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(54) Title: CROSSLINKABLE POLYMER COMPOSITION COMPRISING ORGANIC PEROXIDE

(57) Abstract: The present invention relates to a crosslinkable polymer composition for use as a sheathing for electrical and/or optical cable or lines, to a cable and/or line with the claimed polymer composition, and to a method for preparing a sheathing for cable and/or line. The crosslinkable polymer composition according to the present invention comprises an elastomeric polymer, a mixture of dialkyl peroxide type peroxide and peroxyketal type peroxide, and antioxidant.



CROSSLINKABLE POLYMER COMPOSITION COMPRISING ORGANIC PEROXIDE**Field of the invention**

The present invention relates to a crosslinkable polymer composition comprising a mixture of organic peroxides, more particularly for the production of sheathing for cable or line.

In addition, present invention relates to a method for preparing sheathing for cable or line with the polymer composition of the present invention, which following production via mixing of the components is crosslinked, and more particularly compressed and crosslinked.

Furthermore, the present invention relates to a cable or line having sheathing that is produced with the polymer composition of the present invention.

Background of the invention

Chemical and physical crosslinking reaction provides thermosetting properties for rubber. Such crosslinking reaction typically enables rubber to have increased mechanical properties, thermal- or chemical-resistance. The rubber crosslinking reaction can be divided depending on the crosslinking method, into sulfur crosslinking, organic peroxide crosslinking, radiation crosslinking, silane crosslinking and the like.

Among them, the organic peroxide crosslinking method is widely used for crosslinking rubber in cable industry, since its mechanism is relatively simple and the polymer produced by the method has an enhanced thermal-resistance. Organic peroxide is degraded via light and heat to form free-radicals, and the activity can be determined depending on its decomposition temperature. As the decomposition temperature is lower, the crosslinking reaction occurs faster. Crosslinking speed is an important factor in preparing cables and lines. When the crosslinking speed is increased, the extrusion line speed becomes faster, and thus the productivity is increased and the expenses can be reduced.

However, as crosslinking speed is increased, the oxidation reaction occurs faster. In addition, if decomposition temperature is low, undesired scorch may occur during the procedure of mixing and extruding, which may decrease the mechanical property and workability of rubber.

One of methods commonly used for raising crosslinking speed, is adding a co-crosslinking agent. Co-crosslinking agent activates organic peroxide crosslinking reaction so as to raise crosslinking speed or crosslinking density. However, if co-crosslinking agent is added to the polymer excessively, it may decrease scorch stability and other mechanical properties or decrease workability due to the increased adhesion with metal interface.

Therefore, there still remains in the related art demands for crosslinkable polymer composition having rapid crosslinking speed, while maintaining good scorch stability.

Prior Art

[Patent Publication No.1] US 4180531 (December 25, 1975)

Brief description of the invention

It is an object of the present invention to provide a crosslinkable polymer composition, more particularly for use in sheathing for electrical and/or optical cable or line, having good crosslinking speed and scorch stability.

It is another object of the present invention to provide a cable or line with a sheathing made of said crosslinkable polymer composition.

More particularly, the present invention provides a cable or line with a crosslinked sheathing obtained from said crosslinkable polymer composition.

It is another object of the present invention to provide a method for producing sheathing for cable or line, from said crosslinkable polymer composition.

In order to achieve the object, the present invention provides a crosslinkable polymer composition comprising an elastomeric polymer; an organic peroxide comprising a mixture of dialkyl peroxide type peroxide and peroxyketal type peroxide; and antioxidant. The crosslinkable polymer composition of the present invention can be used for preparing sheathing for electric and/or optical cables or lines.

In one embodiment, the elastomeric polymer according to the present invention comprises chlorinated polyethylene (CPE) and ethylene vinyl acetate copolymer (EVA).

In another embodiment, the antioxidant of the present invention comprises phenol based antioxidant or UV stabilizer, preferably, both of them.

In one preferable embodiment, the polymer composition according to the present invention comprises the organic peroxide in an amount of 0.5 to 10 parts by weight, with respect to 100 parts by weight of elastomeric polymer.

In another preferable embodiment, the composition of the invention comprises dialkyl peroxide type peroxide in amount of 1 to 5 parts by weight with respect to 100 parts by weight of elastomeric polymer, and peroxyketal type peroxide in an amount of 2 to parts by weight, preferably 5 to 10 parts by weight with respect to 100 parts by weight of elastomeric polymer.

In another preferable embodiment, the polymer composition according to present invention comprises antioxidant in an amount of 0.1 to 5 parts by weight with respect to 100 parts by weight of elastomeric polymer.

In addition, in order to achieve the object, the present invention provides a cable or line with a sheathing that is made of a crosslinkable polymer composition comprising elastomeric polymer, an organic peroxide comprising a mixture of dialkyl peroxide type peroxide and peroxyketal type peroxide, and antioxidant.

More particularly, the present invention provides a cable or line with a crosslinked sheathing that is obtained from a

crosslinkable polymer composition comprising elastomeric polymer, an organic peroxide comprising a mixture of dialkyl peroxide type peroxide and peroxyketal type peroxide, and antioxidant.

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Furthermore, the present invention provides a method for produce a sheathing for electrical and/or optical cable or line, comprising:

- i) mixing the cross linkable polymer composition, and
- 10 ii) crosslinking the mixture obtained from step i), and more preferably compressing the mixture obtained from step i) so as to crosslink the mixture.

In the present invention, the term "sheathing" means more particularly the outermost layer of a cable or line.

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Advantageous effects

The crosslinkable polymer composition according to the present invention can guarantee a good crosslinking speed, which can increase extrusion line speed when producing a wire, and ultimately, can raise productivity.

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In addition, the polymer composition according to the present invention, can show an optimized scorch stability while guaranteeing a good crosslinking speed and good mechanical properties, so that the composition can be used for preparing sheathing for electrical and/or optical cables or lines.

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Detailed description of the invention

Hereinafter, the invention will be described in more detail.

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The crosslinkable polymer composition according to the present invention comprises elastomeric polymer, organic peroxide and antioxidant.

Elastomeric polymer

The polymer composition according to the present invention comprises an elastomeric polymer, preferably, chlorinated polyethylene (CPE) and ethylene vinyl acetate copolymer (EVA).

The chlorinated polyethylene (CPE) is a polymer having polyethylene main chain with chlorine atoms randomly replacing the hydrogen atoms along the chain. By adding chlorine atoms, the polymer has an enhanced fire retardant and advantageous chemical resistance and thermo-resistance.

The ethylene vinyl acetate copolymer (EVA) is a copolymer that is produced via polymerization of ethylene monomer and vinyl acetate monomer. The physical properties of EVA are determined depending on the degree of polymerization and content of vinyl acetate, and typically EVA presents good chemical-resistance, weather resistance, and ozone-resistance.

In a preferred embodiment, EVA has vinyl acetate content from 20 to 70%, and more preferably from 25 to 40%.

Organic peroxide

The polymer composition according to the present invention comprises organic peroxide as a crosslinking agent. Organic peroxide may generally be considered as derivatives of hydrogen peroxide (H_2O_2) in which one or both of hydrogen atoms is/are replaced by organic group(s). Said organic peroxide produces free radicals by light or heat, and the radicals form c-c linkage with adjacent polymer chains, so that the rubber that presents enhanced mechanical strength, thermo-resistance, chemical resistance and electrical properties can be produced.

The organic peroxide according to the present invention comprises a mixture of dialkyl peroxide type peroxide and peroxyketal type peroxide. Said mixture provides a rapid crosslinking speed and proper scorch stability to the polymer composition.

In one preferable embodiment, the dialkyl peroxide type peroxide is selected from the group consisting of Dicumyl peroxide, 2,5-dimethyl-2,5-Di-(t-butylperoxy)hexane and Bis(t-

butylperoxy isopropyl)benzene, and preferably is Bis(t-butylperoxy isopropyl)benzene.

In another preferable embodiment, peroxyketal type peroxide is 1,4-bis-(t-butylperoxy)-3,3,5-trimethylcyclohexane or n-butyl 4,4-di(t-butylperoxy)valerate, and preferably n-butyl 4,4-di(t-butylperoxy)valerate.

In another preferable embodiment, the content of organic peroxide in the composition according to the present invention is 0.5 to 10 parts by weight, with respect to 100 parts by weight of elastomeric polymer. In another preferable embodiment, the composition comprises:

- dialkyl peroxide type peroxide in an amount of 0.5 to 5 parts by weight with respect to 100 parts by weight of elastomeric polymer, and

- peroxyketal type peroxide in an amount of 2 to 10 parts by weight, preferably 5 to 10 parts by weight, with respect to 100 parts by weight of elastomeric polymer.

Antioxidant

The polymer composition according to the present invention comprises antioxidant. The antioxidant can prevent polymer composition from oxidation, and enhance weather-resistant/thermos-resistant properties, and scorch stability.

In one embodiment, the antioxidant according to the present invention is selected from the group consisting of:

- phenol based antioxidant (i.e., 2,2'thiodiethylene bis [3-(3,5-di-t-butyl-4-hydroxyphenyl)propionate ; 1,3,5-trimethyl-2,4,6-tris(3,5-di-t-butyl-4-hydroxybenzyl)benzene}; 2,2'-methylenebis(6-t-butyl-4-methylphenol); Pentaerythritol tetrakis(3,5-di-tert-butyl-4-hydroxyhydrocinnamate)),
- UV stabilizer (i.e., benzotriazole based UV stabilizer such as 2,4-di-tert-butyl-6-(5-chlorobenzotriazol-2-yl)phenol; 2-(2H-benzotriazole-2-yl)-p-cresol; 2-(2H-benzotriazole-2-yl)-4,6-bis(1-methyl-1-phenylethyl); 2-(2H-benzotriazole-2-yl)-4,6-ditertpentylphenol),

- Amine based antioxidant (i.e., octylated diphenylamine, 2,2,4-trimethyl-1,2-dihydroquinoline (TMQ)),
- Cyclic acetal based antioxidant (i.e., 3,9-di-3-cyclohexen-1-yl-2,4,6,10-tetraoxaspiro[5.5]undecane), and
- 5 - a combination thereof.

In one preferable embodiment, the antioxidant according to the present invention includes phenol based antioxidant or UV stabilizer, and preferably, includes both of them. Particularly, the phenol based antioxidant can prevent scorch stability of the composition from decreasing while the crosslinking speed is increasing. Also, the phenol based antioxidant and UV stabilizer can enhance the mechanical properties of the polymer composition.

In one preferable embodiment, the content of antioxidant in polymer composition is 0.1 to 5 parts by weight with respect to 100 parts by weight of elastomeric polymer. In another preferable embodiment, the polymer composition according to the present invention may comprise phenol based antioxidant and UV stabilizer in an amount of 0.1 to 2.5 parts by weight with respect to 100 parts by weight of elastomeric polymer, separately.

Other additives

The polymer composition according to the present invention may optionally further comprises organic additives and/or inorganic additives. Preferably, the organic additives may include compatibilizer, plasticizer, pigments, coupling agents, or crosslinking aid agents. In preferable embodiment, the inorganic additives may include metal-based inorganic additives and ceramic-based inorganic additives, such as carbon black, calcium carbonate (CaCO_3), talc, china clay, graphite, silica, mica, antimony trioxide, lead oxide, aluminum hydroxide, magnesium hydroxide, magnesium oxide, zinc oxide

In one non-limited embodiment, the content of organic additives is 100 parts by weight or less, preferably 50 parts by weight or less, with respect to 100 parts by weight of elastomeric polymer, and the content of inorganic additives is 300 parts by weight or less, preferably 150 parts by weight with

respect to 100 parts by weight of elastomeric polymer.

Cable or line

5 Another object of the invention is to provide an electrical and/or optical cable or line with a sheathing that is made of polymer composition according to the present invention. In one embodiment, the cable according to the present invention is a cable having sheathing as defined according to standard HD 22.1
10 EM 7. Preferably, said cable can be used as a wind turbine power cable. In another embodiment, the sheathing that is made of polymer composition according to the present invention can be used as inner sheath and/or outer sheath of the cable. Preferably, the inner sheath can be positioned between insulation and braid,
15 and the outer sheath can be positioned at outermost part of the cable. The cable comprising said layer(s) has outstanding flexibility and features such as weather resistance, oil resistance and cold resistance so as to protect cable from surrounding environment.

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Preparation method for sheathing of cable or line

Another object of the present invention is to provide a method for preparing a sheathing for electrical and/or optical cables or lines, comprising:

- 25 i) mixing the cross linkable polymer composition according to the present invention, and
ii) crosslinking the mixture obtained from step i), and more preferably compressing the mixture obtained from step i) so as to crosslink the mixture.

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The sheathing for cable and line according to the present invention can be produced by mixing the crosslinkable polymer composition comprising aforementioned components, using for example two-roll mill. The organic and/or inorganic additives
35 disclosed before may be further added to said mixture depending on the particular properties industrially required to the

crosslinkable polymer composition for sheathing.

In addition, said mixture of crosslinkable polymer composition may be compressed in mold under the high temperature so as to be crosslinked to form a crosslinked product. Preferably, the crosslinking procedure is performed under the temperature of, for example, 150°C or more, particularly 150°C to 220°C, with the pressure of, preferably 12 to 22 MPa, more preferably 16 to 20MPa, by heating, for example using water or inorganic salt as a thermal fluid.

The method according to the present invention has high crosslinking speed, which can increase productivity.

Hereinafter, the present invention is described in further detail in the following Examples which are not in any way intended to limit the scope of the invention as claimed. In addition, it will appear to a person skilled in the art that various modifications may be made to the disclosed embodiments, and that such modifications are intended to be within the scope of the present invention.

Example

Preparation of crosslinkable polymer composition and its crosslinked product (representative example of preparation)

As a representative example of the method for preparing a crosslinkable polymer composition and its crosslinked product according to the invention, the crosslinkable composition is prepared using the components and the mixing ratio as shown Table 1.

[Table 1]

Components	ratio (phr)
CPE(CM 3610)	79
EVA(Elvax 265)	21

antioxidant (Tinuvin 327)	0.5
antioxidant (Anox-70)	0.5
Dialkyl peroxide (PBP-98)	1.0
peroxyketal (Luperox 230XL-40SP)	6

In Table 1, phr refers to parts per 100 parts of elastomeric polymer, the elastomeric polymer in Table 1 being a mixture of CPE and EVA.

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Firstly, we mixed:

- CPE with a chlorine content of 36% commercialized by HANGZHOU KELICHEM under the reference CM 3610;
 - EVA with a vinyl acetate content of 28% commercialized by Dupont with the reference Elvax 265;
 - UV stabilizing antioxidant as 2,4 ditertbutyl-6-(5-chlorobenzotriazol-2-yl) phenol commercialized by Ciba with the reference Tinuvin 327;
 - phenol based antioxidant as 2,2'-thiodiethylene bis[3-(3,5-di-t-butyl-4-hydroxyphenyl)propionate], commercialized by Addivant with the reference Anox-70;
 - A dialkylperoxide as Bis (t-butylperoxy isopropyl)benzene commercialized by NOF with the reference PBP-98;
 - A peroxyketal peroxides as n-butyl 4,4-di(t-butylperoxy)valerate commercialized by Arkema with the reference Luperox 230XL-40SP,
- , with the ratio in Table 1, using a two-roll mill at 80°C for 15 minutes.

The mixture thus obtained was compressed under 170°C/15MPa and crosslinked for a period of time of t_{c90} defined below. And subsequently the pressure was released and the obtained composite was cooled in the air. As a result, the crosslinked product of crosslinkable polymer composition of the present invention was made.

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Evaluation method for crosslinking speed of polymer composition

In order to measure the crosslinking speed of polymer composition, the ODR (Oscillation disk rheometer) test under the condition as defined in ASTM D2084 standard was carried out and determine the time t_{c90} taken to reach to 90% of the maximum ODR torque value, which is increased as the crosslinking reaction is occurred. It is considered that the lower t_{c90} represents the faster crosslinking speed.

Evaluation method for scorch stability of polymer composition

In order to measure scorch stability of polymer composition, Money viscometer test defined in standard ASTM D1646 was carried out, and determined the scorch time (t5) which is a time required for the increase of 5 points from minimum viscosity value of mixture in molten status. It is considered that the higher scorch time (t5) represents the higher scorch stability.

Evaluation method for mechanical properties of crosslinked product of polymer composition

The mechanical properties such as tensile strength (TS) and Elongation at break (EB) of crosslinked product was determined were determined by preparing dumbbell-shaped species as defined in the standard DIN 53504.S2, and then using a universal tensile strength tester under the condition as defined in standard IEC 60811-1-1. When both values of the tensile strength and the elongation at break are higher, the mechanical properties are considered to better on the whole.

Properties determined using different polymer compositions or their crosslinked products 1

In order to identify the effects of peroxides on crosslinking speed, polymer composition was prepared using the components and mixing ratio in Table 2 according to the method that is described in the representative preparation example.

The polymer compositions thus obtained were determined on the crosslinking speed and scorch stability according to the measurement method described before. And the results are presented in the following Table 2.

[Table 2]

	Comparative example 1	Comparative example 2	Example 1	Example 2
CM 3610 (phr)	79	79	79	79
Elvax 265 (phr)	21	21	21	21
Vulkanox HS/LG (phr)	0.15	0.15	0.15	0.15
Vulkazon AFS/LG (phr)	0.35	0.35	0.35	0.35
PBP-98 (phr)	2.6		1.3	1.3
Luperox 230XL-40SP (phr)	–	6.0	3.0	6.0
t_{c90} (min, 180°C)	7.3	3.5	4.0	3.3
Mooney scorch t5(min, 125°C)	>60	25	32	29

In Table 2,

– phr refers to parts per 100 parts of elastomeric polymer, the elastomeric polymer in Table 2 being a mixture of CPE and EVA,

– Vulkanox HS/LG is a trade name of amine based antioxidant as 2,2,4-trimethyl-1,2-dihydroquinoline (TMQ) commercialized by Lanxess,

– Vulkazon AFS/LG is a trade name of a cyclic acetal based antioxidant as 3,9-di-3-cyclohexen-1-yl-2,4,6,10-tetraoxaspiro

[5.5]undecane commercialized by Lanxess, and

– the meaning of other abbreviations described in Table 2 is as defined above in the present specification.

As shown in Table 2, the polymer composition according to example 1 and 2 were shown to have crosslinking speed relatively

faster than that comparative example 1 which does not comprise peroxyketal type peroxide. In addition, in spite of fast crosslinking speed, the composition according to examples 1 and 2 were shown to have enhanced scorch stability compared to that of comparative example 2 which does not comprise dialkyl peroxide type peroxide.

Properties determined using different polymer compositions or their crosslinked products 2

In order to identify the effect of antioxidant on scorch stability, polymer composition was prepared using the components and mixing ration in Table 3 according to the method that is described in the representative preparation example.

The polymer compositions thus obtained were determined on the crosslinking speed, scorch stability and mechanical properties according to the measurement method described before. And the results are presented in the following Table 3.

[Table 3]

	Comparative example					example		
	3	4	5	6	7	3	4	5
CM 3610 (phr)	79	79	79	79	79	79	79	79
Elvax 265 (phr)	21	21	21	21	21	21	21	21
Vulkanox HS/LG (phr)	2	1	1					
Octamine(phr)		1		1				
Tinuvin 327 (phr)			1	1			0.5	0.5
Anox-70 (phr)					2	1	0.5	0.5
PBP-98 (phr)						1.3	1.3	1.0
Luperox 230XL-40SP (phr)	8	8	8	8	8	4	4	6
t_{c90} (min, 180°C)	3.8	3.7	3.8	4.5	5.4	5.7	6.6	5.8
Mooney scorch t_5 (min, 125°C)	39	36	47	44	84	49	50	48
Tensile	9.3	—	9.9	—	8.9	10.5	12.2	11.2

strength(MPa)								
Elongation at break (%)	533	-	534	-	550	312	367	351

In Table 3,

- phr refers to parts per 100 parts of elastomeric polymer, the elastomeric polymer in Table 3 being a mixture of CPE and EVA,

- Octamine is a trade name of amine based antioxidant as octylated diphenylamine, commercialized by CROMPTON Corporation

- the meaning of other abbreviations described in Table 2 is as defined above in the present specification.

As shown in Table 3, among the compositions according to the comparative examples 3 to 7 which comprise only peroxyketal type peroxide as crosslinking agent, compositions having phenol based antioxidant are shown to have the highest scorch stability. In addition, the composition according to example 4 which comprises both phenol based antioxidant and uv stabilizing antioxidant shows enhanced mechanical properties compared with that of composition according to example 3 which comprises only phenol based antioxidant.

Furthermore, the composition comprising 1 phr of dialkylperoxide and 6.0 phr of peroxyketal according to example 5 is shown to have higher crosslinking speed compared to the composition comprising 1.3 phr of dialkylperoxide and 4.0 phr of peroxyketal according to example 4.

Comparison of mechanical properties between the compositions according to comparative example 1 and example 12 of the present invention

The mechanical properties of the compositions according to comparative example 1 and the example 12 are disclosed in the following Table 4.

[Table 4]

component/test	Comparative example 1	Example 12
Peroxide	Dialkylperoxide	dialkylperoxide + peroxyketal
Antioxidant	TMQ + cyclic acetal based antioxidant	Phenol based antioxidant + benzotriazole based UV stabilizer
TC 90(min)	7.3	5.8
Mooney scorch t5 (125°C)	>60	48
Tensile strength (half curing) ¹⁾	10.0	11.3
Tensile strength (optimum curing) ²⁾	11.7	11.2

¹⁾is performed according to compression moulding at 170°C/15MPa for 5 minutes

²⁾ is performed according to compression moulding at 170°C/15MPa for 13 minutes

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As shown Table 4, the composition according to example 12 of the present invention is shown to have about 20% increased crosslinking speed with good scorch stability and mechanical properties (tensile strength), compared to the composition according to the comparative example 1.

Accordingly, the results show that according to present invention, the composition having fast crosslinking speed while guaranteeing optimized scorch stability and physical property can be produced.

Claims

1. A crosslinkable polymer composition, comprising:

- an elastomeric polymer;

- an organic peroxide comprising a mixture of dialkyl peroxide type peroxide and peroxiketal type peroxide; and

- an antioxidant.

2. The crosslinkable polymer composition according to claim 1, wherein the elastomeric polymer comprises chlorinated polyethylene (CPE) and ethylene vinyl acetate copolymer (EVA).

3. The crosslinkable polymer composition according to claim 1 or 2, wherein the antioxidant comprises phenol based antioxidant.

4. The crosslinkable polymer composition according to any one of the preceding claims, wherein the antioxidant comprises phenol based antioxidant and UV stabilizer.

5. The crosslinkable polymer composition according to any one of the preceding claims, wherein the composition comprises the organic peroxide in an amount of 0.5 to 10 parts by weight with respect to 100 parts by weight of elastomeric polymer, and the antioxidant in an amount of 0.1 to 5 parts by weight with respect to 100 parts by weight of elastomeric polymer.

6. The crosslinkable polymer composition according to any one of the preceding claims, wherein the composition comprises 1 to 5 parts by weight of dialkyl type peroxide with respect to 100 parts by weight of elastomeric polymer, and 5 to 10 parts by weight of peroxyketal type peroxide with respect to 100 parts by weight of elastomeric polymer.

7. Electrical and/or optical cable or line with a sheathing made of a polymer composition according to any one of the

preceding claims.

8. Electrical and/or optical cable or line with a
crosslinked sheathing obtained from a polymer composition
5 according to any one of the preceding claims.

9. Method for preparing a sheathing for electrical and/or
optical cable or line, comprising:

- 10 i) mixing the crosslinkable polymer composition according
to any one of the preceding claims; and
ii) crosslinking the mixture obtained from step i), and
more preferably compressing the mixture obtained from step i) so
as to crosslink the mixture.

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INTERNATIONAL SEARCH REPORT

International application No
PCT/IB2017/000458

A. CLASSIFICATION OF SUBJECT MATTER
INV. C08L23/28 H01B3/44
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 H01B 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, CHEM ABS Data, 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	US 2013/062096 A1 (HAN SUH JOON [US] ET AL) 14 March 2013 (2013-03-14) paragraphs [0057] - [0059]; claims 1,5 -----	1-9
X	JP H11 26791 A (MITSUI DU PONT POLYCHEMICAL) 29 January 1999 (1999-01-29) paragraphs [0007], [0013]; claim 1; examples 1,3,4 -----	1-6
X	KR 2012 0040359 A (SAMSUNG TOTAL PETROCHEMICALS [KR]) 27 April 2012 (2012-04-27) claims 1-5,7-9 -----	1-6
X	US 2009/159129 A1 (KATAOKA HISATAKA [JP] ET AL) 25 June 2009 (2009-06-25) paragraph [0091]; claims 1-4, 6, 7, 10 -----	1-6

☐

Further documents are listed in the continuation of Box C.

☒

See patent family annex.

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Date of the actual completion of the international search

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INTERNATIONAL SEARCH REPORT

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