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(54) **Title:** FLAME RETARDANT POLYPROPYLENE COMPOSITION

(57) **Abstract:** The invention relates to a flame retardant polypropylene composition comprising (A) a polypropylene-based polymer, (B) a flame retardant, (C) an anti-blooming particulate material selected from the group consisting of talc and silicone particles having the formula (I) and mixtures thereof $R_xSiO_{2-(x/2)}$ wherein x is a positive number greater than or equal to 1, and each R is independently an aliphatic hydrocarbon group, an aromatic hydrocarbon, or an unsaturated group.

FLAME RETARDANT POLYPROPYLENE COMPOSITION

The present invention relates to a flame-retardant polypropylene composition. The invention further relates to articles comprising such composition, such as toilet components.

Toilet components are often made flame retardant by blending flame retardants into the polymer matrix. The useful flame retardants include halogenated, non-halogenated, and phosphorus flame retardants.

US2014309350 discloses a thermoplastic resin composition comprising polypropylene, an organic flame retardant, a flame retardant synergist and a polyolefin-based wax. According to US2014309350, the toilet component using the composition is free of discoloration defects, is resistant to staining and deterioration caused by urine etc, and an excellent tactile sensation and marring resistance are achieved.

Blooming is a common problem in flame retardant compositions, which is a separation of the additive from the polymer matrix, which has a negative effect on the surface appearance of the article. US2006155027 relates to a method for making flame retardant olefin polymers with reduced blooming on the polymer surface. In US2006155027, this is achieved by using a reactive, peroxide-containing olefin polymer as an initiator. US2002169240 also discloses a non-blooming flame-retardant polyolefin composition. In US2002169240, this is achieved by adding a free radical source such as 2,3-dimethyl-2,3-diphenyl-butane and 2,3-dimethyl-2,3-diphenyl-hexane.

There is still a need in the art for a propylene composition which is flame retardant and in which the problem is blooming is prevented.

Accordingly, the present invention provides a flame retardant polypropylene composition comprising
(A) a polypropylene-based polymer,
(B) a flame retardant,
(C) an anti-blooming particulate material selected from the group consisting of talc and silicone particles having the formula (I) and mixtures thereof

$R_xSiO_{2-(x/2)}$ (I)

wherein x is a positive number greater than or equal to 1, and each R is independently an aliphatic hydrocarbon group, an aromatic hydrocarbon, or an unsaturated group, wherein the composition further comprises (D) a synergist, wherein preferably the amount of (D) is 0.1 to 7.0 wt%, preferably 1.0 to 5.0 wt%, with respect to the total composition..

The inventors have observed that the presence of the flame retardant in a polypropylene composition causes blooming problem, which reduces surface gloss during usage or product displayed in the shelf. It becomes particularly notable when the gloss decreases to below 60 at the testing angle of 60º.

According to the invention, it was surprisingly found that the addition of the anti-blooming particulate material as defined above reduces the level of decrease in the gloss of the article by preventing the blooming problem caused by the flame retardant.

(A) polypropylene-based polymer

Preferably, the amount of the component (A) is 75 to 95 wt%, for example 80 to 90 wt%, with respect to the total composition.

homopolymer and non-heterophasic copolymer

The polypropylene-based polymer may comprise or may be a propylene homopolymer or a propylene copolymer including random copolymers and (multi)block copolymers. The copolymer is preferably a random copolymer. The copolymer may consist of at least 70 wt% of propylene monomer units and up to 30 wt% of ethylene and/or α-olefin monomer units, based on the total weight of the copolymer. Preferably, the α-olefin is selected from the group of α-olefins having 4-10 carbon atoms, for example 1-butene, 1-pentene, 4-methyl-1-pentene, 1-hexene, 1-heptene or 1-octene. The propylene copolymer is preferably a propylene-ethylene copolymer.

The amount of ethylene and/or α-olefin monomer units in the propylene copolymer is preferably 1-15 wt%, more preferably 1-10 wt%, more preferably 1-6 wt%, more preferably 1-4 wt% based on the total weight of the propylene copolymer.

When the polypropylene-based polymer comprises a propylene α-olefin copolymer, the propylene copolymer is preferably a propylene-ethylene random copolymer wherein the amount of ethylene monomer units is 1-15 wt%, more preferably 1-10 wt%, more preferably 1-6 wt%, more preferably 1-4 wt% based on the total weight of the propylene copolymer.

The MFI of some preferred propylene homopolymer or propylene copolymer may be for example at least 10 dg/min, at least 20 dg/min, at least 30 dg/min or at least 40 dg/min and/or at most 100 dg/min, at most 80 dg/min, at most 60 dg/min or at most 50 dg/min, measured according to ISO1133-1:2011 (2.16 kg/230°C).

heterophasic propylene copolymer

The polypropylene-based polymer may comprise or may be a heterophasic propylene copolymer. Heterophasic propylene copolymers, also known as impact propylene copolymers or propylene block copolymers, are an important class of polymers due to their attractive combination of mechanical properties, such as impact strength over a wide temperature range and their low cost. These copolymers find a wide range of applications ranging from the consumer industry (for example packaging and housewares), the automotive industry to electrical applications.

Heterophasic propylene copolymers are generally prepared in one or more reactors, by polymerization of propylene in the presence of a catalyst and subsequent polymerization of an ethylene-α-olefin mixture. The resulting polymeric materials are heterophasic, but the specific morphology usually depends on the preparation method and monomer ratios used.

The heterophasic propylene copolymers which may be employed in the present invention can be produced using any conventional technique known to the skilled person, for example multistage process polymerization, such as bulk polymerization, gas phase polymerization, slurry polymerization, solution polymerization or any combinations thereof. Any conventional catalyst systems, for example, Ziegler-Natta or metallocene may be used. Such techniques and catalysts are described, for example, in WO06/010414; *Polypropylene and other Polyolefins*, by Ser van der Ven, Studies in Polymer Science 7, Elsevier 1990; WO06/010414, US4399054 and US4472524. Preferably, the heterophasic propylene copolymer is made using Ziegler-Natta catalyst.

The heterophasic propylene copolymer may be prepared by a process comprising
- polymerizing propylene and optionally ethylene and/or α-olefin in the presence of a catalyst system to obtain the propylene-based matrix and

- subsequently polymerizing ethylene and α-olefin in the propylene-based matrix in the presence of a catalyst system to obtain the dispersed ethylene-α olefin copolymer.

These steps are preferably performed in different reactors. The catalyst systems for the first step and for the second step may be different or same.

The heterophasic propylene copolymer of the composition of the invention consists of a propylene-based matrix and a dispersed ethylene-α-olefin copolymer. The propylene-based matrix typically forms the continuous phase in the heterophasic propylene copolymer. The amounts of the propylene-based matrix and the dispersed ethylene-α-olefin copolymer may be determined by ^{13}C-NMR, as well known in the art.

The propylene-based polymer may be one type of heterophasic propylene copolymer or a mixture of different types of heterophasic propylene copolymers, e.g. a mixture of a first type of a heterophasic propylene copolymer and a second type of heterophasic propylene copolymer at any weight ratio, e.g. 1:99-99:1 or 50:50.

The heterophasic propylene copolymer consists of
(a) a propylene-based matrix,
wherein the propylene-based matrix consists of a propylene homopolymer and/or a propylene copolymer consisting of at least 70 wt% of propylene monomer units and at most 30 wt% of ethylene and/or α-olefin monomer units, based on the total weight of the propylene-based matrix and
wherein the propylene-based matrix is present in an amount of 60 to 95 wt% based on the total heterophasic propylene copolymer and
(b) a dispersed ethylene-α-olefin copolymer,
wherein the dispersed ethylene-α-olefin copolymer is present in an amount of 40 to 5 wt% based on the total heterophasic propylene copolymer and
wherein the sum of the total amount of propylene-based matrix and total amount of the dispersed ethylene-α-olefin copolymer in the heterophasic propylene copolymer is 100 wt% with respect to the heterophasic propylene copolymer.

The propylene-based matrix consists of a propylene homopolymer and/or a propylene-copolymer consisting of at least 70 wt% of propylene monomer units and up to 30 wt% of ethylene and/or α-olefin monomer units, for example consisting of at least 80 wt% of propylene monomer units and up to 20 wt% of ethylene and/or α-olefin monomer units, for example consisting of at least 90 wt% of propylene monomer units and up to 10 wt% of ethylene and/or α-olefin monomer units, based on the total weight of the propylene-based matrix. Preferably, the α-olefin is selected from the group of α-olefins having 4 to 10 carbon atoms, for example 1-butene, 1-pentene, 4-methyl-1-pentene, 1-hexene, 1-heptene or 1-octene. The propylene copolymer is preferably a propylene-ethylene copolymer.

Preferably, the propylene-based matrix consists of a propylene homopolymer. When the propylene-based matrix consists of a propylene homopolymer, a higher stiffness is obtained compared to the case where the propylene-based matrix is a propylene copolymer as described above, which may be advantageous.

The melt flow index (MFI) of the propylene-based matrix (before the heterophasic propylene copolymer is mixed into the composition of the invention), $MFI_{PP,}$ may be for example at least 0.1 dg/min, at least 0.2 dg/min, at least 0.3 dg/min, at least 0.5 dg/min, at least 1 dg/min, at least 1.5 dg/min, and/or for example at most 50 dg/min, at most 40 dg/min, at most 30 dg/min, at most 25 dg/min, at most 20 dg/min, measured according to ISO1133 (2.16 kg/230°C). The MFI_{PP} may be in the range of for example 0.1 to 50 dg/min, for example from 0.2 to 40 dg/min, for example 0.3 to 30 dg/min, for example 0.5 to 25 dg/min, for example from 1 to 20 dg/min, for example from 1.5 to 10 dg/min, measured according to ISO1133 (2.16 kg/230°C).

The propylene-based matrix is present in an amount of 60 to 95wt%. Preferably, the propylene-based matrix is present in an amount of 60 to 80wt%, for example at least 65 wt% or at least 70 wt% and/or at most 78 wt%, based on the total heterophasic propylene copolymer.

The propylene-based matrix is preferably semi-crystalline, that is it is not 100% amorphous, nor is it 100% crystalline. For example, the propylene-based matrix is at least 40% crystalline, for example at least 50%, for example at least 60% crystalline and/or for example at most 80% crystalline, for example at most 70% crystalline. For example, the propylene-based matrix has a crystallinity of 60 to 70%. For purpose of the invention, the degree of crystallinity of the propylene-based matrix is measured using differential scanning calorimetry (DSC) according to ISO11357-1 and ISO11357-3 of 1997, using a scan rate of 10°C/min, a sample of 5mg and the second heating curve using as a theoretical standard for a 100% crystalline material 207.1 J/g.

Besides the propylene-based matrix, the heterophasic propylene copolymer also comprises a dispersed ethylene-α-olefin copolymer. The dispersed ethylene-α-olefin copolymer is also referred to herein as the ‘dispersed phase’. The dispersed phase is embedded in the heterophasic propylene copolymer in a discontinuous form. The particle size of the dispersed phase is typically in the range of 0.05 to 2.0 microns, as may be determined by transmission electron microscopy (TEM). The amount of the dispersed ethylene-α-olefin copolymer in the heterophasic propylene copolymer may herein be sometimes referred as RC.

In some preferred heterophasic propylene copolymers, the amount of ethylene monomer units in the ethylene-α-olefin copolymer is in the range of 5 to 65 wt%, for example at most 40 wt%, at most 30 wt% or at most 20 wt%, based on the total weight of the ethylene-α-olefin copolymer. The amount of ethylene in the dispersed ethylene-α-olefin copolymer in the heterophasic propylene copolymer may herein be sometimes referred as RCC2.

The α-olefin in the ethylene-α-olefin copolymer is preferably chosen from the group of α-olefins having 3 to 8 carbon atoms. Examples of suitable α-olefins having 3 to 8 carbon atoms include but are not limited to propylene, 1-butene, 1-pentene, 4-methyl-1-pentene, 1-hexene, 1-heptene and 1-octene. More preferably, the α-olefin in the ethylene-α-olefin copolymer is chosen from the group of α-olefins having 3 to 4 carbon atoms and any mixture thereof, more preferably the α-olefin is propylene, in which case the ethylene-α-olefin copolymer is ethylene-propylene copolymer.

The MFI of the dispersed ethylene α-olefin copolymer (before the heterophasic propylene copolymer is mixed into the composition of the invention), MFI_{EPR}, may be for example at least 0.001 dg/min, at least 0.01 dg/min, at least 0.1 dg/min, at least 0.3 dg/min, at least 0.7 dg/min, at least 1 dg/min, and/or for example at most 30 dg/min, at most 20 dg/min, at most 15 dg/min at most 10 dg/min, at most 5 dg/min or at most 3 dg/min. The MFI_{EPR} may be in the range for example from 0.001 to 30 dg/min, for example from 0.01 to 20 dg/min, for example 0.1 to 15 dg/min, for example 0.3 to 10 dg/min, for example from 0.7 to 5 dg/min, for example from 1 to 3 dg/min. MFI_{EPR} is calculated taking into account the MFI of the propylene-based matrix (MFI_{PP}) measured according to ISO1133 (2.16 kg/230 ºC), the MFI of the heterophasic propylene copolymer (MFIheterophasic) measured according to ISO1133 (2.16 kg/230 ºC) and the amount of the propylene-based matrix in the heterophasic propylene copolymer (matrix content) and the amount of the dispersed phase in the heterophasic propylene copolymer (rubber content (RC)) according to the following formula:

$$MFIEPR = 10\hat{}(\frac{Log\ MFIheterophasic - matrix\ content * Log\ MFIPP}{rubber\ content})$$

The dispersed ethylene-α-olefin copolymer is present in an amount of 40 to 5 wt% based on the total heterophasic propylene copolymer. In some preferred heterophasic propylene copolymers, the dispersed ethylene-α-olefin copolymer is present in an amount of at least 10 wt%, for example at least 15 wt% or at least 17 wt%, and/or at most 35 wt%, for example at most 30 wt% or 25 wt%, based on the total heterophasic propylene copolymer.

In the heterophasic propylene copolymer in the composition of the invention, the sum of the total weight of the propylene-based matrix and the total weight of the dispersed ethylene-α-olefin copolymer is 100 wt% with respect to the heterophasic propylene copolymer.

Preferably, the heterophasic propylene copolymer has a fraction soluble in p-xylene at 25 ºC (CXS) measured according to ISO 16152:2005 of 40 to 5 wt%. In some preferred heterophasic propylene copolymers, the heterophasic propylene copolymer has CXS of at least 10 wt%, for example at least 15 wt% or at least 17 wt%, and/or at most 35 wt%, for example at most 30 wt% or 25 wt%. In some preferred heterophasic propylene copolymers, the heterophasic propylene copolymer has CXS of 10 to 25 wt%. In some preferred heterophasic propylene copolymers, the heterophasic propylene copolymer has CXS of 25 to 40 wt%.

Preferably, the amount of ethylene monomer units in the heterophasic propylene copolymer (sometimes referred as TC2) is in the range of 3-25 wt% based on the heterophasic propylene copolymer. For example, the amount of ethylene monomer units in the heterophasic propylene copolymer may be at least 5 wt% or at least 7 wt% and/or at most 20 wt%, at most 15 wt% or at most 13 wt%, based on the heterophasic propylene copolymer.

The MFI of some preferred heterophasic propylene copolymers may be for example at least 10 dg/min or at least 15 dg/min and/or at most 50 dg/min or at most 40 dg/min, measured according to ISO1133 (2.16 kg/230°C). The MFI of some preferred heterophasic propylene copolymers may be for example at least 0.1 dg/min, at least 0.2 dg/min, at least 0.3 dg/min, at least 0.5 dg/min, at least 1 dg/min at least 1.5

dg/min, and/or for example at most 8 dg/min or at most 5 dg/min, measured according to ISO1133 (2.16 kg/230°C).

The values of the MFI of the propylene-based matrix (MFI_{PP}) and the MFI of the dispersed ethylene-α-olefin elastomer (MFI_{EPR}) mentioned herein are understood as the values before the heterophasic propylene copolymer is mixed with other components to obtain the composition according to the invention.The value of the MFI of the heterophasic propylene copolymer (MFIheterophasic) refers to the final MFI of the heterophasic propylene copolymer. To exemplify this:
In case the heterophasic propylene copolymer is not subjected to vis-breaking or shifting by melt-mixing with a peroxide, the MFIheterophasic is the original MFI value of the heterophasic propylene copolymer. In case the heterophasic propylene copolymer is subjected to vis-breaking or shifting by melt-mixing with a peroxide, the MFIheterophasic is the value of the heterophasic propylene copolymer after such vis-breaking or shifting.

Preferably, in the heterophasic propylene copolymer according to the invention,
the propylene copolymer of the matrix consists of propylene monomer units and ethylene and/or α-olefin monomer units wherein the α-olefin is selected from the group of α-olefins having 2 or 4 to10 carbon atoms and
the dispersed ethylene-α-olefin copolymer consists of ethylene and α-olefin selected from the group of α-olefins having 3 to 8 carbon atoms.

The propylene-based polymer may also be a combination of any of the propylene-based polymer mentioned above, e.g. a mixture of a propylene homopolymer and a heterophasic propylene copolymer at a weight ratio of 1:99-99:1 or 50:50, a mixture of a propylene homopolymer and a random propylene-ethylene copolymer at a weight ratio of 1:99-99:1 or 50:50 or a mixture of two different types of propylene homopolymer at a weight ratio of 1:99-99:1 or 50:50.

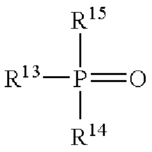
(B) flame retardant

Preferably, the amount of the component (B) is 1 to 25 wt%, more preferably 3 to 20 wt%, more preferably 5 to 15 wt%, with respect to the total composition.

Preferably, the flame retardant is a halogenated flame retardant. The advantages of the present invention are prominent when the flame retardant is a halogenated flame retardant. As the halogenated flame retardant, brominated flame retardants are preferred. Suitable examples include tetrabromobisphenol A derivatives, including bis(2-hydroxyethyl)ether of tetrabromobisphenol A, bis(3-acryloyloxy-2-hydroxypropyl)ether of tetrabromobisphenol A, bis(3-methacryloyloxy-2-hydroxypropyl)ether of tetrabromobisphenol A, bis(3-hydroxypropyl)ether of tetrabromobisphenol A, bis(2,3-dibromopropyl)ether of tetrabromobisphenol A, diallyl ether of tetrabromobisphenol A, and bis(vinylbenzyl)ether of tetrabromobisphenol A; brominated polycarbonates, tetrabromobisphenol A polycarbonate oligomer, brominated polyacrylate such as polypentabromobenzyl acrylate; brominated polystyrenes, such as polydibromostyrenes and polytribromostyrenes; brominated BPA polyepoxides, tetrabromocyclooctanes; dibromoethyldibromocyclohexanes such as 1,2-dibromo-4-(1,2-dibromoethyl)-cyclohexane; ethylene-bis-tetrabromophthalimide; hexabromocyclododecanes; tetrabromophthalic anhydrides; brominated diphenylethers such as decabromodiphenyl ether; poly(2,6-dibromophenylene ether); tris(2,4,6-tribromophenoxy-1,3,5-triazine; tris(tribromoneopentyl)phosphate; and decabromodiphenyl ethane. Particularly preferred examples include bis(2,3-dibromopropyl)ether of tetrabromobisphenol A (commercially available as FR-720 from ICL Industrial products) and polypentabromobenzyl acrylate (commercially available as FR-1025 from ICL Industrial products), tris(tribromoneopentyl)phosphate (commercially available as FR-370 from ICL Industrial products), decabromodiphenyl ether (commercially available as FR-1210 from ICL Industrial products) and decabromodiphenyl ethane (commercially available as FR-1410 from ICL Industrial products). Most preferred is bis(2,3-dibromopropyl)ether of tetrabromobisphenol A.

Alternatively, the flame retardant compound comprises a halogen-free, phosphorus containing compound. Non-limiting examples of phosphorus compounds of the phosphine class are aromatic phosphines, such as triphenylphosphine, tritolylphosphine, trinonylphosphine, trinaphthylphosphine, tetraphenyldiphosphine, tetranaphthyldiphosphine and the like. Suitable phosphine oxides are of the formula (IV)

(IV)



$$R^{13}-\overset{\overset{R^{15}}{|}}{\underset{\underset{R^{14}}{|}}{P}}=O$$

wherein R^{13}, R^{14} and R^{15} are independently at each occurrence, selected from the group consisting of a C_1 to C_{30} aliphatic radical, C_3-C_{30}cycloaliphatic radical, and C_3-C_{30} aromatic radical. Examples of phosphine oxides are triphenylphosphine oxide, tritolylphosphine oxide, trisnonylphenylphosphine oxide, tricyclohexylphosphine oxide, tris(n-butyl)phosphine oxide, tris(n-hexyl)phosphine oxide, tris(n-octyl)phosphine oxide, tris(cyanoethyl)phosphine oxide, benzylbis(cyclohexyl)phosphine oxide, benzylbisphenylphosphine oxide and phenylbis(n-hexyl)phosphine oxide. Other suitable compounds are triphenylphosphine sulfide and its derivatives as described above for phosphine oxides and triphenyl phosphate.

Other examples of phosphorus compounds are hypophosphites, e.g. metal hypophosphites where metal is a alkali metal, alkaline earth metal or a transition metal or Al. Ca, Al, Zn, Ti, Mg, Ba and the like and organic hypophosphites, such as cellulose hypophosphite esters, esters of hypophosphorous acids with diols, e.g. that of 1,10-dodecanediol.

In one embodiment the phosphorus compound may be a phosphinate (e.g. A_1,A_2-P(=O)(OA_3), wherein A_1, A_2 and A_3 are independently at any occurrence a C_1 to C_{30} aliphatic radical, C_3-C_{30} cycloaliphatic radical, and C_3-C_{30} aromatic radical. Examples of phosphinic acids which are suitable constituents of the phosphinates are: dimethylphosphinic acid, ethylimethyphosphinic acid, diethylphosphinic acid, methyl-n-propylphosphinic acid, methanedi(methylphosphinic acid), benzene-1,4-(dimethylphosphinic acid), methylphenylphosphinic acid and diphenylphosphinic acid. Other examples of phosphorus compounds are metal salts of the above dialkyl or diaryl or arylalkyl phosphinic acid, where metal is an alkali metal, Li, Na, K and Cs and the like or alkaline earth metal, Be, Ca, Mg, Ba, Sr and the like or a transition metal, Zn, Ti and the like or other main group elements such as Al, Sn, Sb and the like. These phosphinate salts can be monomeric or polymeric in structure. Some of these compounds are inorganic coordination polymers of aryl(alkyl)phosphinic acids, such as poly-β-sodium(I)ethylphenylphosphinate, zinc salt of diethyl phosphinic acid, etc.

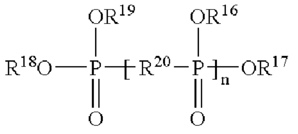
It is also possible to use substituted phosphinic acids and anhydrides, e.g. diphenylphosphinic acid. Other possible compounds are di-p-tolylphosphinic acid and dicresylphosphinic anhydride. Compounds such as the bis(diphenylphosphinic)esters of hydroquinone, ethylene glycol and propylene glycol, inter alia, may also be used. Other suitable compounds are aryl(alkyl)phosphinamides, such as the dimethylamide of diphenylphosphinic acid, and sulfonamidoaryl(alkyl)phosphinic acid derivatives, such as p-tolylsulfonamidodiphenylphosphinic acid. In one embodiment the flame retardant compound is bis(diphenylphosphinic)esters of hydroquinone and ethylene glycol and of the bis(diphenylphosphinate) of hydroquinone.

Other suitable examples are derivatives of phosphorous acid. Suitable compounds are cyclic phosphonates which derive from pentaerythritol, from neopentyl glycol or from pyrocatechol. In another embodiment other phosphorus based flame retardants are triaryl(alkyl)phosphites, such as triphenyl phosphite, tris(4-decylphenyl)phosphite, tris(2,4-di-tert-butylphenyl)phosphite and phenyl didecyl phosphite. It is also possible to use diphosphites, such as propylene glycol 1,2-bis(diphosphite) or cyclic phosphites which derive from pentaerythritol, from neopentylglycol or from pyrocatechol.

In one embodiment the flame retardant is at least one selected from the group consisting of neopentyl glycol methylphosphonate and methyl neopentyl glycol phosphite, pentaerythritol dimethyldiphosphonate, dimethyl pentaerythritol diphosphate, tetraphenyl hypodiphosphate and bisneopentyl hypodiphosphate.

Other effective phosphorus based flame retardants are particularly alkyl- and aryl-substituted phosphates. Examples of these are phenyl bisdodecyl phosphate, phenyl ethyl hydrogen phosphate, phenyl bis(3,5,5-trimethylhexyl)phosphate, ethyl diphenyl phosphate, 2-ethylhexyl ditolyl phosphate, diphenyl hydrogen phosphate, bis(2-ethylhexyl)p-tolyl phosphate, tritolyl phosphate, bis(2-ethylhexyl)phenyl phosphate, di(nonyl)phenyl phosphate, phenyl methyl hydrogenphosphate, di(dodecyl)p-tolyl phosphate, p-tolylbis(2,5,5-trimethylhexyl)phosphate and 2-ethylhexyl diphenyl phosphate. Particularly suitable phosphorus compounds are those in which each radical is aryloxy. Very particularly suitable compounds are triphenyl phosphate, Bisphenol-A bis (diphenyl phosphate) and resorcinol bis(diphenyl phosphate) and its ring-substituted derivatives of formula (V):

(V)



$$R^{18}O-\overset{\displaystyle OR^{19}}{\underset{\displaystyle O}{\overset{|}{\underset{\|}{P}}}}-\left[R^{20}-\overset{\displaystyle OR^{16}}{\underset{\displaystyle O}{\overset{|}{\underset{\|}{P}}}}\right]_n-OR^{17}$$

wherein R^{16} to R^{20} are each occurrence aromatic radicals having from 6 to 20 carbon atoms, preferably phenyl, which may have substitution by alkyl groups having from 1 to 4 carbon atoms, preferably methyl, R^{22} is a bivalent phenol radical, preferably and n is an average value of from 0.1 to 100, preferably from 0.5 to 50, in particular from 0.8 to 10 and very particularly from 1 to 5. It is also possible to use cyclic phosphates like for example diphenyl pentaerythritol diphosphate and phenyl neopentyl phosphate are particularly suitable. Other suitable flame retardants are elemental red phosphorous and also compounds that contain phosphorous nitrogen bonds, such as phosphononitrile chloride, phosphoric acid ester amides, phosphoric acid amides, phosphonic acid amides, phosphinic acid amides, tris(aziridinyl)-phosphinic oxide and tetrakis(hydroxymethyl)phosphonium chloride.

(C) anti-blooming particulate material

In the present invention, the inventors surprisingly found the addition of anti-blooming particulate material is able to prevent the flame retardent from migrating to the surface of the polypropylene composition hence the blooming effect is suppressed.

Preferably, the amount of the anti-blooming particulate material in the composition of the invention is 0.5 to 5.0 wt%, for example 0.6 to 3.0 wt% or 0.7 to 2.0 w%, with respect to the total composition. Higher amount of anti-blooming particulate material could lead to deterioration of the impact performance of the polypropylene composition.

When the anti-blooming particulate material is a mixture of the talc and the silicon particles (I), the weight ratio of the talc and the silicon particles (I) may be 1:99 to 99:1, for example 1:50 to 50:1, 1:10 to 10:1, 1:5 to 5:1, 1:3 to 3:1 or 1:2 to 2:1.

talc

Although any type of talc can be used, high aspect ratio talc may be used where higher stiffness is required, whereas very fine talc, i.e. talc with a small particle size can be used if the impact properties of the composition need to be retained. Obviously, mixtures of more than one type of talc also should be considered as falling within the ambit of the present invention. For example, a mixture of high aspect ratio talc and very fine talc may be used wherein the ratio of the different talcs can be used as a tool for tailoring the mechanical properties of the moulding composition. Commonly used talc materials are commercially available for example from Imerys under the brand names high aspect ratio HAR talc, Jetfine talc or Steamic talc. Further suitable talc material is available from Imifabi as HTPultra5L.

The talc is preferably a high aspect ratio talc, meaning a talc having a lamellarity index (LI) of more than 2.90, the lamellarity index defined as:

$$LI = \frac{d50(L) - d50(S)}{d50(S)}$$

wherein d50(L) is the median particle size d50 [mass percent] determined by laser diffraction technique in accordance with ISO 13320-1 and d50(S) is the median particle size d50 [mass percent] as determined by sedimentation technique in accordance with ISO 13317-3. The LI is preferably at least 3.10, more preferably at least 3.50 such as from 3.50 to 9.00 or 4.10 to 7.00. The LI is determined on the basis of the talc prior to being compounded with the other components of the polypropylene composition.

Preferably, the talc has d50 as determined by sedimentation analysis, Stokes' law (ISO 13317-3) of at most 3.00 µm, for example 1.00 to 2.50 µm or 0.50 to 1.00 µm.

In some embodiments, the talc has BET specific surface area determined according to ISO9277-2010 of 15 to 25m^2/g according to DIN66131/2.

In some embodiments, the talc has BET specific surface area determined according to DIN66131/2 of 10 to 15 m^2/g.

<u>silicone particles</u>

Silicone particles comprise a three-dimensional polymer chain of the formula

$R_xSiO_{2-(x/2)}$ (I)

wherein x is a positive number greater than or equal to 1, and each R is independently an aliphatic hydrocarbon group, an aromatic hydrocarbon or an unsaturated group

Preferably, x is a positive number greater than or equal to 1, specifically, 1 to 1.9, more specifically, 1 to 1.5, and even more specifically, 1 to 1.2; and each R is independently an organic group, such as an aliphatic hydrocarbon group, e.g., methyl, ethyl, or butyl; or an aromatic hydrocarbon, e.g., phenyl, and can comprise an unsaturated group, e.g., vinyl.

In exemplary embodiments, R is a hydrocarbon group having 1 to 8, specifically, 1 to 5, carbon atoms, more specifically, methyl. Specifically mentioned silicon resin particles comprise methylsilsequioxane.

The crosslinked silicone particles have d50 of preferably 0.01 to 50 µm, more preferably 1 to 30 µm, more preferably 1.8 to 10 µm. When d50 is smaller than 0.01 µm or larger than 50 µm, light diffusion property may become unsatisfactory. The average particle diameter indicates a 50 % value (d50) of an integral particle size distribution obtained by a laser diffraction/scattering method. The number of particle size distributions may be single or plural. That is, it is possible to combine two or more different kinds of crosslinked silicone particles which differ in average particle diameter. However, preferably, the crosslinked silicone particles have a narrow particle size distribution. It is preferred that the crosslinked silicone particles have a distribution in which at least 70 wt% of all particles are included in an average particle diameter range of 1.8 to 2.2 µm. The shape of the light diffusing agent is preferably almost globular from the viewpoint of light diffusion property and more preferably almost spherical. The globular shape includes an elliptical shape.

The bulk specific gravity of the silicone particles may be 0.35 to 0.67 kilograms per liter.

The refractive index of the light diffusing agent is preferably 1.30 to 1.80, more preferably 1.33 to 1.70 and much more preferably 1.35 to 1.65. When it is contained in the resin composition, it exhibits a satisfactory light diffusing function.

Preferably, the specific surface area of the crosslinked silicone particles is at least 10 m^2/g, preferably at least 20 m^2/g, preferably at least 30 m^2/g as determined by BET nitrogen absorption technique according ISO 9277-2010.

Commercially available silicone-based particles suitable for use in the present invention include the TOSPEARL series of Toshiba Silicone Co., Ltd., the TORAYFIL series of Dow Corning Toray Co., Ltd. and the silicone powders of Shin-Etsu Chemical Co., Ltd.

(D) synergist

The composition further comprise a fire-retardant compound which is not a halogenated flame retardant, which may serve as a synergist. In such a composition, the amount of the halogenated compound may be reduced, thus leading to a lower amount of halogen/bromine in the composition, which makes the composition more economic.

Examples of synergists include antimony compounds (e.g. antimony-trioxide, -tetraoxide, -pentaoxide, and sodium antimonate), tin compounds (e.g. tin-oxide and -hydroxide, dibutyl tin maleate), molybdenum compounds (e.g. molybdenum oxide, ammonium molybdate), zirconium compounds (e.g. zirconium-oxide and -hydroxide), boron compounds (e.g. zinc-borate, barium-metaborate), zinc compounds such as zinc stannate, silicon compounds such as silicon oil, fluoro compounds such as polytetrafluoroethylene, and hydroxystannate or any mixtures of two or more of them.

Such compounds serve as synergists, which reduce the overall, required amount of flame-retardant compounds in the polyolefin composition.

The amount of the component (D) is 0.1 to 7.0 wt%, preferably 1.0 to 5.0 wt%, with respect to the total composition.

(E) other optional additives

The composition according to the invention may further comprise various additives. The additives may include nucleating agents, stabilisers, *e.g.* heat stabilisers, anti-oxidants, UV stabilizers; colorants, like pigments and dyes; clarifiers; surface tension modifiers; lubricants; mould-release agents; flow improving agents; plasticizers; anti-static agents and blowing agents.

The amount of the component (E) may be 0 to 10 wt%, for example 0.05 to 5.0 wt%, or 0.1 to 1.0 wt%, with respect to the total composition.

composition

The sum of all components added in the process of the invention to form the composition comprising (A), (B), (C) and the optional components (D) and (E) should add up to 100% by weight of the total composition.

Preferably, the total of components (A) and (B) is at least 85 wt% or at least 90 wt% of the total composition.

Preferably, the total of components (A), (B) and (C) is at least 85 wt%, at least 90 wt% or at least 95 wt% of the total composition.

Preferably, the total of components (A), (B), (C) and (D) is at least 90 wt%, at least 95 wt%, at least 98 wt% or at least 99 wt% of the total composition.

Process for making composition

The composition of the invention may be obtained by a process comprising melt-mixing (A), (B), (C) and optionally (D) and/or (E) by using any suitable means. Accordingly, the invention further relates to a process for the preparation of the composition according to the invention comprising melt mixing (A), (B), (C) and optionally (D) and/or (E). Preferably, the composition of the invention is made in a form that allows easy processing into a shaped article in a subsequent step, like in pellet or granular form.

Preferably, the composition of the invention is in pellet or granular form as obtained by mixing all components in an apparatus like an extruder; the advantage being a composition with homogeneous and well-defined concentrations of the additives.

With melt-mixing is meant that the components (B) and (C) and optionally (D) and/or optionally (E) are mixed with (A) at a temperature that exceeds the melting point of (A). Melt-mixing may be done using techniques known to the skilled person, for example in an extruder. Generally, in the process of the invention, melt-mixing is performed at a temperature in the range from 170-300°C.

Suitable conditions for melt-mixing, such as temperature, pressure, amount of shear, screw speed and screw design when an extruder is used are known to the skilled person.

When using an extruder, a conventional extruder such as a twin-screw extruder may be used. The temperature can vary through the different zones of the extruder as required. For example, the temperature may vary from 100°C in the feed zone to 300°C at the die. Preferably, the temperature in the extruder varies from 200 to 265°C. Likewise, the screw speed of the extruder may be varied as needed. Typical screw speed is in the range from about 100rpm to about 400rpm.

Properties of composition

MFI

The MFI of the composition according to the invention may be in the range of for example 5 to 50 dg/min, for example from 7 to 40 dg/min, for example 10 to 30 dg/min, for example 15 to 35 dg/min, measured according to ISO1133-1:2011 (2.16 kg/230°C).

Gloss

Preferably, the composition has a gloss as measured according to ASTM D2457-13 at 60 º after 24 hours of conditioning at a temperature of 50 ºC (Gloss_{24}) of more than 60.

Preferably, the composition has a gloss as measured according to ASTM D2457-13 at 60 º after 4.8 hours of conditioning at a temperature of 50 ºC ($\text{Gloss}_{4.8}$) of more than 65, more preferably more than 68.

Preferably, the composition has a gloss as measured according to ASTM D2457-13 at 60 º after 0 hours of conditioning at a temperature of 50 ºC (Gloss_{0}) of more than 80.

Preferably, $(Gloss_0-Gloss_{24})/Gloss_0$ is at most 0.30, more preferably at most 0.25.

Flame retardancy

Preferably, the composition according to the invention has a flame retardancy of V0 according to the UL94 test standard at a sample thickness of 1.5 mm, wherein the sample is conditioned at 23 ºC and 50 percent relative humidity for 48 hours prior to testing.

Preferably, the composition according to the invention has a flame retardancy of V0 according to the UL94 test standard at a sample thickness of 1.5 mm, wherein the sample is conditioned at 70 ºC ±2 ºC for 168 hours ±2 hours and cooled in a desiccator for at least 48 hours prior to testing.

Notched Izod impact strength

Preferably, the composition according to the invention has an Notched Izod impact strength according to ISO 180:2000 at 23 ºC (test geometry: 80*10*4 mm) of at least 1.0 kJ/m2.

Flexural modulus

Preferably, the composition according to the invention has a flexural modulus according to ISO 178:2010 (parallel; test geometry: 80*10*4 mm thickness, 2.0 mm/min) of at least 1000 MPa.

Flexural strength

Preferably, the composition according to the invention has a flexural strength according to ISO 178:2010 (parallel; test geometry: 80*10*4 mm thickness, 2.0 mm/min) of at least 30 MPa.

Density

Preferably, the composition according to the invention has a density according to 1183-1:2012 of 0.9980 to 1.0020 g/cm3.

Further aspects

The composition according to the invention may be processed by any conventional technique known in the art into an article. Suitable examples of processing techniques wherein the composition according to the invention may be used include injection moulding, injection blow moulding, injection stretch blow moulding, rotational moulding, compression moulding, extrusion, extrusion compression moulding, extrusion blow moulding, sheet extrusion, film extrusion, cast film extrusion, foam extrusion, thermoforming and thin-walled injection moulding.

The invention further relates to an article comprising the composition according to the invention. In particular, the invention relates to an article comprising the composition according to the invention, wherein the article is made by one of the processing techniques mentioned above.

Preferably, the article according to the invention is toilet components.

The invention further relates to the use of the composition according to the invention for making toilet components.

It is noted that the invention relates to all possible combinations of features described herein, preferred in particular are those combinations of features that are present in the claims. It will therefore be appreciated that all combinations of features relating to the composition according to the invention; all combinations of features relating to the process according to the invention and all combinations of features relating to the composition according to the invention and features relating to the process according to the invention are described herein.

It is further noted that the term ‘comprising’ does not exclude the presence of other elements. However, it is also to be understood that a description on a product/composition comprising certain components also discloses a product/composition consisting of these components. The product/composition consisting of these components may be advantageous in that it offers a simpler, more economical process for the preparation of the product/composition. Similarly, it is also to be understood that a description on a process comprising certain steps also discloses a process consisting of these steps. The process consisting of these steps may be advantageous in that it offers a simpler, more economical process.

When values are mentioned for a lower limit and an upper limit for a parameter, ranges made by the combinations of the values of the lower limit and the values of the upper limit are also understood to be disclosed.

The invention is now elucidated by way of the following examples, without however being limited thereto.

Examples

Materials as shown in Table 1 were used in the experiments.

Table 1

PP1	PP Homopolymer, MFI 47 g/10min
PP2	PP Homopolymer, MFI 6 g/10min
Flame retardant (FR)	Bis (2,3-dibromopropyl ether) of tetrabromobisphenol A
Synergist	Sb_2O_3
Antioxidant 1 (AO1)	AO1010
Antioxidant 2 (AO2)	AO168
Color agent	TiO_2 from Huntsman
Talc1	HAR T84 from Imerys: d50 of 2.0 µm by sedimentation analysis, Stokes' Law (ISO 13317-3); BET specific surface area by ISO9277 of 19.5 m^2/g
Talc2	HTPultra5L from Imifabi: d50 of 0.65 µm by sedimentation analysis, Stoke-s Law (ISO 13317-3), BET specific surface area by DIN66131/2 of 13 m^2/g
Silicon	Tospearl 120 from Momentive: silicone resin particles with d50 of 2 µm, specific surface area 15-35 m^2/g

The components of the compositions as shown in Table 2 were melt-mixed by a twin-screw extruder to obtain pellets. Various properties were measured.

The gloss was measured according to ASTM D2457-13 using a colorchip of 90*50*3.20 mm at 60º after 0 hour, 4.8 hours and 24 hours of conditioning in an oven at a temperature of 50 ºC.

The flame retardancy was measured according to the UL94 test standard at a sample thickness of 1.5 mm, wherein the sample was conditioned at 23 ºC ±2 ºC and 50 percent ± 10% relative humidity for 48 hours prior to testing. The flame retardancy was also measured according to the UL94 test standard at a sample thickness of 1.5 mm, wherein the sample was conditioned at 70 ºC ±2 ºC for 168 hours ±2 hours and cooled in a desiccator for at least 48 hours prior to testing.

The flexural modulus and flexural strength were measured according to ISO 178:2010 (parallel; test geometry: 80*10*4 mm, 2.0 mm/min).

The Izod impact strength was measured according to ISO 180:2000 at -20 ºC and 23 ºC (test geometry: 80*10*4 mm).

The density was measured according to 1183-1:2012.

The melt flow rate was measured according to ISO1133-1:2011 (2.16 kg/230°C).

Table 2 (amounts are in weight parts)

	CE1	CE2	CE3	Ex4	Ex5	Ex6	Ex7	Ex8	Ex9
PP1	55.20	56.00	55.20	54.40	54.40	54.40	54.00	53.70	54.00
PP2	29.00	29.00	29.00	29.00	29.00	29.00	29.00	29.00	29.00
Flame retardant (FR)		11.00	11.00	11.00	11.00	11.00	11.00	11.00	11.00
Synergist	3.80	3.80	3.80	3.80	3.80	3.80	3.80	3.80	3.80
Antioxidant 1 (AO1)	0.10	0.10	0.10	0.10	0.10	0.10	0.10	0.10	0.10
Antioxidant 2 (AO2)	0.10	0.10	0.10	0.10	0.10	0.10	0.10	0.10	0.10
Color agent	0.80		0.80	0.80	0.80	0.80	0.80	0.80	0.80
Silicon				0.80		0.40	0.40	1.50	0.40
Talc 1									0.80
Talc 2					0.8	0.40	0.8		
Gloss @ 60 _ 0 day	84.37	83.3	83.1	83.53	83.8	83.9	84.03	82.47	82.87
Gloss @ 60 _ 0.2 day	77.13	60.2	67.9	69.1	69.87	69.17	69.83	68.3	68.47
Gloss @ 60 _ 1 day	73.87	52.1	51.9	63.13	62.5	61.1	62.63	61.23	59.6
Flexural Modulus		1448.42	1500.6	1446.76	1550.6	1529.14	1515.6	1448.7	1575.6
Flexural Strength		39.94	40.18	39.1	41.92	40.48	40.5	39.24	42.16
Impact Strength @ -20		1.75	1.78	1.92	1.71	1.89	1.72	1.9	1.74
Impact Strength @ RT		2.29	2.67	2.55	2.16	2.28	2.42	2.9	2.87
MFI-Ind / 1		23.33	24.74	23.2	23.94	23.67	24.51	22.94	23.81
Density		0.9962	0.9906	0.9942	1.0046	1.0028	1.0015	0.9999	0.9997
FR rating @ 1.5 mm normal	HB	V0	V0	V0	V0	V0	V0	V0	V0
FR rating @ 1.5 mm Aging	HB	V0	V0	V0	V0	V0	V0	V0	V0

From the comparison of CE1 versus CE3, it can be understood that the presence of the flame retardant leads to a substantial decrease of the gloss after one day.

From the comparison of CE3 versus Ex4-Ex9, it can be understood that the decrease in the gloss is reduced by the addition of the anti-blooming particulate material.

From the comparison of CE3 versus Ex 4 and Ex 5, it can be understood that the use of silicon leads to a higher impact strength and the use of talc leads to higher flexural properties.

CLAIMS

1. A flame retardant polypropylene composition comprising
(A) a polypropylene-based polymer,
(B) a flame retardant,
(C) an anti-blooming particulate material selected from the group consisting of talc and silicone particles having the formula (I) and mixtures thereof
$R_xSiO_{2-(x/2)}$ (I)
wherein x is a positive number greater than or equal to 1, and each R is independently an aliphatic hydrocarbon group, an aromatic hydrocarbon, or an unsaturated group, wherein the composition further comprises (D) a synergist, wherein preferably the amount of (D) is 0.1 to 7.0 wt%, preferably 1.0 to 5.0 wt%, with respect to the total composition.

2. The composition according to any one of the preceding claims, wherein the amount of (C) the anti-blooming particulate material is 0.5 to 5.0 wt%, for example 0.6 to 3.0 wt% or 0.7 to 2.0 w%, with respect to the total composition.

3. The composition according to any one of the preceding claims, wherein (C) the anti-blooming particulate material comprises talc having a lamellarity index (LI) of more than 2.90, the lamellarity index defined as:

$$LI = \frac{d50(L) - d50(S)}{d50(S)}$$

wherein d50(L) is the median particle size d50 [mass percent] determined by laser diffraction technique in accordance with ISO 13320-1 and d50(S) is the median particle size d50 [mass percent] as determined by sedimentation technique in accordance with ISO 13317-3.

4. The composition according to any one of the preceding claims, wherein the anti-blooming particulate material comprises methylsilsesquioxane.

5. The composition according to any one of the preceding claims, wherein (A) the polypropylene-based polymer is a propylene homopolymer or a propylene random copolymer consisting of at least 70 wt% of propylene monomer units and at most 30 wt% of α-olefin monomer units based on the propylene random copolymer.

6. The composition according to any one of the preceding claims, wherein the amount of (A) the polypropylene-based polymer is 75 to 95 wt%, for example 80 to 90 wt%, with respect to the total composition.

7. The composition according to any one of the preceding claims, wherein (B) the flame retardant is a halogenated flame retardant, preferably a brominated flame retardant, preferably selected from the group consisting of bis(2-hydroxyethyl)ether of tetrabromobisphenol A, bis(3-acryloyloxy-2-hydroxypropyl)ether of tetrabromobisphenol A, bis(3-methacryloyloxy-2-hydroxypropyl)ether of tetrabromobisphenol A, bis(3-hydroxypropyl)ether of tetrabromobisphenol A, bis(2,3-dibromopropyl)ether of tetrabromobisphenol A, diallyl ether of tetrabromobisphenol A, and bis(vinylbenzyl)ether of tetrabromobisphenol A; brominated polycarbonates, tetrabromobisphenol A polycarbonate oligomer, brominated polyacrylate such as polypentabromobenzyl acrylate; brominated polystyrenes, such as polydibromostyrenes and polytribromostyrenes; brominated BPA polyepoxides, tetrabromocyclooctanes; dibromoethyldibromocyclohexanes such as 1,2-dibromo-4-(1,2-dibromoethyl)-cyclohexane; ethylene-bis-tetrabromophthalimide; hexabromocyclododecanes; tetrabromophthalic anhydrides; brominated diphenylethers such as decabromodiphenyl ether; poly(2,6-dibromophenylene ether); tris(2,4,6-tribromophenoxy-1,3,5-triazine; tris(tribromoneopentyl)phosphate; and decabromodiphenyl ethane.

8. The composition according to any one of the preceding claims, wherein the amount of the component (B) is 1 to 25 wt%, preferably 3 to 20 wt%, more preferably 5 to 15 wt%, with respect to the total composition.

9. The composition according to any one of the preceding claims, wherein the total of components (A), (B) and (C) is at least 85 wt%, at least 90 wt% or at least 95 wt% of the total composition.

10. The composition according to any one of the preceding claims, wherein ($Gloss_0$-$Gloss_{24}$)/$Gloss_0$ is at most 0.30, more preferably at most 0.25, wherein $Gloss_0$ is a gloss of the composition as measured according to ASTM D2457-13 at 60 º after 0 hours and

Gloss_{24} is a gloss of the composition as measured according to ASTM D2457-13 at 60 º after 24 hours.

11. The composition according to any one of the preceding claims, wherein the composition has a gloss as measured according to ASTM D2457-13 at 60 º after 24 hours of conditioning at a temperature of 50 ºC (Gloss_{24}) of more than 60.

12. The composition according to any one of the preceding claims, wherein the composition has a flame retardancy of V0 according to the UL94 test standard at a sample thickness of 1.5 mm, wherein the sample is conditioned at 23 ºC ±2 ºC and 50 percent ± 10% relative humidity for 48 hours prior to testing, and/or the composition has a flame retardancy of V0 according to the UL94 test standard at a sample thickness of 1.5 mm, wherein the sample is conditioned at 70 ºC ±2 ºC for 168 hours ±2 hours and cooled in a desiccator for at least 48 hours prior to testing.

13. A process for the preparation of the composition according to any one of the preceding claims, comprising melt mixing (A), (B), and (C) and optional components.

14. An article comprising the composition according to any one of claims 1-12, preferably wherein the article is a toilet component.

INTERNATIONAL SEARCH REPORT

International application No
PCT/EP2019/074482

A. CLASSIFICATION OF SUBJECT MATTER
INV. C08L23/12
ADD.

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

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
C08L C08K

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)

EPO-Internal, WPI Data

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	EP 2 711 391 A1 (SAUDI BASIC IND CORP [SA]) 26 March 2014 (2014-03-26) paragraph [0063]; claims -----	1-14
X	US 2014/309350 A1 (KABEYA MASAYUKI [JP] ET AL) 16 October 2014 (2014-10-16) paragraphs [0013], [0032], [0034], [0036], [0043], [0082], [0086], [0087], [0090] - [0093], [0116] - [0118], [0125], [0126], [0140]; claims 2-4,10; table 2 -----	1-14
X	WO 2016/087309 A1 (SABIC GLOBAL TECHNOLOGIES BV [NL]) 9 June 2016 (2016-06-09) page 11, line 34; claims ----- -/--	1-14

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

☒ See patent family annex.

* Special categories of cited documents :

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

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

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

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

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

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

"&" document member of the same patent family

Date of the actual completion of the international search 5 December 2019	Date of mailing of the international search report 12/12/2019
Name and mailing address of the ISA/ European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Fax: (+31-70) 340-3016	Authorized officer Iraegui Retolaza, E

International application No
PCT/EP2019/074482

C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	EP 3 173 438 A1 (SABIC GLOBAL TECHNOLOGIES BV [NL]) 31 May 2017 (2017-05-31) paragraph [0015]; claims -----	1-14

1

INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No
PCT/EP2019/074482

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