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(71) Applicant: SABIC GLOBAL TECHNOLOGIES B.V.
[NL/NL]; Plasticslaan 1, 4612 PX Bergen Op Zoom (NL).

(72) Inventors: WANG, Huanbing; 2550 Xiupu Road, Pudong, Shanghai 201319 (CN). SHI, Hongtao; 2550 Xiupu Road, Pudong, Shanghai 201319 (CN).

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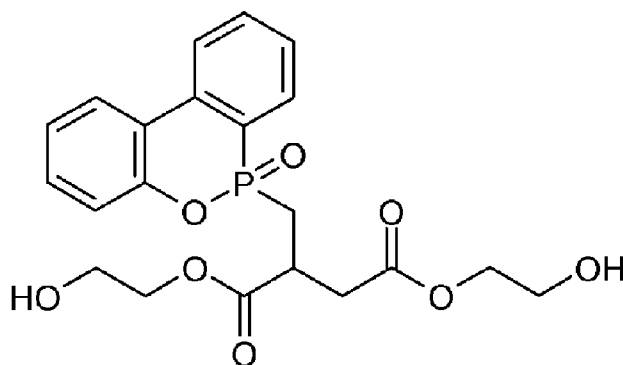
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(54) Title: POLYCARBONATE BLENDS WITH IMPROVED HEAT RESISTANCE



(I)

(57) Abstract: The disclosure concerns polycarbonate compositions comprising 55-90 wt% polycarbonate; 0-30 wt% filler; and 5-15 wt% flame retardant that comprises a homopolymer of (I) the composition having a heat deflection temperature (HDT) value of at least 100 °C at a load of 1.82 mPa on a 3.2 mm sample as measured according to ASTM D648.

POLYCARBONATE BLENDS WITH IMPROVED HEAT RESISTANCE

RELATED APPLICATIONS

[0001] The application claims benefit of U.S. Patent Application No. 62/150,593 filed on April 21, 2015, the disclosure of which is incorporated herein in its entirety.

TECHNICAL FIELD

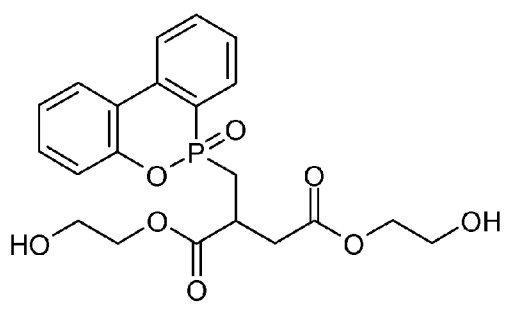
[0002] The disclosure concerns polycarbonate blends showing improved heat resistance.

BACKGROUND

[0003] Flame retardants like bisphenol A bis(diphenyl phosphate) (BPADP) or resorcinol bis(diphenyl phosphate) (RDP) are known to adversely affect the heat resistance of polymer compositions. In polycarbonate (PC) blends, a 2-4 °C reduction in heat deflection temperature (HDT) is typically observed with 1% phosphate flame retardant (FR) addition. The deterioration is particularly pronounced for PC and its blends versus many other polymer compositions. There is a need in the art for flame retardant PC compositions that maintain good heat deflection temperature.

SUMMARY

[0004] In some aspects the disclosure concerns polycarbonate compositions comprising: 55-90 wt% polycarbonate; 0-30 wt% filler; and 5-15 wt% flame retardant that comprises a homopolymer of



the composition having a heat deflection temperature (HDT) value of at least 100 °C at a load of 1.82 mPa on a 3.2 mm sample as measured according to ASTM D648; wherein all weight percent values are based on the total weight of the composition and the combined weight percent value of all components does not exceed 100 wt%. Certain polycarbonate compositions comprise a blend of polycarbonates. Some polycarbonate compositions have 10-30 wt% filler.

The polycarbonate composition may comprise a combination of (i) the homopolymer and (ii) a phosphate-containing flame retardant such as resorcinol diphosphate (RDP).

[0005] In other aspects, the disclosure concerns articles comprising polycarbonate compositions described herein.

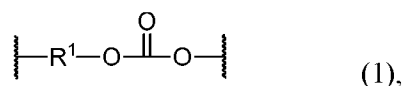
[0006] In addition, the disclosure concerns methods of making such polycarbonate compositions and articles comprising such polycarbonate compositions.

DETAILED DESCRIPTION OF ILLUSTRATIVE EMBODIMENTS

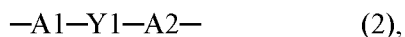
Polycarbonate Polymer

[0007] The terms "polycarbonate" or "polycarbonates" as used herein includes copolycarbonates, homopolycarbonates and (co)polyester carbonates. As used herein, the term "polycarbonate composition(s)" refers to polycarbonate-containing compositions of the instant invention.

[0008] The term polycarbonate can be further defined as having repeating structural units of the formula (1):



in which at least 60 percent of the total number of R1 groups are aromatic organic radicals and the balance thereof are aliphatic, alicyclic, or aromatic radicals. In a further aspect, each R1 is an aromatic organic radical and, more preferably, a radical of the formula (2):



wherein each of A1 and A2 is a monocyclic divalent aryl radical and Y1 is a bridging radical having one or two atoms that separate A1 from A2. In various aspects, one atom separates A1 from A2. For example, radicals of this type include, but are not limited to, radicals such as $-\text{O}-$, $-\text{S}-$, $-\text{S}(\text{O})-$, $-\text{S}(\text{O}_2)-$, $-\text{C}(\text{O})-$, methylene, cyclohexyl-methylene, 2-[2.2.1]-bicycloheptylidene, ethylidene, isopropylidene, neopentylidene, cyclohexylidene, cyclopentadecylidene, cyclododecylidene, and adamantylidene. The bridging radical Y1 is preferably a hydrocarbon group or a saturated hydrocarbon group such as methylene, cyclohexylidene, or isopropylidene. Polycarbonate materials include materials disclosed and described in U.S. Patent No. 7,786,246, which is hereby incorporated by reference in its entirety for the specific purpose of disclosing various polycarbonate compositions and methods for manufacture of the same.

[0009] The polycarbonate may be substantially free of halogen atoms. By “substantially free” it is intended that less than 1 wt % of the polycarbonate comprises halogen atoms.

[0010] A melt polycarbonate product may be utilized. The melt polycarbonate process is based on continuous reaction of a dihydroxy compound and a carbonate source in a molten stage. The reaction can occur in a series of reactors where the combined effect of catalyst, temperature, vacuum, and agitation allows for monomer reaction and removal of reaction by-products to displace the reaction equilibrium and effect polymer chain growth. A common polycarbonate made in melt polymerization reactions is derived from bisphenol A (BPA) via reaction with diphenyl carbonate (DPC). This reaction can be catalyzed by, for example, tetramethyl ammonium hydroxide (TMAOH) or tetrabutyl phosphonium acetate (TBPA), which can be added in to a monomer mixture prior to being introduced to a first polymerization unit and sodium hydroxide (NaOH), which can be added to the first reactor or upstream of the first reactor and after a monomer mixer.

[0011] The polycarbonate produced by interfacial polymerization may be utilized. In some processes, bisphenol A and phosgene are reacted in an interfacial polymerization process. Typically, the disodium salt of bisphenol A is dissolved in water and reacted with phosgene which is typically dissolved in a solvent that not miscible with water (such as a chlorinated organic solvent like methylene chloride).

[0012] It is preferred that the polycarbonate is present in an amount of about 55 to about 90 wt% based on the weight of the composition. The polycarbonate may be present in an amount of about 55 to about 85 wt% of the composition. The polycarbonate may be present in an amount of about 60 to about 90 wt%, or about 60 to about 85 wt%, or about 70 to about 85 wt%.

Filler

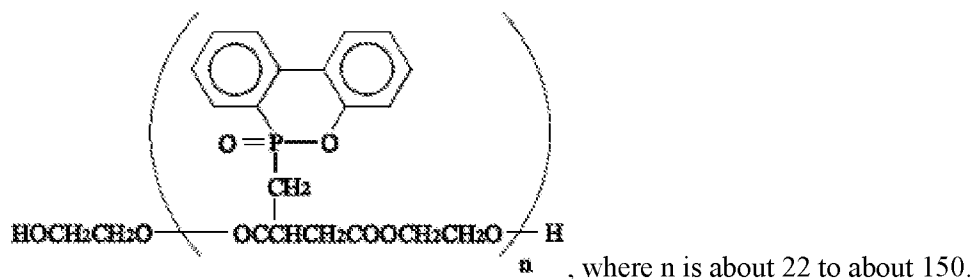
[0013] Possible fillers or reinforcing agents include, for example, mica, clay, feldspar, quartz, quartzite, perlite, tripoli, diatomaceous earth, aluminum silicate (mullite), synthetic calcium silicate, fused silica, fumed silica, sand, boron-nitride powder, boron-silicate powder, calcium sulfate, calcium carbonates (such as chalk, limestone, marble, and synthetic precipitated calcium carbonates) talc (including fibrous, modular, needle shaped, and lamellar talc), wollastonite, hollow or solid glass spheres, silicate spheres, cenospheres, aluminosilicate or (armospheres), kaolin, whiskers of silicon carbide, alumina, boron carbide, iron, nickel, or copper, continuous and chopped carbon fibers or glass fibers, molybdenum sulfide, zinc sulfide, barium titanate, barium ferrite, barium sulfate, heavy spar, TiO₂, aluminum oxide, magnesium

oxide, particulate or fibrous aluminum, bronze, zinc, copper, or nickel, glass flakes, flaked silicon carbide, flaked aluminum diboride, flaked aluminum, steel flakes, natural fillers such as wood flour, fibrous cellulose, cotton, sisal, jute, starch, lignin, ground nut shells, or rice grain husks, reinforcing organic fibrous fillers such as poly(ether ketone), polyimide, polybenzoxazole, poly(phenylene sulfide), polyesters, polyethylene, aromatic polyamides, aromatic polyimides, polyetherimides, polytetrafluoroethylene, and poly(vinyl alcohol), as well combinations comprising at least one of the foregoing fillers or reinforcing agents. The fillers and reinforcing agents can be coated with a layer of metallic material to facilitate conductivity, or surface treated with silanes to improve adhesion and dispersion with the polymer matrix.

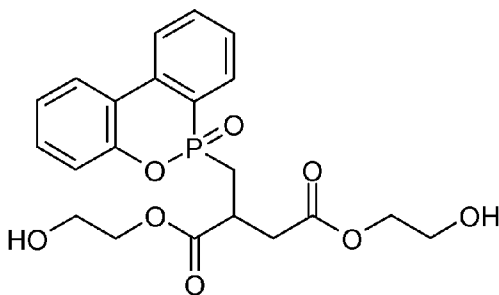
[0014] It is preferred that the filler is present in an amount of about 0 to about 30 wt% based on the weight of the composition. The filler is present may be present in an amount of about 1 to about 30 wt% of the composition or an amount of about 10 to about 30 wt% or an about 0 to about 20 wt% or about 5 to about 20 wt%.

Flame Retardant (FR)

[0015] Preferred flame retardants are of the formula:



This flame retardant can serve as the sole flame retardant or in combination with a secondary flame retardant. It is preferred that the flame retardant is present in an amount of about 5 to about 15 wt% based on the weight of the composition. The flame retardant may be present in an amount of about 7 to about 15 wt%, or about 8 to about 12 wt%. In one example, the above flame retardant is marketed under the trade name ME-P8 by Sanko and is a homopolymer of the compound pictured below.



[0016] The compound is sometimes referred to as butanedioic acid, (6H-dibenz[c,e][1,2]oxaphosphorin-6-ylmethyl), bis(2-hydroxyethyl)ester, P-oxide homopolymer. The homopolymer is denoted as CAS No. 403614-60-8 and 7.8-8.2 wt % P. The homopolymer may have a molecular weight (Mw) of about 3,000 to about 12,000; or about 10,000.

[0017] In addition to the above described flame retardants, additional flame retardants such as organic compounds that include phosphorus, bromine, and/or chlorine may be used. Non-brominated and non-chlorinated phosphorus-containing flame retardants can be preferred in certain applications for regulatory reasons, for example organic phosphates and organic compounds containing phosphorus-nitrogen bonds.

[0018] Additional flame retardants include phosphorus-containing flame retardants. Examples include phosphazene, aryl phosphate, bisphenol A disphosphate, resorcinol bis-diphenylphosphate, bisphenol A bis(diphenyl phosphate) (BABDP), or resorcinol diphosphate (RDP), or a combination thereof. Certain flame retardants are discussed in US 2014/0107266, which is incorporated herein in its entirety. A combination of flame retardants may show synergistic properties. Preferred second flame retardants include resorcinol-bis(diphenylphosphate) (RDP) and bisphenol-A diphenyl phosphate (DPADP).

[0019] Inorganic flame retardants can also be used, for example salts of C₁₋₁₆ alkyl sulfonate salts such as potassium perfluorobutane sulfonate (Rimar salt), potassium perfluorooctane sulfonate, tetraethylammonium perfluorohexane sulfonate, and potassium diphenylsulfone sulfonate. Salts such as Na₂CO₃, K₂CO₃, MgCO₃, CaCO₃, and BaCO₃, or fluoro-anion complexes such as Li₃AlF₆, BaSiF₆, KBF₄, K₃AlF₆, KAlF₄, K₂SiF₆, and/or Na₃AlF₆ may also be used. When present, inorganic flame retardant salts are present in amounts of 0.01 to 10 parts by weight, more specifically 0.02 to 1 parts by weight, based on 100 parts by weight of the total composition, excluding any filler.

Additional Components

[0020] The additive composition can include an impact modifier, flow modifier, antioxidant, heat stabilizer, light stabilizer, ultraviolet (UV) light stabilizer, UV absorbing additive, plasticizer, lubricant, release agent (such as a mold release agent), antistatic agent, anti-fog agent, antimicrobial agent, colorant (e.g. a dye or pigment), surface effect additive, radiation stabilizer, anti-drip agent (e.g., a PTFE-encapsulated styrene-acrylonitrile copolymer (TSAN)), or a combination comprising one or more of the foregoing. For example, a combination of a heat stabilizer, mold release agent, and ultraviolet light stabilizer can be used. In general, the

additives are used in the amounts generally known to be effective. For example, the total amount of the additive composition can be 0.001 to 10.0 wt%, or 0.01 to 5 wt%, each based on the total weight of all ingredients in the composition.

[0021] The polycarbonate composition can include various additives ordinarily incorporated into polymer compositions of this type, with the proviso that the additive(s) are selected so as to not significantly adversely affect the desired properties of the thermoplastic composition (good compatibility for example). Such additives can be mixed at a suitable time during the mixing of the components for forming the composition.

[0022] Examples of impact modifiers include natural rubber, fluoroelastomers, ethylene-propylene rubber (EPR), ethylene-butene rubber, ethylene-propylene-diene monomer rubber (EPDM), acrylate rubbers, hydrogenated nitrile rubber (HNBR), silicone elastomers, styrene-butadiene-styrene (SBS), styrene-butadiene rubber (SBR), styrene-(ethylene-butene)-styrene (SEBS), acrylonitrile-butadiene-styrene (ABS), acrylonitrile-ethylene-propylene-diene-styrene (AES), styrene-isoprene-styrene (SIS), styrene-(ethylene-propylene)-styrene (SEPS), methyl methacrylate-butadiene-styrene (MBS), high rubber graft (HRG), and the like. Some suitable impact modifiers include PC(polycarbonate)/ABS (such as Cycloy PC/ABS) and MBS type formulations.

[0023] Heat stabilizer additives include organophosphites (e.g. triphenyl phosphite, tris-(2,6-dimethylphenyl)phosphite, tris-(mixed mono-and di-nonylphenyl)phosphite or the like), phosphonates (e.g. dimethylbenzene phosphonate or the like), phosphates (e.g., trimethyl phosphate, or the like), or combinations comprising at least one of the foregoing heat stabilizers. The heat stabilizer can be tris(2,4-di-t-butylphenyl) phosphate available as IRGAPHOSTM 168. Heat stabilizers are generally used in amounts of 0.01 to 5 wt%, based on the total weight of polymer in the composition.

[0024] There is considerable overlap among plasticizers, lubricants, and mold release agents, which include, for example, glycerol tristearate (GTS), phthalic acid esters (e.g. octyl-4,5-epoxy-hexahydrophthalate), tris-(octoxycarbonyl)isocyanurate, tristearin, di- or polyfunctional aromatic phosphates (e.g. resorcinol tetraphenyl diphosphate (RDP), the bis(diphenyl) phosphate of hydroquinone and the bis(diphenyl) phosphate of bisphenol A); poly-alpha-olefins; epoxidized soybean oil; silicones, including silicone oils (e.g., poly(dimethyl diphenyl siloxanes); esters, for example, fatty acid esters (e.g. alkyl stearyl esters, such as, methyl stearate, stearyl stearate, and the like), waxes (e.g. beeswax, montan wax, paraffin wax, or the like), or combinations comprising at least one of the foregoing plasticizers, lubricants, and

mold release agents. These are generally used in amounts of 0.01 to 5 wt%, based on the total weight of the polymer in the composition.

[0025] Light stabilizers, in particular ultraviolet light (UV) absorbing additives, also referred to as UV stabilizers, include hydroxybenzophenones (e.g., 2-hydroxy-4-n-octoxy benzophenone), hydroxybenzotriazines, cyanoacrylates, oxanilides, benzoxazinones (e.g., 2,2'-(1,4- phenylene)bis(4H-3,1-benzoxazin-4-one, commercially available under the trade name CYASORB UV-3638 from Cytec), aryl salicylates, hydroxybenzotriazoles (e.g., 2-(2-hydroxy-5-methylphenyl)benzotriazole, 2-(2-hydroxy-5-tert-octylphenyl)benzotriazole, and 2-(2H-benzotriazol-2-yl)-4-(1,1,3,3-tetramethylbutyl)-phenol, commercially available under the trade name CYASORB 5411 from Cytec) or combinations comprising at least one of the foregoing light stabilizers. The UV stabilizers can be present in an amount of 0.01 to 1 wt%, specifically, 0.1 to 0.5 wt%, and more specifically, 0.15 to 0.4 wt%, based upon the total weight of polymer in the composition.

[0026] Antioxidant additives include organophosphites such as tris(nonyl phenyl)phosphite, tris(2,4-di-t-butylphenyl)phosphite, bis(2,4-di-t-butylphenyl)pentaerythritol diphosphite, distearyl pentaerythritol diphosphite; alkylated monophenols or polyphenols; alkylated reaction products of polyphenols with dienes, such as tetrakis[methylene(3,5-di-tert-butyl-4-hydroxyhydrocinnamate)] methane; butylated reaction products of para-cresol or dicyclopentadiene; alkylated hydroquinones; hydroxylated thiodiphenyl ethers; alkylidene-bisphenols; benzyl compounds; esters of beta-(3,5-di-tert-butyl-4-hydroxyphenyl)-propionic acid with monohydric or polyhydric alcohols; esters of beta-(5-tert-butyl-4-hydroxy-3-methylphenyl)-propionic acid with monohydric or polyhydric alcohols; esters of thioalkyl or thioaryl compounds such as distearylthiopropionate, dilaurylthiopropionate, ditridecylthiodipropionate, octadecyl-3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate, pentaerythrityl-tetrakis[3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate; amides of beta-(3,5-di-tert-butyl-4-hydroxyphenyl)-propionic acid, or combinations comprising at least one of the foregoing antioxidants. Antioxidants are used in amounts of 0.01 to 0.1 parts by weight, based on 100 parts by weight of the total composition, excluding any filler.

[0027] Anti-drip agents can also be used in the composition, for example a fibril forming or non-fibril forming fluoropolymer such as polytetrafluoroethylene (PTFE). The anti-drip agent can be encapsulated by a rigid copolymer, for example styrene-acrylonitrile copolymer (SAN). PTFE encapsulated in SAN is known as TSAN. A TSAN comprises 50 wt% PTFE and 50 wt% SAN, based on the total weight of the encapsulated fluoropolymer. The SAN

can comprise, for example, 75 wt% styrene and 25 wt% acrylonitrile based on the total weight of the copolymer. Antidrip agents can be used in amounts of 0.1 to 10 parts by weight, based on 100 parts by weight of the total composition, excluding any filler.

Polycarbonate Compositions

[0028] The polycarbonate compositions can be manufactured by various methods known in the art. For example, powdered polycarbonate, and other components are first blended, optionally with any fillers, in a high speed mixer or by hand mixing. The blend can then be fed into the throat of a twin-screw extruder via a hopper. Alternatively, at least one of the components can be incorporated into the composition by feeding it directly into the extruder at the throat and/or downstream through a sidestuffer, or by being compounded into a masterbatch with a desired polymer and fed into the extruder. The extruder is generally operated at a temperature higher than that necessary to cause the composition to flow. The extrudate can be immediately quenched in a water bath and pelletized.

[0029] It is preferred that the polycarbonate compositions have a heat deflection temperature (HDT) value of at least 100 °C at a load of 1.82 mPa on a 3.2 mm sample as measured according to ASTM D648.

Articles of Manufacture

[0030] In one aspect, the present disclosure pertains to shaped, formed, or molded articles comprising polycarbonate compositions described herein. The polycarbonate compositions can be molded into useful shaped articles by a variety of means such as injection molding, extrusion, rotational molding, blow molding and thermoforming to form articles. The polycarbonate compositions described herein can also be made into film and sheet as well as components of laminate systems. In a further aspect, a method of manufacturing an article comprises melt blending the polycarbonate component, the liquid polymer, catalyst and any optional ingredients; and molding the extruded composition into an article. In a still further aspect, the extruding is done with a twin-screw extruder.

[0031] In a further aspect, the article comprising the disclosed polycarbonate compositions is used in automotive applications. In a still further aspect, the article can be selected from computer and business machine housings such as housings for monitors, handheld electronic device housings such as housings for cell phones, electrical connectors, medical devices, membrane devices, and components of lighting fixtures, ornaments, home appliances,

roofs, greenhouses, sun rooms, swimming pool enclosures, and the like. Other representative articles that may be fabricated using the disclosed copolymer polycarbonate compositions provided herein include headlamp bezels, headlamps, tail lamps, tail lamp housings, tail lamp bezels, license plate enclosures, enclosures for electrical and telecommunication devices, building and construction applications such as glazing, roofs, windows, floors, decorative window furnishings or treatments; treated glass covers for pictures, paintings, posters, and like display items; optical lenses; ophthalmic lenses; corrective ophthalmic lenses; implantable ophthalmic lenses; wall panels, and doors; protected graphics; outdoor and indoor signs; enclosures, housings, panels, and parts for automatic teller machines (ATM); enclosures, computer housings; desk-top computer housings; portable computer housings; lap-top computer housings; palm-held computer housings; monitor housings; printer housings; keyboards; FAX machine housings; copier housings; telephone housings; mobile phone housings; radio sender housings; radio receiver housings; light fixtures; lighting appliances; network interface device housings; and like applications. In a still further aspect, the article used in automotive applications is selected from instrument panels, overhead consoles, interior trim, center consoles, headlamp bezels, headlamps, tail lamps, tail lamp housings, tail lamp bezels, license plate enclosures, steering wheels, radio speaker grilles, mirror housings, grille opening reinforcements, steps, hatch covers, knobs, buttons, and levers. Additional fabrication operations may be performed on articles, such as, but not limited to molding, in-mold decoration, baking in a paint oven, lamination, and/or thermoforming.

[0032] In various aspects, the article comprising the disclosed copolymer polycarbonate compositions are suitable for use in applications such as transparent keypads for mobile phones. Other typical such articles are automotive trim, automotive interior parts, portable telecommunications and appliance fronts. In a further aspect, wherein the article is a film, it can further comprise visual effects pigments (such as coated Al and glass flakes). In a still further aspect, the article is a film comprising a disclosed copolymer composition can be used in direct film applications but also in processes like IMD (In Mould Decoration). In an even further aspect, the article comprising a disclosed copolymer composition is used in lighting applications including automotive headlamp lenses, covers and lenses for other optical devices, as well as transparent films and sheets. The article can also be used in a wide variety of molded products such as medical devices, radio and TV bezels, mobile phone keypads, notebook computer housings and keys, optical display films, automotive parts, and other electronic and consumer products.

[0033] Polymers described herein are suited for use in electronic devices, for the production of thin-walled parts, like housings for cell phones and for automotive applications.

Examples

[0034] The invention is illustrated by the following non-limiting examples.

[0035] The molded samples disclosed herein were tested in accordance with the standards below.

[0036] Melt volume - flow rate ("MFR") was determined according to standard ASTM D1238 under the following test conditions: 300 °C/1.2 kg load/1080 sec dwell time or 300 °C/1.2 kg load/360 sec dwell time or 260 °C/5 kg load or 260 °C/2.16 kg load. Data below are provided for MVR in g³/10 min.

[0037] The notched Izod impact ("NII") test was carried out on 63.5 mm x 12.7 mm x 3.2 mm molded samples (bars) according to ASTM D256 at 0 °C and 23 °C. Data units are J/m.

[0038] Tensile properties were measured a Tensile Type 1 bar (50 mm x 13 mm) in accordance with ASTM D638 using sample bars prepared in accordance with a Tensile Type 1 bar (50 mm x 13 mm). Tensile strength for either at break or at yield is reported in units of MPa.

[0039] Heat deflection temperature (HDT) was determined per ASTM D648 with flatwise specimen orientation with specimen dimensions of 127 mm x 12.7 mm x 3.2 mm at 1.82 MPa. Data are provided below in units of °C.

[0040] Flex modulus and flex strength were measured according to Flex; ISO 178.

[0041] Unless otherwise stated to the contrary herein, all test standards are the most recent standard in effect at the time of filing this application.

[0042] Table 1 summarizes various components used in the examples. Test methods are summarized in the definitions presented herein.

Table 1: Various components used in the illustrative examples.

No.	Item	Description	Supplier	Trade Name
1	PC1	BPA polycarbonate resin made by an interfacial process with Mw of about 29,900. CAS No. 111211-39-3	SABIC Innovative Plastics (Sabic I.P.)	LEXAN TM
2	PC2	BPA polycarbonate resin made by an interfacial process with Mw of about	Sabic I.P.	LEXAN TM

		12,800. CAS No. 111211-39-3		
3	PC3	BPA polycarbonate-polysiloxane copolymer comprising about 20 wt% siloxane, 80 wt% BPA and end-capped	Sabic I.P.	LEXAN TM -EXL
4	IM1	Bulk acrylonitrile-butadiene-styrene comprising about 16-17 wt% butadiene content (Grade C2449), CAS No. 9003-56-9	Sabic I.P.	C29449
5	IM2	High rubber graft emulsion polymerized ABS comprising about 50 wt% polybutadiene, CAS No. 9003-56-9	Kumho	HR 181
6	SAN	Bulk San. CAS No. 9003-54-7	Kumho	SAN 320
7	FR1	Bisphenol A bis(diphenylphosphate). CAS No. 5945-33-5	Dahaichi Chemical Industry Co, Ltd.	BPA-DP/CR-741
8	FR2	Recorcinol diphosphate. CAS No. 57583-54-7	Fyroflex	RDP
9	FR3	9,10-Dihydro-9-oxa-10-phosphenanthrene-10-oxide. CAS No. 35948-25-5	Sanko	DOPO
10	FR4	Butanedioic acid, (6H-dibenz[c,e][1,2]oxaphosphorin-6-ylmethyl), bis(2-hydroxyethyl)ester, P-oxide homopolymer. CAS No. 403614-60-8	Sanko	ME-P8
11	F1	Non-bonding chopped glass fiber. CAS No. 65997-17-3	Owens Corning	415A-14C
12	ADD1	Polytetrafluoroethylene (PTFE) encapsulated by styrene-acrylonitrile copolymer (SAN). Anti-drip. CAS No. 9002-84-0	SABIC I.P.	TSAN
13	ADD2	Pentaerythritol tetrastearate, a mold release agent. CAS No. 115-83-3	FACI Farsco Genova, Italy	PETS

14	ADD3	Hindered phenol, Irganox 1076. CAS No. 2082-79-3	BASF	Irganox TM 1076
15	ADD4	Tris(2,4-di-tert-butylphenyl)phosphite , stabilizer, CAS NO. 31570.04-4	BASF	Irgafos TM 168

[0043] Table 2 shows the properties of unfilled PC blends. In comparison with C1 including 10% phosphate flame retardant (FR) BPADP and C2 including 6.3% DOPO, W1 with 11.25% ME-P8 loading (conversion according to P% content) showed higher heat resistance. 4 than both BPADP and DOPO. The HDT was improved from 89.6 to 107 °C if BPADP was replaced by ME-P8 with the same phosphorous (P) content loading.

[0044] The key purpose of a flame retradant additive is to improve flame retardancy. Importantly, in UL 2.0 mm Vx test for FR performance, no significant difference was observed for the samples.

[0045] The inventive mixture also benefits flow. For example, DOPO degrades PC and flow was increased too high as a result. As such, most mechanical properties were decreased in C2 as the Mw of the PC resin dropped. Mw of PC showed a small drop in W1. The flow was maintained. The modulus of W1 was comparable to C1.

[0046] Concurrent with the drop in the Mw of PC, C2 showed considerably poorer mechanical properties than W1. In comparison with C1, notched Izod impact at 23 °C of W1 with ME-P8 was decreased but the ductile-brittle (D/B) transition of W1 was not as sensitive as C1. In the NII test at 0 °C, W1 showed 20% ductility while C1 showed 0 ductility.

Table 2. Formulation and Properties of unfilled PC blends

		Unit	C1	C2	W1
FORMULATION					
1	PC2	%	35.77	37.62	35.15
2	PC1	%	35.77	37.62	35.15
3	PC3	%	14	14	14
4	IM1	%	3	3	3
5	ADD1	%	1	1	1
6	ADD2	%	0.08	0.08	0.08
7	ADD3	%	0.3	0.3	0.3
8	ADD4	%	0.08	0.08	0.08
9	FR1	%	10		
10	FR3	%		6.3	
11	FR4	%			11.25
Formulation Total		%	100	100	100
PROPERTIES					
HDT, 1.82MPa/3.2mm		°C	89.6	88.4	107
MFR 260°C/2.16KG		g/10min	10.2	999	9
flex Modulus		Mpa	2490	2700	2360
Flex stress yld		MPa	96	48	89
Tens Stress brk		Mpa	66	46	58
NII, 23°C		J/m	816	22	139
Ductility		%	100	0	20
NII, 0°C		J/M	130	22	133
Ductility		%	0	0	20
V0@2.0mm	FOT2	s	1.02	1.48	2.59
	DRIP		0	0	0
GPC	Mw	D	51873	25261	48961
	Mn	D	19458	10968	17229
	d		2.67	2.3	2.84

[0047] Table 3 shows the properties of glass fiber (GF) filled PC blends with DOPO (C1), RDP (C2), RDP/ME-P8 combination (W1-W4) and ME-P8 (W5). The combination of different types of FR (e.g., RDP/ME-P8) showed synergy in some cases. In the comparisons, the total P% is fixed but the ratio between phosphate type FR RDP and phosphinate type FR ME-P8 is adjusted.

[0048] In comparison with C1 including 6.42% DOPO and C2 including 8.5% RDP, W5 with 11.5% ME-P8 showed higher HDT. The HDT was improved from 99.8 (C1) and 94.3 (C2) to 123 °C (W5). Flow, modulus, and impact were comparable between the samples. FR of W5 with ME-P8 was worse than C1 with RDP. The flame out time (FOT) was changed from 5.85 to 14.9 s at 1.5 mm bars test using UL-94 protocol.

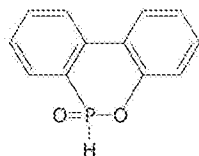
[0049] Samples having the combination of RDP and ME-P8 showed better balance of properties. W1-W5 are samples with RDP/ME-P8 (P%) ratios of 90/10, 75/25, 50/50, and 25/75, respectively. In comparison with C1, W1-W5 showed better mechanical properties. In comparison with C2, W1-W5 showed superior HDT properties while other properties were similar with C1. With ME-P8 increasing, the heat resistance improved and other properties were maintained.

Table 3. Formulation and properties of filled PC blends

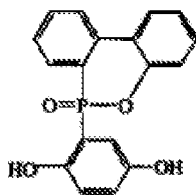
		Unit	C1	C2	W1	W2	W3	W4	W5
FORMULATION									
1	PC2	%	33.16	32.12	31.96	31.7375	31.365	30.9925	30.62
2	PC1	%	33.16	32.12	31.96	31.7375	31.365	30.9925	30.62
3	IM2	%	3.52	3.52	3.52	3.52	3.52	3.52	3.52
4	SAN	%	2.48	2.48	2.48	2.48	2.48	2.48	2.48
5	F1	%	20	20	20	20	20	20	20
6	ADD1	%	0.85	0.85	0.85	0.85	0.85	0.85	0.85
7	ADD2	%	0.08	0.08	0.08	0.08	0.08	0.08	0.08
8	ADD3	%	0.25	0.25	0.25	0.25	0.25	0.25	0.25
9	ADD4	%	0.08	0.08	0.08	0.08	0.08	0.08	0.08
10	FR3	%	6.42						
11	FR2	%		8.5	7.67	6.39	4.26	2.13	
12	FR4	%			1.15	2.875	5.75	8.625	11.5
Formulation Total		%		100	100	100	100	100	100
PROPERTIES									
HDT, 1.82mpa/3.2mm		°C	99.8	94.3	97.2	101	105	116	123
MFR 260°C/2.16KG		g/10min	95.9	6.3	6.7	6	4.9	4.6	7.8
Flex Modulus		Mpa	5190	6040	6190	6110	6170	6090	6130
Flex stress, YLD		MPa	30	103	109	108	109	101	93.5
Tens stress BRK		Mpa	20	62	62	62	63	64	57
Tens elong.brk		%	0.25	4	3.4	3.6	3.4	3.1	1.8
NII, 23°C		J/m	24	83	90	93	95	84	64
Ductility		%	0	0	0	0	0	0	0
NII, 0°C		J/M	26	56	57	59	60	65	56
Ductility		%	0	0	0	0	0	0	0
1.5MM*	FOT2	s	x*	5.9	3.8	3.4	3.8	5.1	14.9
	DRIP		x*	0	0	0	0	0	0
2MM*	FOT2	s	x*	2.4	2.5	3.1	3.1	4.2	14.6
	DRIP		x*	0	0	0	0	0	0

* The UL bars cannot be molded since too crisp.

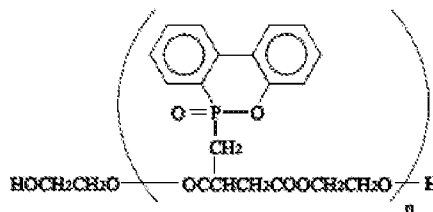
[0050] Table 4 shows results including C3 which contains HCA-HQ as flame retardant, C2 which has DOPO-HCA as a flame retardant, and W6 having ME-P8 as a flame retardant. W6 shows improved HDT, MFR, and Notched Izod results.



DOPO HCA



HCA-HQ



ME-P8

Table 4. Formulation and properties of filled PC blends

		Unit	C1	C2	W6	with HCA-HQ
FORMULATION						
1	PC2	%	35.77	37.62	35.15	36.07
2	PC1	%	35.77	37.62	35.15	36.07
3	PC3	%	14	14	14	14
4	IM1	%	3	3	3	3
5	ADD1	%	1	1	1	1
6	ADD2	%	0.08	0.08	0.08	0.08
7	ADD3	%	0.3	0.3	0.3	0.3
8	ADD4	%	0.08	0.08	0.08	0.08
9	FR1 BPADP	%	10			
10	FR3 DOPO(HCA)	%		6.3		
11	FR4 DOPO derivative (HCA-HQ)	%				9.4
12	FR5 (ME-P8)	%			11.25	
Formulation Total		%	100	100	100	100
PROPERTIES						
HDT, 1.82MPa/3.2mm		°C	89.6	88.4	107	96.4
MFR 260°C/2.16KG		g/10min	10.2	999	9	28.9
flex Modulus		Mpa	2490	2700	2360	2660
Flex stress yld		MPa	96	48	89	107
Tens Stress brk		Mpa	66	46	58	53
NII, 23°C		J/m	816	22	139	91
Ductility		%	100	0	20	0
NII, 0°C		J/M	130	22	133	85
Ductility		%	0	0	20	0
VO@2.0 mm	FOT2	s	1.02	1.48	2.59	1.23
	DRIP		0	0	0	0
GPC	Mw	D	51873	25261	48961	48099
	Mn	D	19458	10968	17229	18246

	D		2.67	2.3	2.84	2.64
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Aspects

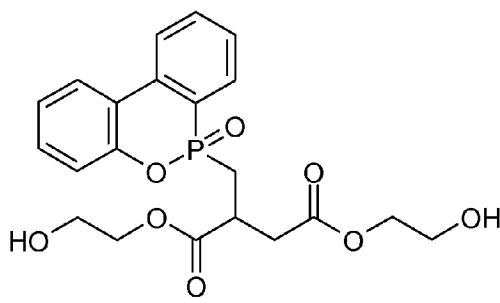
[0051] The present disclosure comprises at least the following aspects.

[0052] Aspect 1. A composition comprising:

55-90 wt% polycarbonate;

0-30 wt% filler; and

5-15 wt% flame retardant that comprises a homopolymer of



the composition having a heat deflection temperature (HDT) value of at least 100 °C at a load of 1.82 mPa on a 3.2 mm sample as measured according to ASTM D648;

wherein all weight percent values are based on the total weight of the composition and the combined weight percent value of all components does not exceed 100 wt%.

[0053] Aspect 2. The composition of Aspect 1 comprising a blend of at least two polycarbonates.

[0054] Aspect 3. The composition of Aspect 1 or Aspect 2, comprising 55-85 wt% of polycarbonate and 10-30 wt% filler.

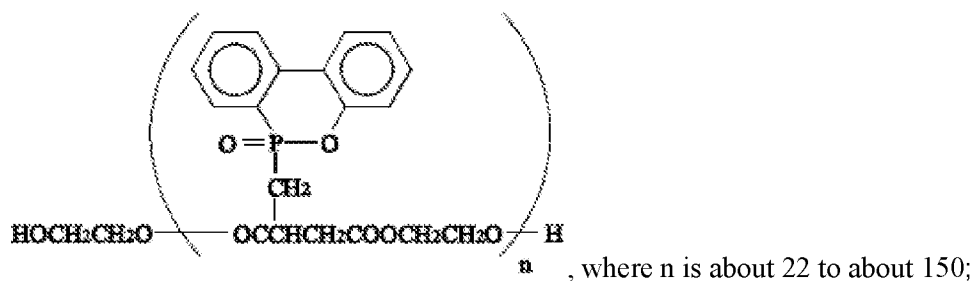
[0055] Aspect 4. The composition of Aspect 1 or Aspect 2, comprising:

55-85 wt% polycarbonate;

1-30 wt% filler; and

5-15 wt% flame retardant, the flame retardant comprising

(i) the homopolymer having the formula



(ii) 0-8 wt% of a second phosphate-containing flame retardant.

[0056] Aspect 5. The composition of Aspect 4, comprising about 1 to about 11 wt% of the homopolymer and about 2 to about 8 wt% of the second phosphate-containing flame retardant.

[0057] Aspect 6. The composition of any one of Aspects 1-5, comprising 70-85 wt% polycarbonate.

[0058] Aspect 7. The composition of claim 5 or claim 6, comprising about 2 to about 8 wt% of the second phosphate-containing flame retardant.

[0059] Aspect 8. The composition of claim 5 or claim 7, comprising about 3 to about 11 wt% of the homopolymer and about 3 to about 6 wt% of the second phosphate-containing flame retardant.

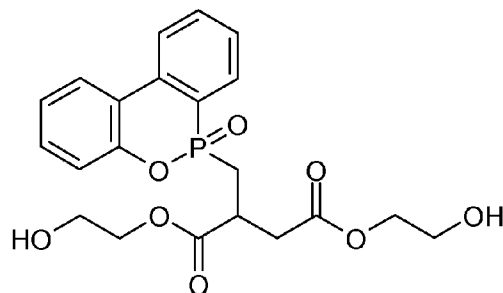
[0060] Aspect 9. The composition of any one of Aspects 1-8 having a notched Izod impact (NII) value measured at 23 °C of at least 84 J/m when measured by ASTM D256.

[0061] Aspect 10. The composition of any one of Aspects 1-8 having a notched Izod impact (NII) value measured at 0 °C of at least 57 J/m when measured by ASTM D256.

[0062] Aspect 11. The composition of any one of Aspects 4-10, wherein the second phosphate-containing flame retardant comprises resorcinol diphosphate.

[0063] Aspect 12. The composition of any one of Aspects 1-11, wherein the filler comprises glass.

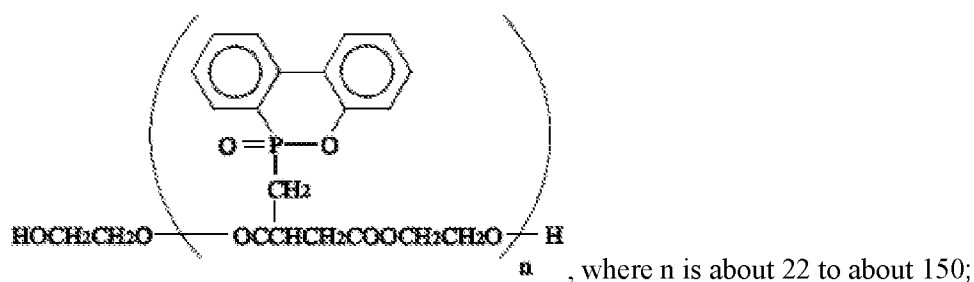
[0064] Aspect 13. A method of forming a composition comprising blending 55-90 wt% polycarbonate; 0-30 wt% filler; and 5-15 wt% flame retardant comprising a homopolymer of the compound of the formula



to produce a composition having a heat deflection temperature (HDT) value of at least 100 °C at a load of 1.82 mPa on a 3.2 mm sample as measured according to ASTM D648; wherein all weight percent values are based on the total weight of the composition and the combined weight percent value of all components does not exceed 100 wt%.

[0065] Aspect 14. The method of Aspect 13, wherein the flame retardant comprises

(i) the homopolymer having the formula



(ii) 0-8 wt% of a second phosphate-containing flame retardant.

[0066] Aspect 15. The method of Aspect 14, comprising about 1 to about 11 wt% of the homopolymer and about 2 to about 8 wt% of the second phosphate-containing flame retardant.

[0067] Aspect 16. The method of Aspect 14 or Aspect 15, wherein the second phosphate-containing flame retardant comprises resorcinol diphosphate.

[0068] Aspect 17. The method of any one of Aspects 13-16, comprising 70-85 wt% polycarbonate.

[0069] Aspect 18. An article comprising the composition of any one of Aspects 1-12.

[0070] Aspect 19. The article of Aspect 18, wherein the article is wherein the article is a component in business equipment, a monitor, a portable electronic device or an automobile.

[0071] Aspect 20. A work piece comprising the composition of any one of Aspects 1-12.

Definitions

[0072] It is to be understood that the terminology used herein is for the purpose of describing particular aspects only and is not intended to be limiting. As used in the specification and in the claims, the term “comprising” can include the embodiments “consisting of” and “consisting essentially of.” Unless defined otherwise, all technical and scientific terms used herein have the same meaning as commonly understood by one of ordinary skill in the art to which this disclosure belongs. In this specification and in the claims which follow, reference will be made to a number of terms which shall be defined herein.

[0073] As used in the specification and the appended claims, the singular forms “a,” “an” and “the” include plural equivalents unless the context clearly dictates otherwise. Thus, for example, reference to “a polycarbonate polymer” includes mixtures of two or more polycarbonate polymers.

[0074] As used herein, the term “combination” is inclusive of blends, mixtures, alloys, reaction products, and the like.

[0075] Ranges can be expressed herein as from one particular value to another particular value. When such a range is expressed, another aspect includes from the one particular value and/or to the other particular value. Similarly, when values are expressed as approximations, by use of the antecedent ‘about,’ it will be understood that the particular value forms another aspect. It will be further understood that the endpoints of each of the ranges are significant both in relation to the other endpoint, and independently of the other endpoint. It is also understood that there are a number of values disclosed herein, and that each value is also herein disclosed as “about” that particular value in addition to the value itself. For example, if the value “10” is disclosed, then “about 10” is also disclosed. It is also understood that each unit between two particular units are also disclosed. For example, if 10 and 15 are disclosed, then 11, 12, 13, and 14 are also disclosed.

[0076] As used herein, the terms “about” and “at or about” mean that the amount or value in question can be the value designated some other value approximately or about the same. It is generally understood, as used herein, that it is the nominal value indicated $\pm 5\%$ variation unless otherwise indicated or inferred. The term is intended to convey that similar values promote equivalent results or effects recited in the claims. That is, it is understood that amounts, sizes, formulations, parameters, and other quantities and characteristics are not and need not be exact, but can be approximate and/or larger or smaller, as desired, reflecting tolerances, conversion factors, rounding off, measurement error and the like, and other factors known to

those of skill in the art. In general, an amount, size, formulation, parameter or other quantity or characteristic is “about” or “approximate” whether or not expressly stated to be such. It is understood that where “about” is used before a quantitative value, the parameter also includes the specific quantitative value itself, unless specifically stated otherwise.

[0077] Disclosed are the components to be used to prepare the polycarbonate compositions of the disclosure as well as the compositions themselves to be used within the methods disclosed herein. These and other materials are disclosed herein, and it is understood that when combinations, subsets, interactions, groups, etc. of these materials are disclosed that while specific reference of each various individual and collective combinations and permutation of these compounds cannot be explicitly disclosed, each is specifically contemplated and described herein. For example, if a particular compound is disclosed and discussed and a number of modifications that can be made to a number of molecules including the compounds are discussed, specifically contemplated is each and every combination and permutation of the compound and the modifications that are possible unless specifically indicated to the contrary. Thus, if a class of molecules A, B, and C are disclosed as well as a class of molecules D, E, and F and an example of a combination molecule, A-D is disclosed, then even if each is not individually recited each is individually and collectively contemplated meaning combinations, A-E, A-F, B-D, B-E, B-F, C-D, C-E, and C-F are considered disclosed. Likewise, any subset or combination of these is also disclosed. Thus, for example, the sub-group of A-E, B-F, and C-E would be considered disclosed. This concept applies to all aspects of this application including, but not limited to, steps in methods of making and using the compositions of the disclosure. Thus, if there are a variety of additional steps that can be performed it is understood that each of these additional steps can be performed with any specific aspect or combination of aspects of the methods of the disclosure.

[0078] References in the specification and concluding claims to parts by weight, of a particular element or component in a composition or article, denotes the weight relationship between the element or component and any other elements or components in the composition or article for which a part by weight is expressed. Thus, in a compound containing 2 parts by weight of component X and 5 parts by weight component Y, X and Y are present at a weight ratio of 2:5, and are present in such ratio regardless of whether additional components are contained in the compound.

[0079] As used herein the terms “weight percent,” “wt. %,” and “wt.%” of a component, which can be used interchangeably, unless specifically stated to the contrary, are

based on the total weight of the formulation or composition in which the component is included. For example if a particular element or component in a composition or article is said to have 8% by weight, it is understood that this percentage is relative to a total compositional percentage of 100% by weight.

[0080] The abbreviation “mm” stands for millimeters.

[0081] The abbreviation “°C” means degrees Celsius.

[0082] The abbreviation “kg” stands for kilograms.

[0083] The abbreviation “sec” stands for the time unit second(s).

[0084] The abbreviation “MPa” means megapascal.

[0085] “J” is the abbreviation for Joule(s).

[0086] The abbreviation “M” or “m” stands for meter(s).

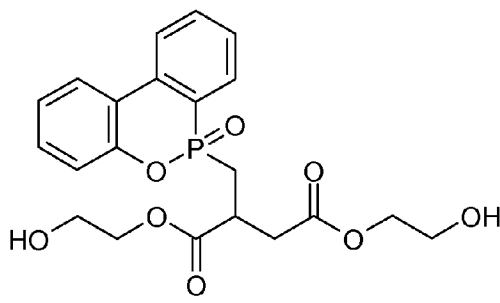
[0087] As used herein, the terms “weight average molecular weight” or “Mw” can be used interchangeably, and are defined by the formula:

$$M_w = \frac{\sum N_i M_i^2}{\sum N_i M_i},$$

where M_i is the molecular weight of a chain and N_i is the number of chains of that molecular weight. Compared to M_n , M_w takes into account the molecular weight of a given chain in determining contributions to the molecular weight average. Thus, the greater the molecular weight of a given chain, the more the chain contributes to the M_w . M_w can be determined for polymers, e.g. polycarbonate polymers, by methods well known to a person having ordinary skill in the art using molecular weight standards, e.g. polycarbonate standards or polystyrene standards, preferably certified or traceable molecular weight standards.

What is claimed:

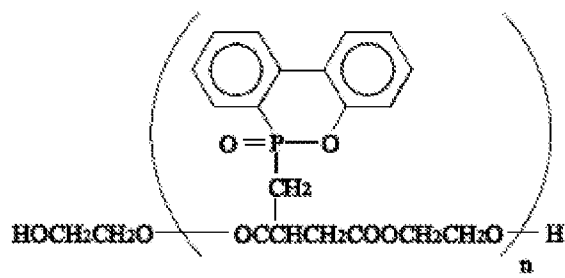
1. A composition comprising:
 - 55-90 wt% polycarbonate;
 - 0-30 wt% filler; and
 - 5-15 wt% flame retardant that comprises a homopolymer of



the composition having a heat deflection temperature (HDT) value of at least 100 °C at a load of 1.82 mPa on a 3.2 mm sample as measured according to ASTM D648;

wherein all weight percent values are based on the total weight of the composition and the combined weight percent value of all components does not exceed 100 wt%.

2. The composition of claim 1, comprising a blend of at least two polycarbonates.
3. The composition of claim 1 or claim 2, comprising:
 - 55-85 wt% of polycarbonate and
 - 10-30 wt% filler.
4. The composition of any one of claims 1-3, comprising:
 - 55-85 wt% polycarbonate;
 - 1-30 wt% filler; and
 - 5-15 wt% flame retardant, the flame retardant comprising
 - (i) the homopolymer having the formula

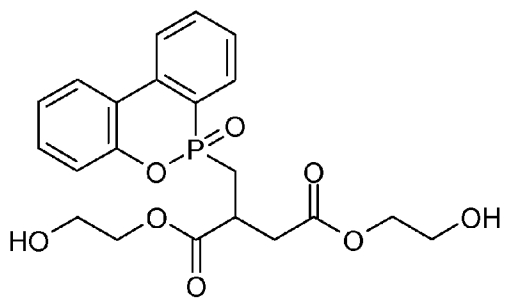


, where n is about 22 to about 150; and

(ii) 0-8 wt% of a second phosphate-containing flame retardant.

5. The composition of any one of claims 1-5, comprising about 70 wt% to about 85 wt% of polycarbonate.
6. The composition of claim 4, comprising about 2 to about 8 wt% of the second phosphate-containing flame retardant.
7. The composition of claim 4 or claim 5, comprising about 1 to about 11 wt% of the homopolymer and about 2 to about 8 wt% of the second phosphate-containing flame retardant.
8. The composition of claim 4 or claim 5, comprising about 3 to about 11 wt% of the homopolymer and about 3 to about 6 wt% of the second phosphate-containing flame retardant.
9. The composition of any one of claims 1-8 having a notched Izod impact (NII) value measured at 23 °C of at least 84 J/m when measured by ASTM D256.
10. The composition of any one of claims 1-9 having a notched Izod impact (NII) value measured at 0 °C of at least 57 J/m when measured by ASTM D256.
11. The composition of any one of claims 4-10, wherein the second phosphate-containing flame retardant comprises resorcinol diphosphate.
12. The composition of any one of claims 1-11, wherein the filler comprises glass.

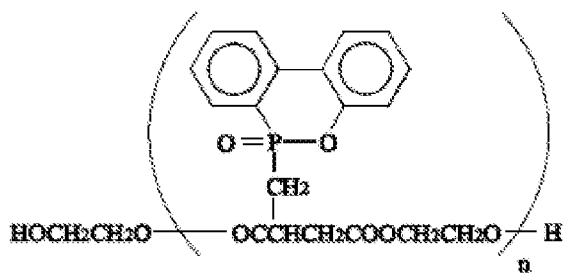
13. A method of forming a composition comprising blending 55-90 wt% polycarbonate; 0-30 wt% filler; and 5-15 wt% flame retardant comprising a homopolymer of the compound of the formula



to produce a composition having a heat deflection temperature (HDT) value of at least 100 °C at a load of 1.82 mPa on a 3.2 mm sample as measured according to ASTM D648; wherein all weight percent values are based on the total weight of the composition and the combined weight percent value of all components does not exceed 100 wt%.

14. The method of claim 13, wherein the flame retardant comprises

(i) the homopolymer having the formula



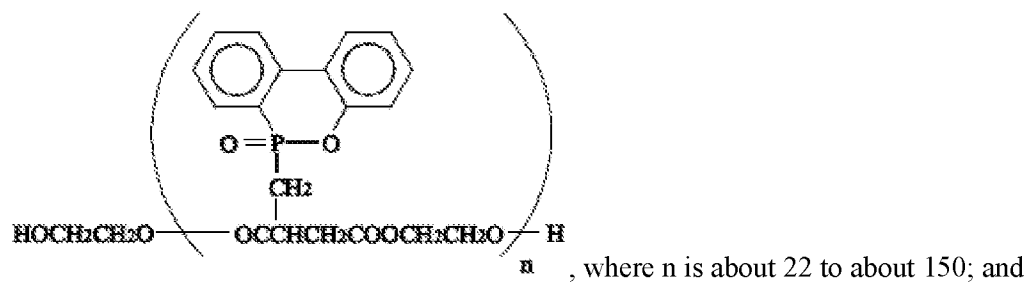
, where n is about 22 to about 150; and

(ii) a second phosphate-containing flame retardant.

15. The method of claim 14, comprising about 1 to about 11 wt% of the homopolymer and about 2 to about 8 wt% of the second phosphate-containing flame retardant.

16. The method of claim 14 or claim 15, wherein the second phosphate-containing flame retardant comprises resorcinol diphosphate.

17. The method of any one of claims 13-16, comprising about 70 wt% to about 85 wt% of polycarbonate.
18. An article comprising the composition of any one of claims 1-12.
19. The article of claim 18, wherein the article is a component in business equipment, a monitor, a portable electronic device, or an automobile.
20. The articles of claim 18 or claim 19, wherein the flame retardant comprises
- (i) the homopolymer having the formula



- (ii) a second phosphate-containing flame retardant.

INTERNATIONAL SEARCH REPORT

International application No
PCT/IB2016/052208

A. CLASSIFICATION OF SUBJECT MATTER

INV. C08L69/00 C08L67/02
ADD. C08G63/692

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)

C08G C08L

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

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

EPO-Internal, WPI Data, CHEM ABS Data

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X A	JP 2009 144042 A (KANEKA CORP) 2 July 2009 (2009-07-02) paragraph [0002] - paragraph [0101]; claims 1, 2, 4; examples Manufacture example 1, A2, Comparative example 1; tables 1, 2	1,9,10, 13,18,19 2-8,11, 12, 14-17,20
A	----- EP 2 770 027 A1 (TOYO BOSEKI [JP]) 27 August 2014 (2014-08-27) paragraph [0016] - paragraph [0039]; examples 1, 2; table 1	1-20
A	----- JP 2005 194499 A (TOYO BOSEKI; NAGASE CHEMTEX CORP) 21 July 2005 (2005-07-21) paragraph [0014] - paragraph [0123]; examples Compound (x), reference example 1, 1; table 1	1-20
	----- -/-	



Further documents are listed in the continuation of Box C.



See patent family annex.

* Special categories of cited documents :

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

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

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

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

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

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

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

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

"&" document member of the same patent family

Date of the actual completion of the international search

8 June 2016

Date of mailing of the international search report

17/06/2016

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

Enescu, Cristina

INTERNATIONAL SEARCH REPORT

International application No

PCT/IB2016/052208

C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT		
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
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Information on patent family members

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