolymer or terpolymer may be present in an amount of from 85 wt% to 98 wt% of the polymeric component and the elastomeric polymer may be present in an amount of from 2 wt% to 15 wt% of the polymeric component (e.g. the random

polypropylene copolymer or terpolymer may be present in an amount of from 90 wt% to 98 wt% of the polymeric component and the elastomeric polymer may be present in an amount of from 2 wt% to 10 wt% of the polymeric component, such as from 2 wt% to 5 wt%). In particular embodiments, the random polypropylene copolymer or terpolymer may be present in an amount of from 95 wt% to 98 wt% of the polymeric component and the elastomeric polymer may be present in an amount of from 2 wt% to 5 wt% of the polymeric component.

When used herein the term "elastomeric polymer" refers to a polymer or polymer blends that may be amorphous above its glass transition temperature (Tg), allowing for flexibility, deformability and that can recover its original state to a large degree following deformation. In certain embodiments, the elastomeric polymers may be formed with cross-linked polymer chains, wherein the cross-linkages enable an elastomeric polymer object to recover all or substantially all of its original configuration/shape when an applied stress is removed from the object instead of being permanently deformed.

The elastomeric polymer may be a low density polyethylene-based material (e.g. a polyethylene polymer). In certain embodiments, the elastomeric polymer may have a density of from 0.850 to 0.900 g/cm^3, for example a density of from 0.860 to 0.899 g/cm^3, or a density of from 0.870 to 0.899 g/cm^3 (e.g. from 0.880 to 0.899 g/cm^3 or from 0.890 to 0.899 g/cm^3). Particular embodiments that may be mentioned herein include an elastomeric polymer having a density of from 0.895 to 0.899 g/cm^3. In certain embodiments, the elastomeric polymer may be a copolymer comprising ethylene and an α-olefin as comonomers (e.g. the copolymer may comprise ethylene and a C_4-C_{10} α-olefin as comonomers).

When used herein, the C_4-C_{10} α-Olefin comonomers may include, but are not limited to, any suitable C_4-C_{10} α-Olefin, such as 1-butene, 1-pentene, 1-hexene, 1-heptene, 1-octene, 1-nonene, 1-decene or any suitable derivatives thereof.

When the elastomeric polymer is a copolymer (e.g. a copolymer of ethylene and an α-olefin), the copolymer may comprise more than 55 wt% ethylene, such as more than 65 wt%, more than 70 wt% ethylene, such as 75 wt% ethylene. For example, the elastomeric copolymer may comprise more than 55 wt% ethylene (e.g. from 55 to 90 wt% (such as from 55 to 85 wt%) ethylene, with the remainder being an α-olefin). Elastomeric polymers that may be mentioned herein are Excellen™ EUL731-M, Excellen™ FX CX4008 and ENGAGE™ 8407.

The polymer blend composition may have a melt flow rate of from 2 to 40 g/10 minutes, such as from 8 to 39 g/10 minutes, more particularly of from 20 to 35 g/10 minutes such as from 25 to 35 g/10 minutes. Additionally or alternatively, the melt flow rate of the polymer blend composition may be from 8 to 40 g/10 minutes (e.g. from 8 to 39 g/10 minutes or from 10 to 40 g/10 minutes, such as from 15 to 39 g/10 minutes, more particularly of from 20 to 35 g/10 minutes such as from 25 to 35 g/10 minutes). A method for measuring the melt flow rate is provided in the examples section below. Alternatively, if there is not enough sample for experimental melt flow rate (i.e. melt index) determinations or if it is necessary to determine melt flow rate of fractions of a polymer blend composition, an alternative molecular weight measurement, such as gel permeation chromatography can be used together with know correlations between molecular weight and melt flow rate to determine the melt flow rate for the polymer blend composition. For an example, see A. Giijsels, Ind. Polym. Process, 9, 252 (1994).

The organic peroxide may be present in an amount of from 0.001 to 1.0 parts by weight, per 100 parts by weight of the polymeric component such as from 0.005 to 0.50 parts by weight per 100 parts by weight of the polymeric component. In certain embodiments, the organic peroxide may be present in an amount of from 0.010 to 0.10 parts by weight, per 100 parts by weight of the polymeric component such as from 0.015 to 0.080 parts by weight, per 100 parts by weight of the polymeric component (e.g. from 0.020 to 0.040 parts by weight).

The organic peroxide may be selected from one or more of the group consisting of hydroperoxides, dialkyl peroxides, diacyl peroxides, peroxymonocarbonates, peroxydicarbonates, peroxyesters, ketone peroxides, peroxyketals, and alkyl peroxy carbonates.

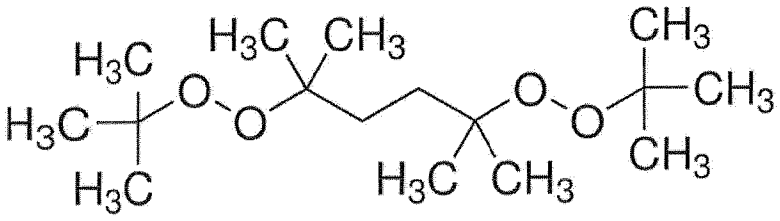
Hydroperoxides that may be mentioned herein include, but are not limited to, p-menthane hydroperoxide.

Peroxymonocarbonates that may be mentioned herein include, but are not limited to, tert-hexylperoxy isopropyl monocarbonate.

Peroxyesters that may be mentioned herein include, but are not limited to, tert-butylperoxy-3,5,5-trimethyl hexanoate, tert-butyl peroxy laurate, tert-butylperoxyacetate and tert-butylperoxybenzoate.

Peroxyketals that may be mentioned herein include, but are not limited to, n-butyl-4,4-bis(tert-peroxy)valerate, 1,1-bis(tert-butylperoxy)cyclohexane, 2,2-bis(4,4-di-tert-butylperoxy)cyclohexyl)propane, 1,1-bis(tert-butylperoxy)cyclododecane and di-tert-butylperoxyisophthalate.

Diakyl peroxides that may be mentioned herein include, but are not limited to, 2,5-dimethyl-2,5-di-(benzoylperoxy)hexane, 2,2-bis(tert-butylperoxy)butane, tert-butyl cumyl peroxide, di-tert-butyl peroxide, dicumyl peroxide, α-α'-bis(tert-butylperoxy-m-isopropyl)benzene, 1,3-bis(tert-butylperoxydiisopropyl)benzene, 2,5-bis(tert-butylperoxy)-2,5-dimethylhex-3-yne, 2-methyl-2-[(2-methyl-2-propanyl)peroxy]propane, 2-methyl-2-[(2-methyl-2-butanyl)peroxy]butane, {2-[(2-methyl-2-propanyl)peroxy]-2-propanyl}benzene, 1,3-bis{1-[(2-methyl-2-propanyl)peroxy]-2-propanyl}benzene, 2,5-dimethyl-2,5-bis[(2-methyl-2-propanyl)peroxy]hexane, 1,1'-(dioxydi-2,2-propanediyl)dibenzene, and 2,5-bis(*tert*-butylperoxy)-2,5-dimethylhexane. A particular dialkyl peroxide that may be mentioned herein is 2,5-bis(*tert*-butylperoxy)-2,5-dimethylhexane, also represented by the chemical structure shown below.



In certain embodiments, the organic peroxide may include Trigonox™101-20PP and/or Luperox™ 101 PP20.

While not wishing to be bound by theory, it is believed that the organic peroxide may act as chain transfer agent to influence the molecular viscosity and/or melt flow rate of the polymer blend composition. In addition, the molecular weight distribution of the polymer blend composition is narrowed when an organic peroxide is added to the composition, as compared to other conventional chain transfer agents such as hydrogen.

The polymer blend composition may also include additives such as, but not limited to, neutralising agents, anti-oxidants, light stabilisers, α-nucleating agents or any combination thereof.

Particular neutralising agents that may be mentioned herein include a metal salt of a fatty acid and/or a carbonate mineral. In certain embodiments, the neutralising agent may be any metal stearate such as zinc stearate, sodium stearate, calcium stearate, magnesium stearate or any combination thereof. For example, the neutralising agent may be calcium stearate. In other embodiments, the neutralising agent may include a carbonate mineral such as hydrotalcite (e.g. magnesium aluminium hydroxy carbonate). When present in the composition, the neutralising agent may be present in an amount of from 0.001 to 1 parts by weight per 100 parts by weight of the polymeric component (e.g. from 0.01 to 0.10 parts by weight, such as 0.05 parts by weight per 100 parts by weight of the polymeric component).

Anti-oxidants that may be mentioned herein includes any suitable phosphite and/or phosphate. For example, the anti-oxidant may include tris-(2,4-ditert-butyl phenyl) phosphate, bis(2,4-di-t-butyl phenyl) pentaerythritol diphosphite, even more particularly Irgafos™ 168. When present in the composition, the anti-oxidant may be present in an amount of from 0.001 to 1 parts by weight per 100 parts by weight of the polymeric component (e.g. from 0.05 to 0.20, such as from 0.10 to 0.15 parts by weight per 100 parts by weight of the polymeric component).

Light stabilisers that may be mentioned herein include benzophenones, benzotriazoles, hindered amine light stabilisers, and inorganic light stabilizers, or any combination thereof. For example, the light stabiliser may include hindered amine light stabilisers such as poly-[1-(2'–hydroxyethyl)-2,2,6,6-tetramethyl-4-hydroxy–piperidyl succinate], or, more particularly, Tinuvin™ 622 LD. When present in the composition, the light stabiliser may be present in an amount of from 0.001 to 1 parts by weight per 100 parts by weight of the polymeric component (e.g. from 0.05 to 0.15, such as 0.1 parts by weight per 100 parts by weight of the polymeric component).

α-Nucleating agents that may be mentioned herein include, but are not limited to one or more of a salt of benzoic acid (e.g. a sodium salt), bis-(T-tert butyl-benozate) aluminium hydroxide, a sorbitol and a phosphate ester or a salt/solvate thereof. For example, the α-nucleating agent may be one or more selected from the group consisting of the sodium salt of benzoic acid, bis-(T-tert butyl-benozate) aluminium hydroxide, 1,3:2,4-bis-(3,4-dimethyl benzylidene) sorbitol, 1,2,3-trideoxy-4,6:5,7-bis-O-[(4-propylphenyl)methylene]-nonitol and the sodium salt of 2,4,8,10-Tetra(tert-butyl)-6-hydroxy-12H-dibenzo[d,g][1,3,2]-dioxaphosphocin-6-oxide. Additionally or alternatively, the α-nucleating agent may include Millad™ 3988i, Millad™ NX8000J, ADK STAB NA-21 or any combination thereof. When

present in the composition, the α-nucleating agent may be present in an amount of from 0.001 to 1 parts by weight per 100 parts by weight of the polymeric component (e.g. from 0.15 to 0.25 parts by weight per 100 parts by weight of the polymeric component).

In certain embodiments of the invention that may be mentioned herein, the polymer blend composition does not contain an inorganic filler material. When used herein "filler material" refers to any material that can be used to increase the bulk of a composition, without reacting with any of the other components within the composition. In yet further embodiments of the invention that may be mentioned herein, the polymer blend composition does not contain any inorganic material. When used herein "inorganic" refers to a chemical compound that does not contain any material that can be termed "organic", that is a compound that comprises carbon and hydrogen atoms, optionally in combination with one or more nitrogen, sulfur, oxygen and phosphorous atoms or salts thereof (e.g. metal salts thereof).

The random polypropylene copolymers or terpolymers present in the polymeric component (including those presented in Table 1 below) may be produced using a conventional polymerization technique and a conventional catalyst.

Examples of the conventional catalyst include:

(1) a catalyst comprising a solid catalyst component obtained by reacting a magnesium compound with a titanium compound, and an organoaluminium compound;

(2) a catalyst comprising a solid catalyst component obtained by reacting a magnesium compound with a titanium compound, an organoaluminium compound, and, optionally, a third component such as an electron donating compound; and

(3) a metallocene based catalyst.

In certain embodiments, catalysts of type (2) are used. Catalysts of type (2) may comprise a solid catalyst component comprising magnesium, titanium, and halogen (e.g. $TiCl_4$, $MgCl_2$) as essential components (i.e. a heterogeneous Ziegler–Natta catalyst suitable for use in the polymerisation of propylene), an organoaluminium compound (e.g. $Al(C_2H_5)_3$), and an electron donating compound may optionally be used. Examples of this type of catalyst include the catalysts disclosed in JP 61-218606 A, JP 61-287904 A, or JP 7-216017 A.

Examples of conventional polymerization include:

(a) slurry polymerization and solvent polymerization, each using an inactive hydrocarbon solvent;

(b) liquid phase polymerization using a monomer as a solvent without using any inactive hydrocarbon solvent,

(c) gas phase polymerization; and

(d) liquid phase-gas phase polymerization, in which liquid phase polymerization and gas phase polymerization are conducted continuously.

In the production of the random polypropylene copolymer or terpolymer, a random polypropylene copolymer or terpolymer formed as a result of polymerization may be heated under reduced pressure at a temperature lower than the temperature at which the polypropylene melts in order to remove the remaining solvent and oligomers generated as by-products of the polymerisation process. Examples of the method of heating under reduced pressure include the methods of drying under reduced pressure disclosed in JP 55-75410 A and JP 2-80433 A.

Random polypropylene copolymers or terpolymers may be combined with any suitable elastomeric polymer to form the polymeric component as described herein. The polymeric component may then be used to produce the polymer blend compositions, as described herein. The polymer blend compositions may be provided as, but not limited to, pellets, granules or sheets.

The formation of polymer blend compositions can be performed using any suitable technique including, but not limited to, blending the desired components (e.g. polymeric component, organic peroxide and/or additives) in the desired proportions using conventional blending techniques and apparatus, including high speed mixers from Mitsui Mike Machinery Co, Banbury mixer (available from Farrel Corp., Ansonia, Conn.) or laboratory extruders, such as the Polylab Twin Screw Extruder (available from Thermo Electron (Karsruhe), Karsruhe, Germany) which are suitable for preparing small batches of material. The mixing apparatus used may include any suitable tank capacity and/or processing capacity, for example, the mixing apparatus may include a tank capacity of 75 litres and a process capacity of 50 litres.

The polymer blend compositions may be prepared using other types of mixing equipment capable of premixing and directly feeding materials into downstream processing apparatus. Such downstream processing apparatus may include, but is not limited to, an extruder or any

suitable polymer manufacturing equipment to produce polymer blend compositions as pellet samples.

When producing the polymer blend compositions, the desired components may be premixed using a high speed mixer to provide "premixed material". The premixing may be performed for from 10 seconds to 3600 seconds or more specifically, from 20 seconds 1800 seconds, such as 30 seconds.

The premixing of desired components may be performed at a speed of from 500 to 3000 rpm, such as from 750 to 2500 rpm or more particularly from 820 rpm to 1640 rpm.

The premixed material may be converted into polymer blend composition pellets using other kinds of equipment capable of melting, mixing and extruding the polymer blend compositions. Extruded polymer blend compositions may then be converted into polymer blend composition pellet and/or granule samples using any suitable equipment, for example an underwater pellet cutter or pellet maker. Commercial scale pelletizing extruders may also be used for preparing larger quantities of the blend.

The polymer blend compositions disclosed herein may be used to prepare various kinds of moulded articles suitable for different applications. For example, the polymer blend compositions may be used to prepare moulded articles including, but not limited to, automotive components, housings/covers for electrical appliances and packaging for consumer goods. Particular moulded articles that may be mentioned herein relate to articles for use in the healthcare and medical fields (e.g. containers and syringes).

The moulded articles may be prepared from the polymer blend compositions by any suitable moulding process. Examples of suitable moulding processes include injection moulding, compression moulding, extrusion and extrusion compression moulding. The moulding process may include, but is not limited to, a step of feeding the desired polymer blend composition into any suitable moulding apparatus. The polymer blend composition may be in the form of a polymer blend composition pellet and/or granule samples.

The moulding process may also include a step of heating the polymer blend composition in a heating barrel/chamber to produce a polypropylene polymer melt. The step of heating the polymer blend composition to produce a polypropylene polymer melt may be performed at a

temperature of from 150°C to 250°C, more particularly, from 175 to 240°C such as from 190 to 235°C.

The polypropylene polymer melt may then be fed into a mould (also known as a die) having one or more cavities of any desired shape. The polypropylene polymer melt located within the mould may be solidified using any suitable conditions to produce a moulded article. For example, the polypropylene polymer melt may be solidified by cooling with any suitable fluid (e.g. water or air) to produce the moulded article. The polypropylene polymer melt may also be subjected to high pressures to produce the moulded article. These high pressures may be applied during the step of heating the polymer blend composition in the heating barrel/chamber. Cooling of the injected polymer melt may be performed by subjecting the article to a temperature of from 10°C to 50°C under the clamped force of the injection moulding machine.

The moulded article may then be removed from the mould before being optionally subjected to further processing or conditioning. For example, the article may be placed in a separated container such that it does not contact any other plastic materials (e.g. a box of tray made from paper or cardboard) and subjected to irradiation.

EXAMPLES

Materials used

Unless otherwise stated, materials were obtained from commercial sources and used without further purification.

2,5-Bis(*tert*-butylperoxy)-2,5-dimethylhexane (an organic peroxide) was obtained as Luperox™ 101 PP20 from Arkema or as Trigonox 101-20PP from Akzo Nobel. Both materials listed provide the organic peroxide mixed with a carrier material (polypropylene) and in a concentration of from 10 wt% to 30 wt%. Unless otherwise stated herein, the peroxide is provided at a concentration of 20 wt%.

Excellen™ EUL731-M (an elastomeric polymer; a copolymer of ethylene and 1-butene) was obtained from Sumitomo Chemical Co Ltd, Japan. Excellen™ EUL731-M has a melt flow rate of 10 g/10 mins measured at 190°C and a load of 2.16 kg. Excellen™ EUL731-M has a density of 895 kg /m^3 (0.895 g/cm^3).

Excellen™ FX CX4008 (an elastomeric polymer; a copolymer of ethylene and 1-hexene) was obtained from Sumitomo Chemical Co Ltd Japan. Excellen ™ FX CX4008 has a melt flow rate of 8 g/10 mins measured at 190°C and a load of 2.16 kg. Excellen ™ FX CX4008 has a density of 880 kg/m^3 (0.880 g/cm^3).

ENGAGE™ 8407 (an elastomeric polymer; a copolymer of ethylene and 1-octene) was obtained from The Dow Chemical Company. ENGAGE ™ 8407 has a melt flow rate of 30 g/10 mins measured at 190°C and a load of 2.16 kg. ENGAGE ™ 8407 has a density of 870 kg/m^3 (0.870 g/cm^3).

Calcium stearate, obtained from FACI, was used as a neutralising agent. Alternatively, DHT-4C (Hydrotalcite; Magnesium Aluminium Hydroxy Carbonate) from Kyowa Chemical Industry Co Ltd was used as the neutralising agent instead.

Tris-(2,4-ditert-butyl phenyl) phosphate (an anti-oxidant) was obtained from BASF as Irgafos™ 168.

Poly-[1-(2'–hydroxyethyl)-2,2,6,6-tetramethyl-4-hydroxy–piperidyl succinate] (a hindered amine light stabilising agent) was obtained from BASF as Tinuvin™ 622 LD.

1,3:2,4-Bis-(3,4-dimethyl benzylidene) sorbitol (an α-Nucleating Agent) was obtained as Millad™ 3988i from Milliken Chemicals. 1,2,3-Trideoxy-4,6:5,7-bis-O-[(4-propylphenyl)methylene]-nonitol (an α-Nucleating Agent) was obtained as Millad™ NX8000J from Milliken Chemicals. 2,4,8,10-Tetra(tert-butyl)-6-hydroxy-12H-dibenzo[d,g][1,3,2]dioxaphosphocin-6-oxide, sodium salt (an α-Nucleating Agent) was obtained as ADK STAB NA-21 from ADEKA Corporation.

Preparation 1

Polypropylene homopolymer, and Random polyproyplene copolymers and terpolymers

The homopolymers, random copolymers and terpolymers described herein are prepared using a Ziegler–Natta catalyst, using $Al(C_2H_5)_3$ as a co-catalyst using the liquid phase-gas phase polymerisation technique. Examples of suitable catalysts are disclosed in JP 61-218606 A, JP 61-287904 A, or JP 7-216017 A.

The polypropylenes were formed as a result of polymerization are heated under reduced pressure at a temperature lower than the temperature at which the polypropylene melts in order to remove the remaining solvent and oligomers generated as by-products of the polymerisation process. Examples of the method used to make the current polypropylenes are disclosed in JP 55-75410 A and JP 2-80433 A. The polypropylenes prepared are listed in Table 1.

Polypropylene (PP) No.	**Polymer Type**	**Comonomer (content wt%)**	**Tm (°C)**	**Melt Flow Rate (MFR)**	**CXS (%wt)**
PP1	Copolymer	ethylene (4.0%)	137	7	5.8
PP2	Copolymer	ethylene (2.7%)	144	7	2.7
PP3	Copolymer	ethylene (2.5%)	146	24	2.2
PP4	Copolymer	ethylene (3.1%)	143	7	3.0
PP5	Copolymer	ethylene (3.8%)	138	6	5.7
PP6	Copolymer	ethylene (2.2%)	146	25	2.2
PP7	Terpolymer	ethylene (0.7%); 1-butene (9.1%)	139	7	1.1

Polypropylene (PP) No.	Polymer Type	Comonomer (content wt%)	Tm (°C)	Melt Flow Rate (MFR)	CXS (%wt)
PP8	Terpolymer	ethylene (1.8%); 1-butene (8.9%)	129	6	4.1
PP9	Copolymer	ethylene (2.7%)	143	6	2.7
PP10	Copolymer	ethylene (1.6%)	151	19	2.5
PP11	Homo-polymer	-	158	10	2.2

Table 1

Preparation 2

The compositions were prepared using the amounts provided in Table 2.

Example	Composition
1	(PP4 (98 wt% of polymer component) and EUL731-M (2 wt% of polymer component)) (100 parts by weight (pbw)) Trigonox 101-20PP (0.17 pbw per 100 pbw of polymer component; i.e. 0.034 pbw of peroxide per 100 pbw of polymer component) Calcium Stearate (0.05 pbw per 100 pbw of polymer component) Irgafos 168 (0.15 pbw per 100 pbw of polymer component) Tinuvin 622 LD (0.10 pbw per 100 pbw of polymer component) Millad 3988i (0.20 pbw per 100 pbw of polymer component)

Example	Composition
2	(PP2 (95 wt% of polymer component) and EUL731-M (5 wt% of polymer component)) (100 pbw) Trigonox 101-20PP (0.18 pbw per 100 pbw of polymer component; i.e. 0.036 pbw of peroxide per 100 pbw of polymer component) Calcium Stearate (0.05 pbw per 100 pbw of polymer component) Irgafos 168 (0.10 pbw per 100 pbw of polymer component) Tinuvin 622 LD (0.10 pbw per 100 pbw of polymer component) Millad NX8000J (0.22 pbw per 100 pbw of polymer component)
3	(PP1 (95 wt% of polymer component) and EUL731-M (5 wt% of polymer component)) (100 pbw) Trigonox 101-20PP (0.17 pbw per 100 pbw of polymer component; i.e. 0.034 pbw of peroxide per 100 pbw of polymer component) Calcium Stearate (0.05 pbw per 100 pbw of polymer component) Irgafos 168 (0.10 pbw per 100 pbw of polymer component) Tinuvin 622 LD (0.10 pbw per 100 pbw of polymer component) Millad NX8000J (0.22 pbw per 100 pbw of polymer component)

Example	Composition
4	(PP9 (98 wt% of polymer component) and EUL731-M (2 wt% of polymer component)) (100 pbw) Trigonox 101-20PP (0.20 pbw per 100 pbw of polymer component; i.e. 0.040 pbw of peroxide per 100 pbw of polymer component) Calcium Stearate (0.05 pbw per 100 pbw of polymer component) Irgafos 168 (0.15 pbw per 100 pbw of polymer component) Tinuvin 622 LD (0.10 pbw per 100 pbw of polymer component) Millad 3988i (0.20 pbw per 100 pbw of polymer component)
5	(PP5 (98 wt% of polymer component) and EUL731-M (2 wt% of polymer component)) (100 pbw) Trigonox 101-20PP (0.20 pbw per 100 pbw of polymer component; i.e. 0.040 pbw of peroxide per 100 pbw of polymer component) Calcium Stearate (0.05 pbw per 100 pbw of polymer component) Irgafos 168 (0.15 pbw per 100 pbw of polymer component) Tinuvin 622 LD (0.10 pbw per 100 pbw of polymer component) Millad 3988i (0.20 pbw per 100 pbw of polymer component)
6	(PP7 (98 wt% of polymer component) and EUL731-M (2wt% of polymer component) (100pbw)) Trigonox 101-20PP (0.18 pbw per 100 pbw of polymer component; i.e. 0.036 pbw of peroxide per 100 pbw of polymer component) Calcium Stearate (0.05 pbw per 100 pbw of polymer component) Irgafos 168 (0.15 pbw per 100 pbw of polymer component) Tinuvin 622 LD (0.10 pbw per 100 pbw of polymer component) Millad 3988i (0.20 pbw per 100 pbw of polymer component)

Example	Composition
7	(PP8 (98 wt% of polymer component) and EUL731-M (2 wt% of polymer component) (100pbw)) Trigonox 101-20PP (0.17 pbw per 100 pbw of polymer component; i.e. 0.034 pbw of peroxide per 100 pbw of polymer component) Calcium Stearate (0.05 pbw per 100 pbw of polymer component) Irgafos 168 (0.15 pbw per 100 pbw of polymer component) Tinuvin 622 LD (0.10 pbw per 100 pbw of polymer component) Millad 3988i (0.20 pbw per 100 pbw of polymer component)
8	(PP4 (98 wt% of polymer component) and ENGAGE8407 (2 wt% of polymer component) (100pbw)) Trigonox 101-20PP (0.18 pbw per 100 pbw of polymer component; i.e. 0.036 pbw of peroxide per 100 pbw of polymer component) Calcium Stearate (0.05 pbw per 100 pbw of polymer component) Irgafos 168 (0.15 pbw per 100 pbw of polymer component) Tinuvin 622 LD (0.10 pbw per 100 pbw of polymer component) Millad 3988i (0.20 pbw per 100 pbw of polymer component)
9	(PP4 (98 wt% of polymer component) and FX CX4008 (2 wt% of polymer component) (100pbw)) Trigonox 101-20PP (0.18 pbw per 100 pbw of polymer component; i.e. 0.036 pbw of peroxide per 100 pbw of polymer component) Calcium Stearate (0.05 pbw per 100 pbw of polymer component) Irgafos 168 (0.15 pbw per 100 pbw of polymer component) Tinuvin 622 LD (0.10 pbw per 100 pbw of polymer component) Millad 3988i (0.20 pbw per 100 pbw of polymer component)

Example	Composition
Comparative Example 1	(PP3 (98 wt% of polymer component) and EUL731-M (2 wt% of polymer component)) (100 pbw) DHT-4C (Neutraliser) (0.05 pbw per 100 pbw of polymer component) Irgafos 168 (0.10 pbw per 100 pbw of polymer component) Tinuvin 622 LD (0.10 pbw per 100 pbw of polymer component) NA-21 (Nucleating Agent) (0.20 pbw per 100 pbw of polymer component)
Comparative Example 2	(PP3 (95 wt% of polymer component) and EUL731-M (5 wt% of polymer component)) (100 pbw) Calcium Stearate (0.05 pbw per 100 pbw of polymer component) Irgafos 168 (0.10 pbw per 100 pbw of polymer component) Tinuvin 622 LD (0.10 pbw per 100 pbw of polymer component) Millad NX8000J (0.22 pbw per 100 pbw of polymer component)
Comparative Example 3	(PP3 (90 wt% of polymer component) and EUL731-M (10 wt% of polymer component)) (100 pbw) Calcium Stearate (0.05 pbw per 100 pbw of polymer component) Irgafos 168 (0.10 pbw per 100 pbw of polymer component) Tinuvin 622 LD (0.10 pbw per 100 pbw of polymer component) Millad NX8000J (0.22 pbw per 100 pbw of polymer component)
Comparative Example 4	(PP6 (98 wt%) and EUL731-M (2 wt%)) (100 pbw) Calcium Stearate (0.05 pbw per 100 pbw of polymer component) Irgafos 168 (0.15 pbw per 100 pbw of polymer component) Tinuvin 622 LD (0.10 pbw per 100 pbw of polymer component) Millad 3988i (0.20 pbw per 100 pbw of polymer component)

Example	Composition
Comparative Example 5	(PP6 (95 wt%) and EUL731-M (5 wt%)) (100 pbw) Calcium Stearate (0.05 pbw per 100 pbw of polymer component) Irgafos 168 (0.15 pbw per 100 pbw of polymer component) Tinuvin 622 LD (0.10 pbw per 100 pbw of polymer component) Millad 3988i (0.20 pbw per 100 pbw of polymer component)
Comparative Example 6	(PP10 (100 wt%)) (100 pbw) Calcium Stearate (0.05 pbw per 100 pbw of polymer component) Irgafos 168 (0.15 pbw per 100 pbw of polymer component) Tinuvin 622 LD (0.10 pbw per 100 pbw of polymer component) Millad 3988i (0.20 pbw per 100 pbw of polymer component)
Comparative Example 7	(PP10 (98 wt%) and EUL731-M (2 wt%)) (100 pbw) Calcium Stearate (0.05 pbw per 100 pbw of polymer component) Irgafos 168 (0.15 pbw per 100 pbw of polymer component) Tinuvin 622 LD (0.10 pbw per 100 pbw of polymer component) Millad 3988i (0.20 pbw per 100 pbw of polymer component)
Comparative Example 8	(PP11 (98 wt%) and EUL731-M (2 wt%)) (100 pbw) Trigonox 101-20PP (0.18 pbw per 100 pbw of polymer component; i.e. 0.036 pbw of peroxide per 100 pbw of polymer component) Calcium Stearate (0.05 pbw per 100 pbw of polymer component) Irgafos 168 (0.15 pbw per 100 pbw of polymer component) Tinuvin 622 LD (0.10 pbw per 100 pbw of polymer component) Millad 3988i (0.20 pbw per 100 pbw of polymer component)

Example	Composition
Comparative Example 9	(PP11 (100 wt% of polymer component) (100pbw)) Trigonox 101-20PP (0.17 pbw per 100 pbw of polymer component; i.e. 0.034 pbw of peroxide per 100 pbw of polymer component) Calcium Stearate (0.05 pbw per 100 pbw of polymer component) Irgafos 168 (0.15 pbw per 100 pbw of polymer component) Tinuvin 622 LD (0.10 pbw per 100 pbw of polymer component) Millad 3988i (0.20 pbw per 100 pbw of polymer component)
Comparative Example 10	(PP4 (100 wt%)) (100 pbw) Trigonox 101-20PP (0.17 pbw per 100 pbw of polymer component; i.e. 0.034 pbw of peroxide per 100 pbw of polymer component) Calcium Stearate (0.05 pbw per 100 pbw of polymer component) Irgafos 168 (0.15 pbw per 100 pbw of polymer component) Tinuvin 622 LD (0.10 pbw per 100 pbw of polymer component) Millad 3988i (0.20 pbw per 100 pbw of polymer component)

Table 2

a) Pellet manufacture

All of the ingredients listed in Table 2 were dry-premixed using a high speed mixer for about 30 seconds at a speed of from 820 to 1640 rpm. The high speed mixer is manufactured by Mitsui Mike Machinery Co., having a tank capacity of 75 L and a process capacity of 50 L.

The premixed material was then fed into an extruder to produce pellet samples. A single screw extruder was used (manufactured by Tanabe Plastic Machinery Co.), having the following parameters:

(a) a screw diameter of 65mm;

(b) L/D of 32;

(c) Die hole size of 4mm; and

(d) extruder temperature of between 200 -210°C (inclusive).

Following extrusion the polymer blend were cut into pellets.

b) Sheet manufacture

The pellets produced in step (a) were converted into a sheet for property measurement by an injection moulding process, wherein the pellets are provided to an injection moulding machine (Model SH100C from Sumitomo Heavy Industries, having a screw diameter of 32 mm) and are formed into a sheet having 1.4 mm thickness through, using a barrel temperature of between 190 to 235°C (inclusive). The resulting sheets were conditioned for at least 12 hours at 23°C and 50% RH before analysis.

Analysis

Melt Flow Rate

Determined according to ASTM D1238 – Standard Test Method for Melt Flow Rates of Thermoplastics by Extrusion Plastometer.

Machine: Toyoseiki Melt Indexer F-B01
Temperature: 230°C
Load: 2.16 kg
Conditioning Time: 6 minutes
Orifice Dimension: 2.095 mm inner diameter, 8.0 mm length

Density Testing for Elastomeric Polymer

Samples for density testing were prepared by compression moulding. Before subjecting the specimens to compression, the specimen was pre-heated to 150°C and was then subjected to bumping to remove any gas bubbles. Each specimen had the dimensions of 30 mm x 25 mm x 1mm and was subjected to a moulding temperature of 150°C at a moulding pressure of 50 kg/cm^3 for 5 minutes using Tester Sangyo Co. Ltd, Model: SA-303. The specimen was then subjected to a cooling temperature of 23°C and a cooling pressure of 20 kg/cm^3 for 3 minutes using Tester Sangyo Co. Ltd, Model: SA-302. Subsequently, the specimen was annealed at 100°C for 1 hour in distilled water, after which the specimen was conditioned in a standard laboratory atmosphere (23°C, 50% relative humidity) for 16 hours.

The density of the specimens produced by the procedure above were then tested using ASTM D792 – standard test methods for density and specific gravity (relative density) of plastics by displacement, using Test Method A – for testing solid plastics in water.

The tests were conducted using equipment from Ohaus Corporation, Model: DV215D (Balance) & 77402-00 (Density Kit).

Melting Temperature (Tm)

Polymer sample was first compressed into sheet of 0.3 mm or 0.5 mm thickness by using a compressing moulding machine.

Moulding temperature: 230°C
Preheating Period: 180 seconds
Pressure – kg/cm^2/ Moulding time –minutes: I-50/60, II- 50/30, III-50/30
Cooling Temperature: 180 seconds
Cooling pressure: 20 kg/cm^2

The moulded sheet or film sample was punched into small circular pieces and a sample of 10.000±0.1 mg was obtained on an accurate mass balance.

The melting temperature was measured using a Differential Scanning Calorimeter, where the weighed sample of circular pieces were cur into smaller pieces that better fit into the aluminium sample pan of the equipment.

The sample was annealed by rapidly heating it up to 220°C, which temperature was then held for 5 minutes and then the sample was cooled down to 65°C. The sample was then heated from 65°C to 220°C at 20°C/min. The endothermic peak temperature recorded at this step is the Melting Temperature, as reported herein.

Instrument: Perkin Elmer Diamond DSC

Sheet Haze

Circular specimens having a 50 mm diameter were obtained from the sheets obtained in Preparation 2(b) using a circular hand-punch.

Haze is measured using a direct-reading haze meter. The measurement and computation principle are given in ASTM D1003. The haze meter is calibrated without the presence of a specimen and the calibration is checked with a haze meter.

Calculation of Haze in percentage terms is as follows: Haze $= T_d / T_t X100$

T_t – total transmittance
T_d – diffuse transmittance

Ethylene content analysis

The polymer samples were compressed into sheet of 0.3 mm thickness using a compressing moulding machine using the parameters below.

Moulding temperature: 230°C
Preheating Period: 180 seconds
Pressure – kg/cm^2/ Moulding time –minutes: I-50/60, II- 50/30, III-50/30
Cooling Temperature: 180 seconds
Cooling pressure: 20 kg/cm^2

Ethylene content (wt%) for the compressed sheet was measured using an IR spectrum measurement method described in the Polymer Analysis Hand Book (issued by Asakura Publishing Co. Ltd., 1985) on page 256.

1-Butene content analysis

The polymer samples were compressed into sheet of 0.3 mm thickness using a compressing moulding machine using the parameters below.

Moulding temperature: 230°C
Preheating Period: 180 seconds

Pressure – kg/cm^2/ Moulding time –minutes: I-50/60, II- 50/30, III-50/30
Cooling Temperature: 180 seconds
Cooling pressure: 20 kg/cm^2

1-butene content was measured using the IR spectrum measurement method described in Polymer Analysis Handbook (published by Kinokuniya Co., Ltd., 1995) on page 619.

The content of components soluble in 20°C xylene (CXS, unit: % by weight)

A sample of 1 g was dissolved completely in 100 mL of boiling xylene, and then cooled to 20°C and left at rest for 4 hours. Subsequently, the resultant mixture was separated into precipitates and a solution by filtration, and the filtrate was dried at 70°C under reduced pressure, affording a residue. The residue was weighed and the content of components soluble in 20°C xylene (henceforth called CXS) was calculated.

Falling Weight Impact (FWI) Strength

Circular specimens of 65 mm diameter with a thickness of 1.4 mm were directly obtained by injecting the materials of Preparation 2(a) into a circular disc mould in an injection moulding machine having a barrel temperature of 190-235°C. The mould was a 2 cavity type mould.

The circular specimens were conditioned for at least 12 hours at 23°C and 50% RH prior to testing the FWI strength.

Equipment: Injection Moulding Machine Model SH100C from Sumitomo Heavy Industries
Screw diameter: 32 mm

The apparatus that is used to test Falling Weight Impact strength is called a Falling Weight Impact tester or Du Pont Type Impact Tester.

Specifications

Falling height: 25 to 1800mm
Falling Weight: 1000g , 2000g, 3000g
Impact head (dart): ½"
Specimen holder: 66 Ø X38Ø mm hold
Dimension of the specimens used: 65 Ø X 1.4 mm thickness

Specimen Clamping system: Pneumatic clamping system

Procedure in brief:

Each test specimen was placed in a specimen holder and clamped to the falling weight apparatus. The impact head hits the specimen perpendicularly when the load is released. The load rests in a pillar containing a release pin, when the pin is released, the load falls onto the specimen in the specimen holder, the resulting impact may (or may not) break the specimen, which is determined in the manner outlined below.

The purpose of the test is to obtain three data points having an approximate breakage rate of 10-30%, 40-60% and 70-90%, respectively for each material. Any minor or major opening in the specimen resulting from impact is treated as a breakage. Both brittle and ductile failures are also considered to be a breakage.

In order to ensure that a suitable load/weight (kg) and height (cm) were selected for testing, four specimens were initially used. If all four specimens broke or did not break, then the height was reduced or increased, respectively and the process repeated until it appeared that the desired breakage rate had been reached. When the height reached a maximum or minimum of the apparatus limit, then an appropriate higher or lower load was selected respectively and the process repeated.

Once the height and weight to achieve the desired breakage rate have been identified by iterative means, 16 specimens were tested using the same weight and height for to calculate the breakage percentage using the formula:

Breakage percentage = (Number of broken specimen/Number of tested specimen) X100

The resulting data represents one data point for the material at one of the breakage ranged of 10-30%, 40-60% and 70-90%.

By plotting the impact energy (kg.cm) versus the corresponding breakage percentage, the impact energy at 0% failure is then extrapolated using a best-fit straight line plot and reported as the falling weight impact strength in Joules for the tested material.

Electron Beam Irradiation

The specimens were irradiated by JISCo (Japan Irradiation Service Co) Japan. The specimens were subjected to cyclical exposure to the electron beam to achieve a desired radiation dose of 57 KGy.

Apparatus: Cockcroft-Walton type Electron Beam Processing System manufactured by NHV Corporation.

Energy of Electron Beam: 5 MeV

Temperature during radiation: about 23°C

Results

The results obtained from falling weight impact, before and after irradiation (for selected samples) are provided in Table 3.

Example No.	Falling Weight Impact 1.4 mm sheet at 23°C (J)		Sheet Haze 1.4 mm sheet at 23°C (%)	
	No Irradiation	After EB Irradiation at 57 KGy	No Irradiation	After EB Irradiation at 57 KGy
1	9.7	7.6	15	17
2	10.0	-	-	-
3	12.0	-	-	-
4	11.6	-	-	-
5	13.4	-	-	-
6	13.9	-	-	-
7	15.2	-	-	-
8	13.0	-	-	-
9	12.5	-	-	-
Comp. 1	4.5	1	22	23
Comp. 2	5.4	-	-	-
Comp. 3	5.4	-	-	-

Example No.	Falling Weight Impact 1.4 mm sheet at 23°C (J)		Sheet Haze 1.4 mm sheet at 23°C (%)	
	No Irradiation	After EB Irradiation at 57 KGy	No Irradiation	After EB Irradiation at 57 KGy
Comp. 4	5.1	-	-	-
Comp. 5	6.3	-	-	-
Comp. 6	1.0	-	-	-
Comp. 7	5.3	-	-	-
Comp. 8	5.5	-	21	-
Comp. 9	1.0	-	16	-
Comp. 10	6.1	-	10	-

Table 3

Example 1 shows that the moulded material shows a falling weight impact that is approximately double that achieved in any of Comparative Examples 1-3. In addition, the composition of Example 1 retains its strength even after being subjected to electron beam (EB) irradiation. This is not the case for Comparative Example 1, where the composition's strength is severely weakened.

As can also be seen, the compositions of Examples 2 and 3 result in a falling weight impact that is slightly higher than Example 1, due to the inclusion of a greater amount of elastomeric polymer. In contrast, Comparative Example 10 shows that removing the elastomeric polymer from the composition significantly reduces the falling weight impact value. However, as can be seen by comparison to Comparative Examples 2 and 3, the increase attributed to the compositions of the invention remains almost double that achieved in conventional formulations containing a higher percentage of elastomeric material, showing that the combination of the components is essential to achieving the desired effects of increased falling weight impact, both before and after irradiation.

The melt flow rates of the compositions are provided in Table 4.

Example No.	Melt Flow Rate (g/10 mins)
1	30[a]
2	30[a]
3	25[a]
4	28[a]
5	30[a]
6	30[a]
7	26[a]
8	32[a]
9	29[a]
Comp. 1	25[b]
Comp. 2	25[b]
Comp. 3	25[b]
Comp. 4	25[b]
Comp. 5	24[b]
Comp. 6	23[b]
Comp. 7	18[b]
Comp. 8	34[a]
Comp. 9	32[a]
Comp. 10	30[a]

a - composition MFR measured after addition of organic peroxide.

b - composition does not contain organic peroxide.

Table 4

CLAIMS

1. A polymer blend composition, comprising:

(a) a polymeric component comprising a random polypropylene copolymer or terpolymer having a Tm of from 120°C to 145°C and an elastomeric polymer having a density of from 0.850 to 0.900 g/cm^3; and

(b) an organic peroxide, wherein:

the organic peroxide is present in an amount of from 0.001 to 1 parts by weight, per 100 parts by weight of the polymeric component (e.g. from 0.005 to 0.5 parts by weight per 100 parts by weight of the polymeric component);

the melt flow rate of the polymer blend composition is from 8 to 40 g/10 minutes (e.g. 15 to 39 g/10 minutes); and

the falling weight impact of the blend at 23°C after moulding into a 1.4 mm sheet is from 4 to 30 J.

2. The polymer blend composition of Claim 1, wherein the random polypropylene copolymer or terpolymer has a Tm of from 129°C to 144°C.

3. The polymer blend composition of any one of the preceding claims, wherein the random polypropylene copolymer comprises ethylene or a C_4-C_{10} α-Olefin as a comonomer, or the random polypropylene terpolymer comprises both ethylene and a C_4-C_{10} α-Olefin as comonomers (e.g. the random polypropylene copolymer or terpolymer is a random polypropylene copolymer containing ethylene).

4. The polymer blend composition of any one of the preceding claims, wherein the polymer blend composition has a melt flow rate of from 20 to 35 g/10 minutes (e.g. 25 to 32 g/10 minutes).

5. The polymer blend composition of any one of the preceding claims, wherein the elastomeric polymer has a density of from 0.860 to 0.899 g/cm^3.

6. The polymer blend composition of Claim 5, wherein the elastomeric polymer has a density of from 0.870 to 0.899 g/cm^3.

7. The polymer blend composition of any one of the preceding claims, wherein the elastomeric polymer is a copolymer of ethylene and an α-olefin, optionally wherein the α-olefin is a C_4-C_{10} α-olefin.

8. The polymer blend composition of Claim 7, wherein the elastomeric polymer comprises more than 55 wt% ethylene.

9. The polymer blend composition of any one of the preceding claims wherein:

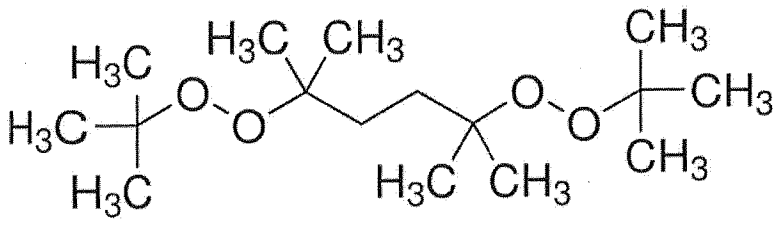
the random polypropylene copolymer or terpolymer is present in an amount of from 85 wt% to 98 wt% of the polymeric component; and

the elastomeric polymer is present in an amount of from 2 wt% to 15 wt% of the polymeric component (e.g. the random polypropylene copolymer or terpolymer is present in an amount of from 90 wt% to 98 wt% of the polymeric component and the elastomeric polymer is present in an amount of from 2 wt% to 10 wt% of the polymeric component).

10. The polymer blend composition of any one of the preceding claims, wherein the organic peroxide is selected from the group consisting of hydroperoxides, dialkyl peroxides, diacyl peroxides, peroxydicarbonates, peroxyesters, ketone peroxides, peroxyketals, and alkyl peroxy carbonates.

11. The polymer blend composition of Claim 10, wherein the organic peroxide is a dialkyl peroxide, optionally wherein the dialkyl peroxide is selected from on eor more of the group consisting of 2,5-dimethyl-2,5-di-(benzoylperoxy)hexane, 2,2-bis(tert-butylperoxy)butane, tert-butyl cumyl peroxide, di-tert-butyl peroxide, dicumyl peroxide, α-α'-bis(tert-butylperoxy-m-isopropyl)benzene, 1,3-bis(tert-butylperoxydiisopropyl)benzene, 2,5-bis(tert-butylperoxy)-2,5-dimethylhex-3-yne, 2-methyl-2-[(2-methyl-2-propanyl)peroxy]propane, 2-methyl-2-[(2-methyl-2-butanyl)peroxy]butane, {2-[(2-methyl-2-propanyl)peroxy]-2-propanyl}benzene, 1,3-bis{1-[(2-methyl-2-propanyl)peroxy]-2-propanyl}benzene, 2,5-dimethyl-2,5-bis[(2-methyl-2-propanyl)peroxy]hexane, 1,1'-(dioxydi-2,2-propanediyl)dibenzene, and 2,5-bis(*tert*-butylperoxy)-2,5-dimethylhexane.

12. The polymer blend composition of Claim 11, wherein the dialkyl peroxide is 2,5-bis(*tert*-butylperoxy)-2,5-dimethylhexane:



13. The polymer blend composition of any one of the preceding claims, wherein the composition further comprises one or more of the group selected from:

(i) a neutralising agent;

(ii) an anti-oxidant;

(iii) a light stabiliser; and

(iv) an α-Nucleating Agent.

14. The polymer blend composition of Claim 13, wherein, when present:

(i) the neutralising agent is present in an amount of from 0.001 to 1 parts by weight per 100 parts by weight of the polymeric component (e.g. from 0.01 to 0.10 parts by weight, such as 0.05 parts by weight per 100 parts by weight of the polymeric component); and/or

(ii) the anti-oxidant is present in an amount of from 0.001 to 1 parts by weight per 100 parts by weight of the polymeric component (e.g. from 0.05 to 0.20, such as from 0.10 to 0.15 parts by weight per 100 parts by weight of the polymeric component); and/or

(iii) the light stabiliser is present in an amount of from 0.001 to 1 parts by weight per 100 parts by weight of the polymeric component (e.g. from 0.05 to 0.15, such as 0.1 parts by weight per 100 parts by weight of the polymeric component); and/or

(iv) the α-Nucleating Agent is present in an amount of from 0.001 to 1 parts by weight per 100 parts by weight of the polymeric component (e.g. from 0.15 to 0.25 parts by weight per 100 parts by weight of the polymeric component).

15. The polymer blend composition of Claim 13 or Claim 14, wherein:

(i) the neutralising agent is a metal salt of a fatty acid and/or a carbonate mineral; and/or

(ii) the anti-oxidant is a phosphite and/or phosphate; and/or

(iii) the light stabiliser is one or more of the group consisting of benzophenones, benzotriazoles, hindered amine light stabilisers, and inorganic light stabilizers; and/or

(iv) the α-Nucleating Agent is one or more of the group consisting of a metal salt of benzoic acid, bis-(T-tert butyl-benozate) aluminium hydroxide, a sorbitol, and a phosphate ester or a salt/solvate thereof.

16. The polymer blend composition of Claim 15, wherein:

(i) the neutralising agent is calcium stearate and/or hydrotalcite; and/or

(ii) the anti-oxidant is bis(2,4-di-t-butyl phenyl) pentaerythritol diphosphite or tris-(2,4-ditert-butyl phenyl) phosphate; and/or

(iii) the light stabiliser is a hindered amine light stabiliser (e.g. poly-[1-(2'–hydroxyethyl)-2,2,6,6-tetramethyl-4-hydroxy–piperidyl succinate]); and/or

(iv) the α-Nucleating Agent is one or more selected from the group consisting of the sodium salt of benzoic acid, bis-(T-tert butyl-benozate) aluminium hydroxide, 1,3:2,4-bis-(3,4-dimethyl benzylidene) sorbitol, 1,2,3-trideoxy-4,6:5,7-bis-O-[(4-propylphenyl)methylene]-nonitol and the sodium salt of 2,4,8,10-Tetra(tert-butyl)-6-hydroxy-12H-dibenzo[d,g][1,3,2]dioxaphosphocin-6-oxide.

17. The polymer blend composition according to any one of the preceding claims, wherein the falling weight impact of the blend at 23°C after moulding into a 1.4 mm sheet is from from 6.5 to 25.0 J (e.g. from 9.0 to 15.0 J).

18. The polymer blend composition according to any one of the preceding claims, wherein the decrease in the falling weight impact of the blend at 23°C after moulding into a 1.4 mm sheet is less than 25% following exposure to electron beam radiation at 57 KGy.

19. A process for preparing a polymer blend composition as described in any one of Claims 1 to 18.

20. A moulded article comprising a polymer blend composition as described in any one of Claims 1 to 18.

21. The moulded article according to Claim 21, wherein the moulded article has been subjected to irradiation.

22. A process for preparing a moulded article comprising a polymer blend composition as described in any one of Claims 1 to 18.

23. The process of Claim 22, further comprising the step of irradiating the moulded article.

24. A kit of parts suitable for preparing the polymer blend composition of any one of Claims 1 to 18, said kit comprising:

(a) a random polypropylene copolymer or terpolymer having a Tm of from 120°C to 145°C (e.g. from 135°C to 144°C);

(b) an elastomeric polymer having a density of from 0.850 to 0.900 g/cm^3 (e.g. 0.860 to 0.899 g/cm^3, such as from 0.890 to 0.899 g/cm^3); and

(c) an organic peroxide.

25. The kit of parts of Claim 24, wherein the random polypropylene copolymer comprises ethylene or a C_4-C_{10} α-Olefin as a comonomer, or the random polypropylene terpolymer comprises both ethylene and a C_4-C_{10} α-olefin as comonomers (e.g. the random polypropylene copolymer is a random polypropylene copolymer or terpolymer containing ethylene).

26. The kit of any one of Claim 24 to Claim 25, wherein the elastomeric polymer is a copolymer of ethylene and an α-olefin, optionally wherein the α-olefin is a C_4-C_{10} α-olefin.

27. The kit of any one of Claims 24 to 26, wherein the elastomeric polymer comprises more than 55 wt% ethylene.

28. The kit of any one of Claims 24 to 27, wherein the organic peroxide is selected from the group consisting of hydroperoxides, dialkyl peroxides, diacyl peroxides, peroxydicarbonates, peroxyesters, ketone peroxides, peroxyketals, and alkyl peroxy carbonates (e.g. the organic peroxide is a dialkyl peroxide, such as 2,5-bis(*tert*-butylperoxy)-2,5-dimethylhexane).

29. The kit of any one of Claims 24 to 28, further comprising one or more of the group selected from:

(i) a neutralising agent;

(ii) an anti-oxidant;

(iii) a light stabiliser; and

(iv) an α-Nucleating Agent.

30. The kit of Claim 29, wherein:

(i) the neutralising agent is a metal salt of a fatty acid and/or a carbonate mineral; and/or

(ii) the anti-oxidant is a phosphite and/or phosphate ; and/or

(iii) the light stabiliser is one or more of the group consisting of benzophenones, benzotriazoles, hindered amine light stabilisers, and inorganic light stabilizers; and/or

(iv) the α-Nucleating Agent is one or more of the group consisting of a metal salt of benzoic acid, bis-(T-tert butyl-benozate) aluminium hydroxide, a sorbitol, and a phosphate ester or a salt/solvate thereof.

31. The kit of Claim 30, wherein:

(i) the neutralising agent is calcium stearate and/or hydrotalcite; and/or

(ii) the anti-oxidant is bis(2,4-di-t-butyl phenyl) pentaerythritol diphosphite or tris-(2,4-ditert-butyl phenyl) phosphate; and/or

(iii) the light stabiliser is a hindered amine light stabiliser (e.g. poly-[1-(2'–hydroxyethyl)-2,2,6,6-tetramethyl-4-hydroxy–piperidyl succinate]); and/or

(iv) the α-Nucleating Agent is one or more selected from the group consisting of the sodium salt of benzoic acid, bis-(T-tert butyl-benozate) aluminium hydroxide, 1,3:2,4-bis-(3,4-dimethyl benzylidene) sorbitol, 1,2,3-trideoxy-4,6:5,7-bis-O-[(4-propylphenyl)methylene]-nonitol and the sodium salt of 2,4,8,10-Tetra(tert-butyl)-6-hydroxy-12H-dibenzo[d,g][1,3,2]dioxaphosphocin-6-oxide.

INTERNATIONAL SEARCH REPORT

International application No.
PCT/SG2016/050262

A. CLASSIFICATION OF SUBJECT MATTER

C08L 23/16 (2006.01) C08L 23/08 (2006.01) C08L 23/14 (2006.01) C08K 5/14 (2006.01)

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)

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)

EPODOC; WPIAP; PATENTSCOPE; USPTO PATENTS; USPTO APPLICATIONS; ESPACENET; GOOGLE SCHOLAR; GOOGLE; CAPLUS; IFIALL; RAPRA; WPIDS; Keywords: RANDOM COPOLYMER POLYPROPYLENE; ETHYLENE PROPYLENE COPOLYMER; ETHYLENE PROPYLENE DIENE RUBBER; VERY LOW DENSITY POLYETHYLENE; PEROXIDE synonyms and associated terms and their combinations. In addition, applicant and inventor name searches were conducted in PATENTSCOPE, USPTO PATENTS, USPTO APPLICATIONS, ESPACENET, GOOGLE & GOOGLE SCHOLAR, and in internal databases provided by IP Australia.

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
	Documents are listed in the continuation of Box C	

[X] Further documents are listed in the continuation of Box C [X] See patent family annex

* Special categories of cited documents:

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Date of the actual completion of the international search	Date of mailing of the international search report
28 September 2016	28 September 2016
Name and mailing address of the ISA/AU	**Authorised officer**
AUSTRALIAN PATENT OFFICE PO BOX 200, WODEN ACT 2606, AUSTRALIA Email address: pct@ipaustralia.gov.au	Benjamin Silva AUSTRALIAN PATENT OFFICE (ISO 9001 Quality Certified Service) Telephone No. +61 3 9935 9611

INTERNATIONAL SEARCH REPORT C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT		International application No. **PCT/SG2016/050262**
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
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X	US 2004/0185288 A1 (PENG) 23 September 2004 Abstract; paragraphs 0021, 0024, 0026, 0032-0033, 0044-0045, 0047 & 0053; Table 1	1-8, 10-20, 22 & 24-31
X	PAUL, S. et al., "Impact Modification of Polypropylene Copolymer with a Polyolefinic Elastomer", Journal of Applied Polymer Science, 2000, vol. 76, pages 1480-1484 Abstract; page 1481: Materials; page 1481: Tables I & II	1-12, 17 & 19-28
X	US 5998524 A (SRINIVASAN et al.) 07 December 1999 column 2, line 23 to column 3, line 8; column 3, lines 51-54; column 4, lines 41-46; column 6, lines 56-65; column 7, lines 4-13 & 34-48; column 8, lines 41-58; column 12, lines 48-57	1-31
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A	Total 7620Z Polypropylene, Injection Molding Grade. Retrieved on 23/09/2016 from the internet, <URL: http://www.matweb.com/search/datasheettext.aspx?matguid=9c7ac815adf5499f9bd9e1816db471eb> (ISA was unable to establish the publication date) Whole Document	1-31
A	Total 7622MZ Polypropylene, Injection Molding Grade. Retrieved on 23/09/2016 from the internet, <URL: http:// http://www.matweb.com/search/datasheettext.aspx?matguid=824def3861214a7ba843c83ca09ee892> (ISA was unable to establish the publication date) Whole Document	1-31
A	ENGAGE 8452 The Dow Chemical Company – Polyolefin Elastomer. Retrieved on 23/09/2016 from the internet, <URL: http://catalog.ides.com/Datasheet.aspx?I=43838&FMT=PDF&E=30971> (ISA was unable to establish the publication date) Whole Document	1-31
A	ENGAGE 8200 The Dow Chemical Company – Polyolefin Elastomer. Retrieved on 23/09/2016 from the internet, <URL: http://catalog.ides.com/Datasheet.aspx?I=43838&FMT=PDF&E=30962> (ISA was unable to establish the publication date) Whole Document	1-31
A	AGHARKAR, A., "PP in Rigid Packaging Applications", 2006, [retrieved from the internet on 2/09/2016] <URL: http://www.ipiindia.org/packging/item/pp-for-rigid-pkg-arvind-agharkar11012006> Whole Document	1-31
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INTERNATIONAL SEARCH REPORT C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT		International application No. **PCT/SG2016/050262**
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	Whole Document	1-31
A	WO 1995/032235 A1 (EXXON CHEMICAL PATENTS INC.) 30 November 1995 Whole Document	1-31

INTERNATIONAL SEARCH REPORT Information on patent family members	International application No. **PCT/SG2016/050262**

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		WO 2005012419 A2	10 Feb 2005

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INTERNATIONAL SEARCH REPORT Information on patent family members	International application No. **PCT/SG2016/050262**

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Patent Document/s Cited in Search Report		Patent Family Member/s	
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		MX 9605833 A	31 May 1998

End of Annex

Due to data integration issues this family listing may not include 10 digit Australian applications filed since May 2001.