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(71) Applicant: **BOREALIS AG** [AT/AT]; Trabrennstrasse 6-8, 1020 Vienna (AT).

(72) Inventors: **KLIMKE, Katja**; Borealis Polyolefine GmbH, St.-Peter-Straße 25, 4021 Linz (AT). **HAGSTRAND, Per-Ola**; Borealis AB, 444 86 Stenungsund (SE). **NILSSON, Ulf**; Borealis AB, 444 86 Stenungsund (SE). **GKOURMPIS, Thomas**; Borealis AB, 444 86 Stenungsund (SE). **EFRAIMSSON, Lars**; Borealis AB, 444 86 Stenungsund (SE). **JOHANSSON, Anette**; Borealis AB, 444 86 Stenungsund (SE).

(74) Agent: **MAIWALD GMBH**; Elisenhof, Elisenstraße 3, 80335 München (DE).

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(54) Title: CABLE COMPRISING POLYPROPYLENE COMPOSITION

(57) Abstract: The present invention relates to a cable comprising at least one layer comprising a polypropylene composition, which comprises a copolymer of propylene and comonomer units selected from ethylene and alpha-olefins having from 4 to 12 carbon atoms, wherein the polypropylene composition has a total comonomer content, preferably total ethylene content, of from 4.0 to 11.0 wt.-%, based on the total weight of the polypropylene composition and determined by FT-IR spectroscopy calibrated by quantitative ^{13}C-NMR spectroscopy from crystallization extraction (CRYSTEX); a melt flow rate MFR_2 of from more than 2.5 to 6.0 g/10 min, determined according to ISO 1133 at 230°C and 2.16 kg; a soluble fraction (SF) content, determined by crystallization extraction (CRYSTEX), in the range from 10.0 to 25.0 wt.-%, based on the total weight amount of the polypropylene composition; and a flexural modulus of from more than 470 MPa to 750 MPa, determined according to ISO 178 method A.

Cable comprising polypropylene composition

The present invention relates to a cable comprising at least one layer comprising a polypropylene composition having a soluble fraction (SF) content, determined according to CRYSTEX analysis, in the range from 10.0 to 25.0 wt.-%, which is suitable for high voltage AC cables.

Technical background

Nowadays, ethylene polymer products are used as insulation and semiconducting shields for medium and high voltage cables, due to easy processability and their beneficial electrical properties. In addition, in low voltage applications polyvinyl chloride (PVC) is also commonly used as insulation material, usually in combination with softeners to reach desirable softness of cables. PVC is a thermoplastic which by incorporation of various plasticizers can be used in a wide temperature range. For standard PVC a continuous conductor temperature of max. 70°C is normal. At low temperatures PVC becomes rigid and usage temperatures below -10°C should be avoided. At conductor temperatures over 100°C the plasticizers migrate out and the materials lose their flexibility. However, with the addition of special plasticizers and stabilizers, PVC materials can be produced for conductor temperatures of 90-105°C. But in essence, PVC is mainly used for the 1 kV area, as the higher permittivity and dissipation factor of the material means that the losses increase too much at higher voltages and therefore PVC cables are not normally not used over 1 kV. In addition, softeners have to be added to PVC in order to maintain a high level of flexibility. Insufficient amounts of softeners reduce low temperature properties of PVC significantly. From an environmental point of view, these softeners are not always regarded as problem-free, making them desirable to eliminate.

Especially for medium, high and extra high voltage (MV, HV and EHV) cables insulation material presently is dominated by crosslinked ethylene polymer (XLPE) products. These products have a high operation temperature, a high electric breakdown strength and good mechanical properties. Due to crosslinking XLPE has thermosetting properties.

Lately, attempts were made using thermoplastic material and especially thermoplastic propylene polymers as insulation material for medium, high and extra high voltage (MV, HV and EHV) cables. Further, transmission system operators are expressing an increasing interest for recycling cable components at end-of-life.

WO 2022/200395 A1 and WO 2022/200396 A1 disclose flexible polypropylene compositions suitable for cable insulation, which both comprise flexible heterophasic copolymers of propylene and ethylene. Said heterophasic copolymers of propylene and

ethylene have a rather high amount of amorphous phase, which renders the composition rather sticky during cable extrusion. Further, either the melt flow rate of said heterophasic copolymers of propylene and ethylene is rather low or increased by visbreaking, which to some extent impairs the AC breakdown strength, so that Weibull alpha values of not more than 50 kV/mm are obtained, which does not allow to use these polypropylene compositions for high voltage AC cables.

US 2012/0220727 A1 discloses a heterophasic polypropylene resin with a MFR_2 of at least 1.0 g/10 min comprising a propylene random copolymer matrix phase and an ethylene-propylene copolymer rubber phase dispersed within the matrix phase, wherein the heterophasic polypropylene resin has an amount of XCS phase of 15 to 45 wt.-%, the XCS phase has an ethylene content of 25 wt.-% or lower, and the heterophasic polypropylene resin is characterized by a strain hardening factor of 1.7 to 4.0.

US 4,259,410 discloses an insulation for electric cables, which comprises partially crystalline, randomly copolymer polypropylene having a proportion of ethylene, 1-butene and/or 1-hexene of 1-6 wt.-%, a viscosity value I of 200-400 cc./g, a melt flow index $MFI_{190/5}$ of 1-20 g/10 min, and an elongation value above 700%.

EP 2 602 287 A1 discloses a cable having at least one insulation layer comprising a polymer composition consisting of (a) at least 94 wt.-% of a crystalline polypropylene homo- or copolymer with a MFR_2 of 1 to 10 g/10 min and a comonomer content of below 5 wt.-%, the comonomers are ethylene and/or a C_4 to C_{10} alpha-olefin, (b) 0.5 to 5 wt.-% of an adhesion promotor being a polar modified polypropylene homo- or copolymer, and (c) 0.02 to 1 wt.-% of a soluble alpha-nucleating agent.

In the present invention a polypropylene composition with a rather low amount of soluble fraction determined by crystallization extraction (CRYSTEX), which is a measure for the amorphous phase of the polyolefin composition, and high reactor-made melt flow rate is proposed, which shows a good balance of properties in regard of flexibility, mechanical properties, impact properties, thermal conductivity and electrical breakdown strength and additionally shows a good performance during cable extrusion. Cables comprising said polypropylene composition as insulation layers show superior electrical breakdown strength and are therefore especially suitable for HVAC cable systems.

Summary of the invention

The present invention relates to a cable comprising at least one layer comprising a polypropylene composition, which comprises a copolymer of propylene and comonomer units selected from ethylene and alpha-olefins having from 4 to 12 carbon atoms, wherein the polypropylene composition has

a total comonomer content, preferably total ethylene content, of from 4.0 to 11.0 wt.-%, preferably from 5.0 to 10.0 wt.-%, more preferably from 5.5 to 9.5 wt.-%, still more preferably from 6.0 to 9.0 wt.-%, based on the total weight of the polypropylene composition and determined by FT-IR spectroscopy calibrated by quantitative ^{13}C-NMR spectroscopy from crystallization extraction (CRYSTEX);

a melt flow rate MFR_2 of from more than 2.5 to 6.0 g/10 min, preferably from 2.8 to 5.5 g/10 min, still more preferably from 3.0 to 5.0 g/10 min and most preferably from 3.3 to 4.5 g/10 min, determined according to ISO 1133 at 230°C and 2.16 kg;

a soluble fraction (SF) content, determined by crystallization extraction (CRYSTEX), in the range from 10.0 to 25.0 wt.-%, preferably from 12.0 to 23.0 wt.-%, more preferably from 13.5 to 21.5 wt.-% and most preferably from 15.0 to 20.0 wt.-%, based on the total weight amount of the polypropylene composition; and

a flexural modulus of from more than 470 MPa to 750 MPa, more preferably of from 475 MPa to 700 MPa and most preferably of from 500 MPa to 650 MPa, determined according to ISO 178 method A.

Said at least one layer is preferably an insulation layer.

Said cable is preferably a high voltage AC cable.

Definitions

A heterophasic polypropylene is a propylene-based copolymer with a semi-crystalline matrix phase, which can be a propylene homopolymer or a random copolymer of propylene and at least one alpha-olefin comonomer, and an elastomeric phase dispersed therein. The elastomeric phase can be a propylene copolymer with a high amount of comonomer, which is not randomly distributed in the polymer chain but are distributed in a comonomer-rich block structure and a propylene-rich block structure.

A heterophasic polypropylene usually differentiates from a one-phasic propylene copolymer in that it shows two distinct glass transition temperatures Tg which are attributed to the matrix phase and the elastomeric phase.

A propylene homopolymer is a polymer, which essentially consists of propylene monomer units. Due to impurities especially during commercial polymerization processes a propylene homopolymer can comprise up to 0.1 mol% comonomer units, preferably up to 0.05 mol% comonomer units and most preferably up to 0.01 mol% comonomer units.

A propylene random copolymer is a copolymer of propylene monomer units and comonomer units in which the comonomer units are distributed randomly over the polypropylene chain. Thereby, a propylene random copolymer includes a fraction, which is insoluble in xylene – xylene cold insoluble (XCI) fraction – in an amount of at least 85 wt%, most preferably of at least 88 wt%, based on the total amount of propylene random copolymer. Accordingly, the propylene random copolymer does not contain an elastomeric polymer phase dispersed therein.

Usually, a propylene polymer comprising at least two propylene polymer fractions (components), which have been produced under different polymerization conditions resulting in different (weight average) molecular weights and/or different comonomer contents for the fractions, preferably produced by polymerizing in multiple polymerization stages with different polymerization conditions, is referred to as “multimodal”. The prefix “multi” relates to the number of different polymer fractions the propylene polymer is consisting of. As an example of multimodal propylene polymer, a propylene polymer consisting of two fractions only is called “bimodal”, whereas a propylene polymer consisting of three fractions only is called “trimodal”.
A unimodal propylene polymer only consists of one fraction.
Thereby, the term “different” means that the propylene polymer fractions differ from each other in at least one property, preferably in the weight average molecular weight – which can also be measured in different melt flow rates of the fractions – or comonomer content or both.

An elastomer is a polymer with viscoelasticity and weak intermolecular forces. The term "elastomer" can be used interchangeably with "rubber".
Polyolefin based elastomers, such as polypropylene based elastomers, i.e. an elastomer with a molar majority of olefin monomer units, such as propylene monomer units, are usually thermoplastic elastomers.
Thermoplastic elastomers have both thermoplastic and elastomeric properties.
Polyolefin based elastomers, such as polypropylene based elastomers, usually show a low density and low viscosity. They can be propylene homopolymers or propylene-alpha olefin copolymers, such as propylene-ethylene copolymers. A specific class of polypropylene based elastomers are propylene homopolymers or propylene-alpha olefin copolymers, such as propylene-ethylene copolymers, which have been polymerized in the presence of a single site catalyst, usually in a solution polymerization process.

Vis-breaking is a post reactor chemical process for modifying semi-crystalline polymers such as propylene polymers. During the vis-breaking process, the propylene polymer backbone is degraded, for example by means of peroxides, such as organic peroxides, via beta scission. The degradation is generally used for increasing the melt flow rate and narrowing the molecular weight distribution.

A low density polyethylene is a polymer with predominately ethylene monomers, which is polymerized in a high pressure process by free radical polymerization. The low density polyethylene is characterized by long polymer side chains, which decrease the density of the polymer irrespective of the amount of comonomer.

In the following amounts are given in % by weight (wt%) unless it is stated otherwise.

Detailed description of the invention

Polypropylene composition

The polypropylene composition as described above or below has a total comonomer content, preferably total ethylene content, of from 4.0 to 11.0 wt.-%, preferably from 5.0 to 10.0 wt.-%, more preferably from 5.5 to 9.5 wt.-%, still more preferably from 6.0 to 9.0 wt.-%, based on the total weight of the polypropylene composition and determined by FT-IR spectroscopy calibrated by quantitative ^{13}C-NMR spectroscopy from crystallization extraction (CRYSTEX);

a melt flow rate MFR_2 of from more than 2.5 to 6.0 g/10 min, preferably from 2.8 to 5.5 g/10 min, still more preferably from 3.0 to 5.0 g/10 min and most preferably from 3.3 to 4.5 g/10 min, determined according to ISO 1133 at 230°C and 2.16 kg;
a soluble fraction (SF) content, determined by crystallization extraction (CRYSTEX), in the range from 10.0 to 25.0 wt.-%, preferably from 12.0 to 23.0 wt.-%, more preferably from 13.5 to 21.5 wt.-% and most preferably from 15.0 to 20.0 wt.-%, based on the total weight amount of the polypropylene composition; and
a flexural modulus of from more than 470 MPa to 750 MPa, more preferably of from 475 MPa to 700 MPa and most preferably of from 500 MPa to 650 MPa, determined according to ISO 178 method A.

The polymeric part of the polypropylene composition is characterized by crystallization extraction (CRYSTEX) using trichlorobenzene (TCB) as a solvent. This method is described below in the determination methods section. The crystalline fraction (CF) contains for the most part the matrix phase and only a small part of the elastomeric phase and the soluble fraction (SF) contains for the most part the elastomeric phase and only a small part of the matrix phase. In some cases, this method results in more useful data compared to xylene extraction, since the crystalline fraction (CF) and the soluble fraction (SF) more accurately correspond to the matrix and elastomeric phases, respectively. Due to the differences in the separation methods of xylene extraction and crystallization extraction (CRYSTEX) the properties of XCS/XCI fractions on the one hand and crystalline/soluble (CF/SF) fractions on the other hand are not exactly the same, meaning that the amounts of matrix phase and elastomeric phase can differ as well as the properties.
Generally, the crystalline fraction (CF) content and the soluble (SF) content of a composition only relate to its polymeric components, i.e. without other components, which are insoluble and therefore not part of the dissolution and crystallization cycles as described below in the determination method.
In the present case the polypropylene composition is usually fully soluble in trichlorobenzene (TCB) so that the crystalline fraction (CF) content and the soluble (SF) content relate to the total content of the polypropylene composition. Thus, the crystalline fraction (CF) content and the soluble fraction (SF) content preferably make up 100 wt.-% of the polypropylene composition.

The polypropylene composition has a soluble fraction (SF) content, determined by crystallization extraction (CRYSTEX), in the range from 10.0 to 25.0 wt.-%, more preferably from 12.0 to 23.0 wt.-%, still more preferably from 13.5 to 21.5 wt.-% and most preferably from 15.0 to 20.0 wt.-%, based on the total weight amount of the polypropylene composition.

Said soluble fraction (SF) preferably has a comonomer content, preferably an ethylene content (C2(SF)), determined by FT-IR spectroscopy calibrated by quantitative ^{13}C-NMR spectroscopy, in the range from 20.0 to 35.0 wt%, more preferably from 21.5 to 32.5 wt% and most preferably from 23.0 wt% to 30.0 wt%, based on the total amount of monomer units in the soluble fraction (SF).

Further, said soluble fraction (SF) preferably has an intrinsic viscosity (iV(SF)), determined according to ISO 1628-3, in the range from 90 to 190 cm³/g, more preferably from 100 to 175 cm³/g and most preferably from 115 to 150 cm³/g.

The polypropylene composition preferably has a crystalline fraction (CF) content, determined by crystallization extraction (CRYSTEX), in the range from 75.0 to 90.0 wt.-%, more preferably from 77.0 to 88.0 wt.-%, still more preferably from 78.5 to 86.5 wt.-% and most preferably from 80.0 to 85.0 wt.-%, based on the total weight amount of the polypropylene composition.

Said crystalline fraction (CF) preferably has a comonomer content, preferably an ethylene content (C2(CF)), determined by FT-IR spectroscopy calibrated by quantitative ^{13}C-NMR spectroscopy, in the range from 1.0 to 15.0 wt.-%, more preferably from 2.0 to 10.0 wt.-% and most preferably from 3.0 to 8.5 wt.-%, based on the total amount of monomer units in the crystalline fraction (CF).

Further, said crystalline fraction (CF) preferably has an intrinsic viscosity (iV(CF)), determined according to ISO 1628-3, in the range from 185 to 325 cm³/g, more preferably from 200 to 300 cm³/g and most preferably from 225 to 270 cm³/g.

The polypropylene composition preferably has a ratio of the comonomer content, preferably ethylene content, of the soluble fraction to the comonomer content, preferably

ethylene content, of the crystalline fraction C2(SF)/C2(CF) of from 1.5 : 1.0 to 10.0 : 1.0, more preferably from 2.5 : 1.0 to 7.5 : 1.0, most preferably from 4.0 : 1.0 to 6.0 : 1.0.

Further, the polypropylene composition preferably has a ratio of the intrinsic viscosity of the soluble fraction to the intrinsic viscosity of the crystalline fraction iV(SF)/iV(CF) of from 0.35 : 1.00 to 0.90 : 1.00, preferably from 0.40 : 1.00 to 0.80 : 1.00, more preferably from 0.50 : 1.00 to 0.70 : 1.00.

The polypropylene composition preferably has a total intrinsic viscosity (iV (total)) determined according to ISO 1628-3, in the range from 175 to 300 cm^3/g, more preferably from 200 to 275 cm^3/g and most preferably from 215 to 250 cm^3/g.

The polypropylene composition preferably comprises a xylene cold soluble (XCS) fraction in a total amount of from 10.0 to 30.0 wt.-%, preferably from 11.5 to 27.5 wt.-%, more preferably from 12.5 to 25.0 wt.-%, based on the total weight amount of the polypropylene composition and determined according to ISO16152.

Said xylene cold soluble (XCS) fraction preferably has a comonomer content, preferably ethylene content, of from 20.0 to 35.0 wt.-%, more preferably from 21.5 to 32.5 wt.-% and most preferably from 23.0 to 30.0 wt.-%, based on the total amount of monomer units in the xylene cold soluble (XCS) fraction and determined determined by FT-IR spectroscopy calibrated by quantitative ^{13}C-NMR spectroscopy.

Further, said xylene cold soluble (XCS) fraction preferably has an intrinsic viscosity (iV(XCS)), determined according to ISO 1628-3, in the range from 90 to 210 cm^3/g, more preferably from 100 to 190 cm^3/g and most preferably from 115 to 170 cm^3/g.

The polypropylene composition preferably comprises a fraction insoluble in cold xylene soluble (XCI) in a total amount of from 70.0 to 90.0 wt.-%, preferably from 72.5 to 88.5 wt.-%, more preferably from 75.0 to 87.5 wt.-%, based on the total weight amount of the polypropylene composition and determined according to ISO16152.

Said fraction insoluble in cold xylene soluble (XCI) preferably has a comonomer content, preferably ethylene content, of from 1.0 to 15.0 wt.-%, more preferably from 2.0 to 10.0

wt.-% and most preferably from 2.5 to 8.5 wt.-%, based on the total amount of monomer units in the xylene cold soluble (XCI) fraction and determined by FT-IR spectroscopy calibrated by quantitative ^{13}C-NMR spectroscopy.

Further, said fraction insoluble in cold xylene soluble (XCI) preferably has an intrinsic viscosity (iV(XCI)), determined according to ISO 1628-3, in the range from 185 to 325 cm³/g, more preferably from 200 to 300 cm³/g and most preferably from 220 to 270 cm³/g.

It is preferred that the fraction insoluble in cold xylene soluble (XCI) and the xylene cold soluble (XCS) fraction make up 100 wt.-% of the polypropylene composition.

The polypropylene composition has a flexural modulus of from more than 470 MPa to 750 MPa, more preferably of from 475 MPa to 700 MPa and most preferably of from 500 MPa to 650 MPa, determined according to ISO 178 method A.

Further, the polypropylene composition preferably has a Charpy notched impact strength at 23°C of from 5.0 to 30.0 kJ/m², more preferably from 7.0 to 25.0 kJ/m² and most preferably from 9.0 to 20.0 kJ/m², determined according to ISO 179-1/1eA.

Still further, the polypropylene composition preferably has a Charpy notched impact strength at -20°C of from 0.8 to 5.0 kJ/m², more preferably from 0.8 to 4.0 kJ/m² and most preferably from 0.8 to 3.5 kJ/m², determined according to ISO 179-1/1eA.

Furthermore, the polypropylene composition preferably has a melting temperature Tm of from 132 to 155°C, preferably from 135 to 150°C and most preferably from 137 to 147°C, determined by to DSC analysis according to ISO 11357 / part 3 /method C2.

Further, the polypropylene composition preferably has a crystallization temperature Tc of from 85 to 115°C, preferably from 90 to 110°C and most preferably from 95 to 107°C, determined by to DSC analysis according to ISO 11357 / part 3 /method C2.

The difference of the melting temperature to the crystallization temperature Tm-Tc is preferably in the range of from 35 to 55°C, more preferably 37 to 52°C and most preferably from 39 to 50°C.

The polypropylene composition comprises a copolymer of propylene and comonomer units selected from ethylene and alpha-olefins having from 4 to 12 carbon atoms.

The comonomer units are preferably selected from ethylene and alpha-olefins having from 4 to 8 carbon atoms, such as ethylene, 1-butene, 1-hexene and 1-octene, most preferably from ethylene.

Said copolymer of propylene and comonomer units selected from ethylene and alpha-olefins having from 4 to 12 carbon atoms preferably is a copolymer of propylene and ethylene.

The copolymer of propylene and ethylene preferably consists of propylene and ethylene. Thus, it is preferred that the total ethylene content and the total propylene content make up 100 wt.-% of the copolymer of propylene and ethylene.

Said copolymer of propylene and comonomer units selected from ethylene and alpha-olefins having from 4 to 12 carbon atoms, is preferably a heterophasic copolymer of propylene and ethylene.

The heterophasic copolymer of propylene and ethylene has a matrix phase and an elastomeric phase dispersed in said matrix phase.
The matrix phase is preferably a propylene-ethylene random copolymer.

The copolymer of propylene and comonomer units selected from ethylene and alpha-olefins having from 4 to 12 carbon atoms can be polymerized in a sequential multistage polymerization process, i.e. in a polymerization process in which two or more polymerization reactors are connected in series. Preferably, in the sequential multistage polymerization process, two or more, more preferably three or more, such as three or four, polymerization reactors are connected in series. The term “polymerization reactor” shall indicate that the main polymerization takes place. Thus in case the process

consists of four polymerization reactors, this definition does not exclude the option that the overall process comprises for instance a pre-polymerization step in a pre-polymerization reactor.

When the copolymer of propylene and comonomer units selected from ethylene and alpha-olefins having from 4 to 12 carbon atoms is a heterophasic copolymer of propylene and ethylene, the matrix phase of the heterophasic copolymer of propylene and ethylene is polymerized in first polymerization reactor for producing a unimodal matrix phase or in the first and second polymerization reactor for producing a multimodal matrix phase.
The elastomeric phase of the heterophasic copolymer of propylene and ethylene is preferably polymerized in the subsequent one or two polymerization reactor(s) in the presence of the matrix phase for producing a unimodal elastomeric phase or a multimodal elastomeric phase.

Preferably, the polymerization reactors are selected from slurry phase reactors, such as loop reactors and/or gas phase reactors such as fluidized bed reactors, more preferably from loop reactors and fluidized bed reactors.

A preferred sequential multistage polymerization process is a “loop-gas phase”-process, such as developed by Borealis A/S, Denmark (known as BORSTAR® technology) described e.g. in patent literature, such as in EP 0 887 379, WO 92/12182 WO 2004/000899, WO 2004/111095, WO 99/24478, WO 99/24479 or in WO 00/68315.

A further suitable slurry-gas phase process is the Spheripol® process of LyondellBasell.

Suitable sequential polymerization processes for polymerizing the copolymer of propylene and comonomer units selected from ethylene and alpha-olefins having from 4 to 12 carbon atoms, preferably the copolymer of propylene and ethylene, more preferably the heterophasic copolymer of propylene and ethylene, are e.g. disclosed in WO 2015/117948.

The copolymer of propylene and comonomer units selected from ethylene and alpha-olefins having from 4 to 12 carbon atoms, preferably the copolymer of propylene and

ethylene, more preferably the heterophasic copolymer of propylene and ethylene can be polymerized in the presence of a Ziegler-Natta catalyst.

Suitable Ziegler-Natta catalysts are e.g. disclosed in WO 2015/117948.

The copolymer of propylene and comonomer units selected from ethylene and alpha-olefins having from 4 to 12 carbon atoms, preferably the copolymer of propylene and ethylene, more preferably a heterophasic copolymer of propylene and ethylene, is preferably present in the polypropylene composition in an amount of from 95.0 to 100 wt.-%, preferably from 97.5 to 99.99 wt.-%, still more preferably from 99.0 to 99.95 wt.-%, based on the total weight of the polypropylene composition.

The polypropylene composition can further comprise polymeric components, which are different from the copolymer of propylene and comonomer units selected from ethylene and alpha-olefins having from 4 to 12 carbon atoms in an amount of preferably 0.0 to 10.0 wt% based on the total weight of the polypropylene composition.

In a preferred embodiment the polymeric components of the polypropylene composition consist of copolymer of propylene and comonomer units selected from ethylene and alpha-olefins having from 4 to 12 carbon atoms.

Besides these polymeric components the polypropylene composition can comprise one or more additives in an amount of from 0.0 up to 5.0 wt%, based on the total weight of the polypropylene composition. The one or more additives are preferably selected from acid scavengers, antioxidants, alpha nucleating agents, beta nucleating agents, etc. Such additives are commercially available and for example described in “Plastic Additives Handbook”, 6th edition 2009 of Hans Zweifel (pages 1141 to 1190).

Usually, these additives are added in quantities of 1 to 50000 ppm for each single component.

The one or more additives can be added to the polymeric components in a blending step.

Thereby, the one or more additives can be added to the polymeric components in form of master batches in which one or more additives are blended with a carrier polymer in

concentrated amounts. Any optional carrier polymer is calculated to the amount of additives, based on the total weight of the polypropylene composition.

Preferably, the polypropylene composition contains from 0 to 5.00 wt%, more preferably from 0 to 2.50 wt% of the alpha-nucleating agent, based on the total amount of the polypropylene composition.
The amount of pure alpha-nucleating agent in the polypropylene composition (without optional carrier polymer of a master batch) is preferably in the range of from 0 to 5000 ppm, more preferably from 0 to 4000 ppm, based on the total amount of the polypropylene composition.

The alpha-nucleating agent is generally not restricted.
Preferably the alpha-nucleating agent is selected from soluble alpha-nucleating agents and polymeric alpha-nucleating agents.

The alpha-nucleating agent is preferably selected from the group consisting of

(i) dibenzylidenesorbitol (e.g. 1,3 : 2,4 dibenzylidenesorbitol) and C_1-C_8-alkyl-substituted dibenzylidenesorbitol derivatives, such as methyldibenzylidenesorbitol, ethyldibenzylidenesorbitol or dimethyldibenzylidenesorbitol (e.g. 1,3 : 2,4 di(methylbenzylidene) sorbitol), or substituted nonitol-derivatives, such as 1,2,3-trideoxy-4,6:5,7-bis-O-[(4-propylphenyl)methylene]-nonitol, and
(ii) vinylcycloalkane polymer and vinylalkane polymer (as discussed in more detail below), and
(iii) mixtures thereof.

The alpha-nucleating agent is preferably selected from the group consisting of dibenzylidenesorbitol (e.g. 1,3 : 2,4 dibenzylidene sorbitol), dibenzylidenesorbitol derivative, preferably dimethyldibenzylidenesorbitol (e.g. 1,3 : 2,4 di(methylbenzylidene) sorbitol), or substituted nonitol-derivatives, such as 1,2,3-trideoxy-4,6:5,7-bis-O-[(4-propylphenyl)methylene]-nonitol, vinylcycloalkane polymer, vinylalkane polymer, and mixtures thereof.
Especially preferred are vinylcycloalkane polymers such as e.g. vinylcyclohexane (VCH) polymers. Such polymers can be added e.g. using Borealis Nucleation Technology (BNT).

In a preferred embodiment the alpha-nucleating agent is a soluble alpha-nucleating agent, more preferably a soluble alpha-nucleating agent selected from dibenzylidenesorbitol (e.g. 1,3 : 2,4 dibenzylidenesorbitol) and C_1-C_8-alkyl-substituted dibenzylidenesorbitol derivatives, such as methyldibenzylidenesorbitol, ethyldibenzylidenesorbitol or dimethyldibenzylidenesorbitol (e.g. 1,3 : 2,4 di(methylbenzylidene) sorbitol), or substituted nonitol-derivatives, such as 1,2,3-trideoxy-4,6:5,7-bis-O-[(4-propylphenyl)methylene]-nonitol and mixtures thereof.

The alpha-nucleating agent can be added to the polypropylene composition as an isolated raw material or in a mixture with a carrier polymer, i.e. in a so-called master batch. The amount of the carrier polymer of the master batch thereby is calculated to the amount of the alpha-nucleating agent.

In one embodiment the polypropylene composition comprises an alpha-nucleating agent.
In said embodiment, the pure amount of alpha-nucleating is preferably in the range of from 0.1 to 5000 ppm, more preferably from 1 to 5000 ppm.

In another embodiment, the polypropylene composition does not comprise an alpha-nucleating agent.
In said embodiment, the pure amount of alpha-nucleating is 0 ppm.

Preferably, the polypropylene composition is prepared by melt blending the copolymer of propylene and comonomer units selected from ethylene and alpha-olefins having from 4 to 12 carbon atoms, the optional additional polymeric components and the optional further additives, all as described above or below.

The polypropylene composition has a melt flow rate MFR_2 of from more than 2.5 to 6.0 g/10 min, preferably from 2.8 to 5.5 g/10 min, still more preferably from 3.0 to 5.0 g/10 min and most preferably from 3.3 to 4.5 g/10 min, determined according to ISO 1133 at 230°C and 2.16 kg.

The polypropylene composition is thereby preferably not subjected to vis-breaking.

in one embodiment the polypropylene composition does not comprise, i.e. is free of a dielectric fluid, such as e.g. described in EP 2 739 679.

Cable

The present invention relates to a cable comprising at least one layer comprising a polypropylene composition as described above or below.

It is thereby preferred that the polypropylene composition meets as properties and embodiments of the polypropylene composition as described above or below.

The at least one layer is preferably an insulation layer.

The at least one layer, preferably the insulation layer, preferably comprises from 95.0 to 100 wt.-%, preferably from 97.5 to 100 wt.-%, still more preferably from 99.0 to 100 wt.-%, based on the total weight of the insulation layer, most preferably consists of the polypropylene composition.

The cable usually comprises of at least one conductor and at least one insulation layer comprising the polypropylene composition as described above or below.

The term "conductor" means herein above and below that the conductor comprises one or more wires. The wire can be for any use and be e.g. optical, telecommunication or electrical wire. Moreover, the cable may comprise one or more such conductors. Preferably the conductor is an electrical conductor and comprises one or more metal wires. The cable is preferably a power cable. A power cable is defined to be a cable transferring energy operating at any voltage, typically operating at voltages higher than 1 kV. The voltage applied to the power cable can be alternating (AC), direct (DC), or transient (impulse). The poly polypropylene composition of the invention is very suitable for power cables, especially for power cables operating at voltages 6 kV to 36 kV (medium voltage (MV) cables) and at voltages higher than 36 kV, known as high voltage (HV) cables and extra high voltage (EHV) cables, which EHV cables operate, as well known, at very high voltages. The terms have well known meanings and indicate the operating level of such cables.

For low voltage applications the cable system typically either consists of one conductor and one insulation layer comprising the polypropylene composition as described above or below, or of one conductor, one insulation layer comprising the polypropylene composition as described above or below and an additional jacketing layer, or of one conductor, one semiconductive layer and one insulation layer comprising the polypropylene composition as described above or below.
For medium and high voltage applications the cable system typically consists of one conductor, one inner semiconductive layer, one insulation layer comprising the polypropylene composition as described above or below and one outer semiconductive layer, optionally covered by an additionally jacketing layer.

The semiconductive layers mentioned preferably comprise, more preferably consist of a thermoplastic polyolefin composition, preferably a polyethylene composition or a polypropylene composition, containing a sufficient amount of electrically conducting solid fillers preferably carbon black. It is preferred that the thermoplastic polyolefin composition of the inner semiconductive layer is a polypropylene composition, more preferably a polypropylene composition comprising a heterophasic propylene copolymer as polymeric component. It is further preferred that thermoplastic polyolefin composition of the inner semiconductive layer is a polyethylene composition, more preferably a polyethylene composition comprising a non-crosslinkable polyethylene.

The cable comprising an insulation layer comprising the polypropylene composition according to the invention as described above shows good AC electrical breakdown strength in form of Weibull alpha-value and Weibull beta-value.

The cable preferably has a Weibull alpha-value of at least 55 kV/mm, such as from 55.0 to 80.0 kV/mm, more preferably from 58.0 to 75.0 kV/mm and most preferably from 60.0 to 70.0 kV/mm, when measured on a 10 kV cable in agreement with CENELEC HD 605 5.4.15.3.4 for 6/10 kV cables.

Still further, the cable preferably has a Weibull beta-value of at least 15.0, such as from 15.0 to 250.0, more preferably from 20.0 to 250.0, most preferably from 25.0 to 250.0,

when measured on a 10 kV cable in agreement with CENELEC HD 605 5.4.15.3.4 for 6/10 kV cables.

Thus, the insulation layer comprising the polypropylene composition according to the invention can be used for medium and high voltage cables, preferably for high voltage cables, more preferably a high voltage AC cables.

Benefits of the invention:

The polypropylene composition shows a good balance of properties regarding a good mechanical strength and high crystallization and melting temperature as well as sufficient flexibility and impact properties, which allow the use as cable insulation e.g. for medium and high voltage cables at high operation temperatures.

The rather low soluble fraction (SF) content of the polypropylene composition of not more than 25.0 wt.-% allows for improved extrudability during cable extrusion.

The polyolefin composition thereby reaches melt flow rates of up to 6.0 g/10 min without need of increasing the melt flow rate via vis-breaking, which adds to the improved extrudability during cable extrusion.

Cables comprising an insulation layer comprising the polypropylene composition as described above or below surprisingly show good AC breakdown strength in form of Weibull alpha-value and Weibull beta-value. Thereby, the refraining from vis-breaking further improves the AC breakdown strength compared to polypropylene compositions, which were subjected to vis-breaking.

The good AC breakdown strength in form of Weibull alpha-value and Weibull beta-value can be obtained without addition of a dielectric fluid such as e.g. described in EP 2 739 679.

Examples

The following definitions of terms and determination methods apply for the above general description of the invention as well as to the below examples unless otherwise defined.

1. **Measurement methods**

a) Melt Flow Rate (MFR_2)

The melt flow rate is the quantity of polymer in grams which the test apparatus standardized to ISO 1133 extrudes within 10 minutes at a certain temperature under a certain load.

The melt flow rate MFR_2 of propylene based polymers and the polyolefin composition is measured at 230°C with a load of 2.16 kg according to ISO 1133.

The melt flow rate MFR_5 of the polyolefin composition is measured at 230°C with a load of 5.0 kg according to ISO 1133.

The melt flow rate MFR_2 of the ethylene based polymers is measured at 190°C with a load of 2.16 kg according to ISO 1133.

b) Density

The density is measured according to ISO 1183-1:2004 Method A on compression moulded specimen prepared according to EN IS0 1872-2 (Feb 2007) and is given in kg/m^3.

c) Comonomer content

Comonomer content quantification of poly(propylene-co-ethylene) copolymers

Quantitative $^{13}C\{^1H\}$ NMR spectra were recorded in the solution-state using a Bruker Avance NEO 400 NMR spectrometer operating at 400.15 and 100.62 MHz for ^1H and ^{13}C respectively. All spectra were recorded using a ^{13}C optimised 10 mm extended temperature probe head at 125°C using nitrogen gas for all pneumatics. Approximately 200 mg of material was dissolved in 3 ml of *1,2*-tetrachloroethane-d_2 (TCE-d_2) along with chromium-(III)-acetylacetonate ($Cr(acac)_3$) resulting in a 60 mM solution of relaxation agent in solvent {8} and with approximately 3 mg BHT (2,6-di-tert-butyl-4-methylphenol CAS 128-37-0) . To ensure a homogenous solution, after initial sample preparation in a heat block, the NMR tube was further heated in a rotatory oven for at least 1 hour. Upon insertion into the magnet the tube was spun at 10 Hz. This setup was chosen primarily for the high resolution and quantitatively needed for accurate ethylene content quantification. Standard single-pulse excitation was employed without NOE, using an optimised tip angle, 1 s recycle delay and a bi-level WALTZ16 decoupling scheme {3, 4}. A total of 6144 (6k) transients were acquired per spectra.

Quantitative $^{13}C\{^1H\}$ NMR spectra were processed, integrated and relevant quantitative properties determined from the integrals using proprietary computer programs. All

chemical shifts were indirectly referenced to the central methylene group of the ethylene block (EEE) at 30.00 ppm using the chemical shift of the solvent. This approach allowed comparable referencing even when this structural unit was not present. Characteristic signals corresponding to the incorporation of ethylene were observed {7}.

The comonomer fraction was quantified using the method of Wang et. al. {6} through integration of multiple signals across the whole spectral region in the $^{13}C\{^{1}H\}$ spectra. This method was chosen for its robust nature and ability to account for the presence of regiodefects when needed. Integral regions were slightly adjusted to increase applicability across the whole range of encountered comonomer contents.

For systems where only isolated ethylene in PPEPP sequences was observed the method of Wang et al. was modified to reduce the influence of non-zero integrals of sites that are known to not be present. This approach reduced the overestimation of ethylene content for such systems and was achieved by reduction of the number of sites used to determine the absolute ethylene content to:

$E = 0.5 (S\beta\beta + S\beta\gamma + S\beta\delta + 0.5(S\alpha\beta + S\alpha\gamma))$

Through the use of this set of sites the corresponding integral equation becomes:

$E = 0.5 (I_H + I_G + 0.5(I_C + I_D))$

using the same notation used in the article of Wang et al. {6}. Equations used for absolute propylene content were not modified.

The mole percent comonomer incorporation was calculated from the mole fraction:

E [mol%] = 100 * fE

The weight percent comonomer incorporation was calculated from the mole fraction:

E [wt%] = 100 * (fE * 28.06) / ((fE * 28.06) + ((1-fE) * 42.08))

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d) Differential scanning calorimetry (DSC) analysis, melting temperature (Tm) and crystallization temperature (Tc):

measured with a TA Instrument Q2000 differential scanning calorimetry (DSC) on 5 to 7 mg samples. DSC is run according to ISO 11357 / part 3 /method C2 in a heat / cool /heat cycle with a scan rate of 10°C/min in the temperature range of -30°C to +225°C. Crystallization temperature and heat of crystallization (Hc) are determined from the cooling step, while melting temperature and heat of fusion (Hf) are determined from the second heating step.

When a sample shows two or more melting temperatures and/or crystallization temperatures only the main melting temperature (at the highest Hf) and main crystallization temperature (at the highest Hc) are displayed in the accordant table. The difference of melting temperature and crystallization temperature (Tm-Tc) is given for the main melting temperature and the main crystallization temperature.

e) Crystallisation extraction (CRYSTEX) analysis

Note: Crystallization extraction (CRYSTEX) analyses the polymeric part of each component, with non-polymeric parts, such as any fillers or particulate pigments, not contributing to the reported CRYSTEX data presented.

Determination of Crystalline and soluble fractions and their respective properties (iV and Ethylene content)

The crystalline (CF) and soluble fractions (SF) of the polypropylene (PP) compositions as well as the comonomer content and intrinsic viscosities of the respective fractions were analyzed by use of the Crystex (crystallisation extraction) method. Potential instruments that can be used are Crystex QC or Crystex 42 (Polymer Char; Valencia, Spain). Details of the technique and the method can be found in literature (Ljiljana Jeremic, Andreas Albrecht, Martina Sandholzer & Markus Gahleitner (2020): Rapid characterization of high-impact ethylene–propylene copolymer composition by crystallization extraction separation: comparability to standard separation

methods, International Journal of Polymer Analysis and Characterization, 25:8, 581-596).

The crystalline and amorphous fractions are separated through temperature cycles of dissolution at 160°C, crystallization at 40°C and re-dissolution in 1,2,4-trichlorobenzene at 160°C. Quantification of SF and CF and determination of ethylene content (C2) are achieved by means of an integrated infrared detector (IR4) and for the determination of the intrinsic viscosity (IV) an online 2-capillary viscometer is used.

IR4 detector is a multiple wavelength detector measuring IR absorbance at two different bands (CH3 stretching vibration (centred at app. 2960 cm^{-1}) and the CH stretching vibration (2700-3000 cm^{-1}) that are serving for the determination of the concentration and the Ethylene content in Ethylene-Propylene copolymers. IR4 detector is calibrated with series of 8 EP copolymers with known Ethylene content in the range of 2 wt.-% to 69 wt.-% (determined by 13C-NMR) and each at various concentrations, in the range of 2 and 13mg/ml. To encounter for both features, concentration and ethylene content at the same time for various polymer concentration expected during Crystex analyses the following calibration equations were applied:

$$Conc = a + b*Abs(CH) + c*(Abs(CH))^2 + d*Abs(CH_3) + e*(Abs(CH_3)^2 + f*Abs(CH)*Abs(CH_3) \quad \text{(Equation 1)}$$

$$CH_3/1000C = a + b*Abs(CH) + c* Abs(CH_3) + d * (Abs(CH_3)/Abs(CH)) + e * (Abs(CH_3)/Abs(CH))^2 \quad \text{(Equation 2)}$$

The constants a to f for equation 1 and a to e for equation 2 were determined by using least square regression analysis.

The CH_3/1000C is converted to the ethylene content in wt.-% using following relationship:

$$\text{wt.-\% (Ethylene in EP Copolymers)} = 100 - CH_3/1000TC * 0.3 \quad \text{(Equation 3)}$$

Amount of Soluble Fraction (SF) and Crystalline Fraction (CF) are correlated through the XS calibration to the “Xylene Cold Soluble” (XCS) quantity and respectively Xylene Cold Insoluble (XCI) fractions, determined according to standard gravimetric method as per ISO16152. XS calibration is achieved by testing various EP copolymers with XS content in the range 2-31 wt.-%. A linear calibration curve is used.

Intrinsic viscosity (iV) of the parent EP copolymer and its soluble and crystalline fractions are determined with a use of an online 2-capillary viscometer and are correlated to corresponding IV's determined by standard method in decalin according to ISO 1628-3. Calibration is achieved with various EP copolymers and PP polymers with iV = 2-4 dL/g. The determined calibration curve is linear.

The samples to be analyzed are weighed out in concentrations of 10 mg/ml to 20 mg/ml. After automated filling of the vial with 1,2,4-TCB containing 250 mg/l 2,6-tert-butyl-4-methylphenol (BHT) as antioxidant, the sample is dissolved at 170°C until complete dissolution is achieved with either constant stirring or gentle shaking. To avoid sample degradation, polymer solution is blanketed with the N_2 atmosphere during dissolution. For PP composition containing inorganic fillers or pigments or any other non-TCB soluble polymeric substances removal of these is required. This can be done by hot filtration prior injection.

A defined volume of the polymer solution is injected into the column filled with inert support where the crystallization of the sample and separation of the soluble fraction from the crystalline fraction is taking place. This process is repeated two times. During the first injection the whole sample is measured at high temperature, determining the iV [dl/g] and the C2 [wt.-%] of the PP composition. During the second injection the soluble fraction (at low temperature) and the crystalline fraction (at high temperature) with the crystallization cycle are measured (wt.-% SF, wt.-% C2, iV).

f) Intrinsic viscosity (iV)

The reduced viscosity (also known as viscosity number), η_{red}, and intrinsic viscosity, iV, are determined according to ISO 1628-3: "Determination of the viscosity of polymers in dilute solution using capillary viscometers".

Relative viscosities of a diluted polymer solution with concentration of 1 mg/ml and of the pure solvent (decahydronaphthalene stabilized with 200 ppm 2,6-bis(1,1-dimethylethyl)-4-methylphenol) are determined in an automated capillary viscometer (Lauda PVS1) equipped with 4 Ubbelohde capillaries placed in a thermostatic bath filled with silicone oil. The bath temperature is maintained at 135 °C. The sample is dissolved with constant stirring until complete dissolution is achieved (typically within 90 min).

The efflux time of the polymer solution as well as of the pure solvent are measured several times until three consecutive readings do not differ for more than 0.2s (standard deviation).

The relative viscosity of the polymer solution is determined as the ratio of averaged efflux times in seconds obtained for both, polymer solution and solvent:

$$\eta_{rel} = \frac{t_{solution} - t_{solvent}}{t_{solvent}} \qquad \text{[dimensionless]}$$

Reduced viscosity (η_{red}) is calculated using the equation:

$$\eta_{red} = \frac{t_{solution} - t_{solvent}}{t_{solvent} * C} \qquad \text{[dl/g]}$$

where C is the polymer solution concentration at 135°C: $C = \frac{m}{V\gamma}$,

and *m* is the polymer mass, *V* is the solvent volume, and γ is the ratio of solvent densities at 20°C and 135°C ($\gamma=\rho_{20}/\rho_{135}=1.107$).

The calculation of intrinsic viscosity iV is performed by using the Schulz-Blaschke equation from the single concentration measurement:

$$iV = \frac{\eta_{red}}{1 + K + C + \eta_{red}}$$

where K is a coefficient depending on the polymer structure and concentration. For calculation of the approximate value for IV, K=0.27.

g) Flexural Modulus

The flexural modulus was determined acc. to ISO 178 method A (3-point bending test) on 80 mm × 10 mm × 4 mm specimens. Following the standard, a test speed of 2 mm/min and a span length of 16 times the thickness was used. The testing temperature was 23±2° C. Injection moulding was carried out according to ISO 19069-2 using a melt temperature of 230°C for all materials irrespective of material melt flow rate.

h) Charpy notched impact strength

The Charpy notched impact strength was determined acc. to ISO 179-1/1eA on notched 80 mm × 10 mm × 4 mm specimens (specimens were prepared according to ISO 179-1/1eA). Testing temperatures were 23±2° C or -20±2° C. Injection moulding was carried

out acc. to ISO 19069-2 using a melt temperature of 230°C for all materials irrespective of material melt flow rate.

i) Xylene cold solubles (XCS) content

The quantity of xylene soluble matter in polypropylene is determined according to the ISO16152 (first edition; 2005-07-01).

A weighed amount of a sample is dissolved in hot xylene under reflux conditions at 135°C. The solution is then cooled down under controlled conditions and maintained at 25°C for 30 minutes to ensure controlled crystallization of the insoluble fraction. This insoluble fraction is then separated by filtration. Xylene is evaporated from the filtrate leaving the soluble fraction as a residue. The percentage of this fraction is determined gravimetrically.

$$\%XS = \frac{m_1 \times v_0}{m_0 \times v_1} \times 100$$

where

m_0 is the mass of the sample test portion weighed, in grams

m_1 is the mass of residue, in grams

v_0 is the original volume of solvent taken

v_1 is the volume of the aliquot taken for determination.

j) AC electric breakdown strength (ACBD)

The AC breakdown tests were performed in agreement with CENELEC HD 605 5.4.15.3.4 for 6/10 kV cables. The cable was thus cut into six test samples of 10 meter active length (terminations in addition). The samples were tested to breakdown with a 50 Hz AC step test at ambient temperature, according to the following procedure:

- Start at 18 kV for 5 minutes
- Voltage increasing in step of 6 kV every 5 minutes until breakdown occurs

The calculation of the Weibull parameters of the data set of six breakdown values (conductor stress, i.e. the electric field at the inner semiconductive layer) follows the least squares regression procedure as described in IEC 62539 (2007). The Weibull alpha parameter in this document refers to the scale parameter of the Weibull distribution, i.e. the voltage for which the failure probability is 0.632. The Weibull beta value refers to the shape parameter.

2. Polypropylene composition

The following resins were used for the preparation of the polypropylene compositions of the examples:

a) Polymerization of the heterophasic propylene copolymer powders A and B

- Catalyst

The catalyst used in the polymerization process for the heterophasic propylene copolymer powder A was a Ziegler-Natta catalyst, which is described in the example section of WO 2015/117948. As co-catalyst triethyl-aluminium (TEAL) and as donor dicyclo pentyl dimethoxy silane (D-donor) was used.

The catalyst used in the polymerization process for the heterophasic propylene copolymer powder B was a Ziegler-Natta catalyst, which is described in patent publications EP491566, EP591224 and EP586390. As co-catalyst triethyl-aluminium (TEAL) and as donor dicyclo pentyl dimethoxy silane (D-donor) was used.

- Polymerization of the heterophasic propylene copolymer powders A and B

Heterophasic propylene copolymer powders A and B were produced in a Borstar™ plant in the presence of the above described polymerization catalysts using one liquid-phase loop reactor and two gas phase reactors connected in series under conditions as shown in Table 1. The first reaction zone was a loop reactor and the second and third reaction zones were gas phase reactors. The matrix phase was polymerized in the loop and first gas phase reactor and the elastomeric phase was polymerized in the second gas phase reactor. The catalysts as described above were fed into a prepolymerization reactor which precedes the first reaction zone.

Table 1: Polymerization conditions of the heterophasic propylene copolymer powders A and B:

		POWDER A	POWDER B
Prepolymerization			
Temperature	[°C]	20	20
Residence time	[h]	0.60	0.40
TEAL/Ti ratio	[mol/mol]	353	125
Donor/Ti ratio	[mol/mol]	24	17
Loop			
Split (Loop + Prepol)	[%]	33.3	37.2
Temperature	[°C]	70	70

C2/C3 ratio (calc)	[mol/kmol]	18.8	22.4
H2/C3 ratio	[mol/kmol]	5.3	1.8
MFR (230°C/2.16kg)	[g/10 min]	5.2	3.9
C2 content	[wt%]	2.2	2.5
GPR 1			
Split (GPR1)	[%]	45.8	52.5
Temperature	[°C]	75	85
C2/C3 ratio	[mol/kmol]	69.9	24.6
H2/C3 ratio	[mol/kmol]	24.2	7.5
MFR (230°C/2.16kg)	[g/10 min]	1.8	3.3
C2 content	[wt%]	6.6	4.4
GPR 2			
Split (GPR2)	[%]	20.9	10.3
Temperature	[°C]	80	75
C2/C3 ratio	[mol/kmol]	399	392
H2/C3 ratio	[mol/kmol]	182	152
MFR (230°C/2.16kg)	g/10 min	1.4	3.9
C2 content	[wt%]	13.1	7.9
XCS	[wt%]	35.1	18.0

b) Preparation of the polypropylene compositions

The heterophasic propylene copolymer powders A and B from the polymerization reaction were compounded in a twin screw extruder together with a stabilizer package to obtain the polypropylene compositions of IE1, CE1 and CE2.

During compounding of CE1, BNT alpha-nucleating agent was added and the composition was vis-broken to a melt flow rate MFR_2 (230°C, 2.16 kg) of 3.9 g/10 min as disclosed in the example section of WO 2017/198633.

IE1 and CE1 were used for the production of the insulation layers, whereas CE2 was used as polymeric compound for the inner semiconductive layers of the test cables.

An overview of the production of compositions IE1, CE1 and CE2 is shown in Table 2.

Table 2: Compounding of compositions IE1, CE1 and CE2 in a twin screw extruder:

		IE1	CE1	CE2
HECO-powder		Powder B	Powder A	Powder A
Visbreaking		no	yes	no
Stabilizer onepack 1	[wt.-%]	0.19	-	-
Stabilizer onepack 2	[wt.-%]	-	0.14	-
Stabilizer onepack 3	[wt.-%]	-	-	0.2

Alpha-NA BNT	[wt.-%]	-	2.0	-

Stabilizer packages and alpha-nucleating agents:

- Stabilizer onepack 1 consists of 25.6 wt% Pentaerythrityl-tetrakis(3-(3',5'-di-tert. butyl-4-hydroxyphenyl)-propionate (CAS-No. 6683-19-8), 51.3wt% Tris (2,4-di-*t*-butylphenyl) phosphite (CAS-No. 31570-04-4) and 23.1 wt% synthetic hydrotalcite (CAS-No. 11097-59-9), all commercially available from a variety of companies.

- Stabilizer onepack 2 consists of 29 wt% Pentaerythrityl-tetrakis(3-(3',5'-di-tert. butyl-4-hydroxyphenyl)-propionate (CAS-No. 6683-19-8), 58 wt% Tris (2,4-di-*t*-butylphenyl) phosphite (CAS-No. 31570-04-4) and 13 wt% Magnesium Oxide (CAS-No. 1309-48-4), all commercially available from a variety of companies.

- Stabilizer onepack 3 consists of 21.8 wt% Pentaerythrityl-tetrakis(3-(3',5'-di-tert. butyl-4-hydroxyphenyl)-propionate (CAS-No. 6683-19-8), 43.6 wt% Tris (2,4-di-*t*-butylphenyl) phosphite (CAS-No. 31570-04-4) and 34.6 wt% Calcium stearate (CAS-No. 1592-23-0), all commercially available from a variety of companies.

- Alpha-nucleation via BNT was achieved by adding 2 wt% of a propylene homopolymer with an MFR2 (230°C) of 8.0 g/10 min and a melting temperature of 162 °C, which is produced with a Ziegler-Natta type catalyst in the Borealis nucleation technology (BNT), comprising a polymeric α-nucleating agent, and is distributed by Borealis AG (Austria).

Table 3: Properties of the compounded compositions of IE1, CE1 and CE2

		IE1	CE1	CE2
MFR_2	[g/10 min]	4.0	3.9	1.5
Flex. modulus	[MPa]	620	378	335
Charpy NIS 23°C	[kg/m³]	10.0	78.9	83.9
Charpy NIS -20°C	[kg/m²]	1.0	4.2	6.3
Tm	[°C]	144	148	150
Tc	[°C]	103	115	100
Tm-Tc	[°C]	41	33	50
C2 total (CRYSTEX)	[wt.-%]	8.1	11.8	12.7
iV total (CRYSTEX)	[cm³/g]	222	213	277

CF (CRYSTEX)	[wt.-%]	83.4	65.3	64.3
C2 of CF (CRYSTEX)	[wt.-%]	4.9	5.0	5.2
iV of CF (CRYSTEX)	[cm³/g]	238	217	277
SF (CRYSTEX)	[wt.-%]	16.6	34.7	35.7
C2 of SF(CRYSTEX)	[wt.-%]	24.6	25.3	27.2
iV of SF(CRYSTEX)	[cm³/g]	139	200	261
C2 of SF / C2 of CF	[-]	5.0	5.1	5.2
iV of SF / iV of CF	[-]	0.58	0.94	0.94
XCS	[wt.-%]	16.3	35.6	35.6
C2(XCS)	[wt.-%]	28.4	27.4	29.1
iV(XCS)	[cm³/g]	153	189	251
XCI	[wt.-%]	83.7	64.4	64.4
C2(XCI)	[wt.-%]	4.3	6.3	7.0
iV(XCI)	[cm³/g]	229	216	286

Inventive composition IE1 shows the same melt flow rate as comparative composition CE1 without need for vis-breaking. IE1 shows a lower amount of soluble fraction in the CRYSTEX measurement and XCS measurement compared to CE1. Flexibility, impact properties and melting temperature of IE1 are sufficient for cable properties. Crystallization temperature of CE1 is increased due to the presence of the alpha-nucleating agent.

3. Production of 10 kV cables

10 kV test cables were produced on a Maillefer pilot cable line of catenary continuous vulcanizing (CCV) type.

The conductors of the cable cores had a cross section being 50 mm² of stranded aluminium. The inner semiconductive layer was produced from semiconductive composition SC1 as described below and had a thickness of 1.0 mm. The insulation layer was produced from the above described compositions CE1 and IE1, and had a thickness of 3.4 mm. The outer semiconductive layer was produced from semiconductive compositions SC2 as described below and had a thickness of 1.0 mm. The cables, i.e. cable cores, were produced by extrusion via a triple head. The insulation extruder had size 100 mm, the extruder for conductor screen (inner semiconductive layer) 45 mm, and the extruder for insulation screen (outer semiconductive layer) 60 mm. The line speed was 6.0 m/min.

The vulcanisation tube had a total length of 52.5 meter consisting of a curing section followed by a cooling section. The curing section was filled with N_2 at 10 bar but not heated. The 33-meter-long cooling section was filled with 20-25°C water.
The pilot cables were then subjected to AC breakdown testing.

Semiconductive composition 1 (SC1) was prepared from 66.5 wt% of the polypropylene based composition of CE2 with 33.0 wt% of carbon black Printex Alpha, commercially available from Orion Engineered Carbons GmbH, and 0.5 wt% of maleic anhydride grafted propylene homopolymer Exxelor PO1020 having a melt flow rate at 230°C and a load of 2.16 kg of 430 g/10 min, commercially available from ExxonMobil.

Semiconductive composition 2 (SC2) was prepared from ready-to-use semiconductive composition Borlink LE7710, which is a non-crosslinkable polyethylene based composition comprising carbon black, commercially available from Borealis AG.

Table 4 shows the electric properties of the 10 kV cables of examples C1 and C2 in which the inventive insulation layer prepared from composition IE1 is compared to reference insulation layer prepared from composition CE1.

Table 4: Electric properties of 10 kV cables of C1 and C2

	C1	C2
Insulation layer	CE1	IE1
Inner semiconductive layer	SC1	SC1
Outer semiconductive layer	SC2	SC2
Weibull-alpha (scale) [kV/mm]	52.8	62.0
Weibull-beta (shape)	8.9	30.9

It can be seen that cable C2 comprising the inventive insulation layer IE1 shows an increased Weibull-alpha value and increased Weibull-beta value compared to the cable C1 comprising the reference insulation layer CE1.
The Weibull-alpha value of more than 60 kV/mm allows for use as high voltage AC cable.

Claims

1. A cable comprising at least one layer comprising a polypropylene composition, which comprises a copolymer of propylene and comonomer units selected from ethylene and alpha-olefins having from 4 to 12 carbon atoms,
wherein the polypropylene composition has
a total comonomer content of from 4.0 to 11.0 wt.-%, based on the total weight of the polypropylene composition and determined by FT-IR spectroscopy calibrated by quantitative ^{13}C-NMR spectroscopy from crystallization extraction (CRYSTEX);
a melt flow rate MFR_2 of from more than 2.5 to 6.0 g/10 min, determined according to ISO 1133 at 230°C and 2.16 kg;
a soluble fraction (SF) content, determined by crystallization extraction (CRYSTEX), in the range from 10.0 to 25.0 wt.-%, based on the total weight amount of the polypropylene composition; and
a flexural modulus of from more than 470 MPa to 750 MPa, determined according to ISO 178 method A.

2. The cable according to claim 1, wherein the copolymer of propylene and comonomer units selected from ethylene and alpha-olefins having from 4 to 12 carbon atoms, is a copolymer of propylene and ethylene.

3. The cable according to claim 1 or 2, wherein said soluble fraction (SF) has
a comonomer content, determined by FT-IR spectroscopy calibrated by quantitative ^{13}C-NMR spectroscopy, in the range from 20.0 to 35.0 wt%, based on the total amount of monomer units in the soluble fraction (SF), and/or
an intrinsic viscosity (iV(SF)), determined according to ISO 1628-3, in the range from 90 to 190 cm³/g.

4. The cable according to any one of claims 1 to 3, wherein the polypropylene composition has
a crystalline fraction (CF) content, determined by crystallization extraction (CRYSTEX), in the range from 75.0 to 90.0 wt.-%, based on the total weight amount of the polypropylene composition,
said crystalline fraction (CF) having a comonomer content, determined by FT-IR spectroscopy calibrated by quantitative ^{13}C-NMR spectroscopy, in the range from

1.0 to 15.0 wt%, based on the total amount of monomer units in the crystalline fraction (CF), and/or
an intrinsic viscosity (iV(CF)), determined according to ISO 1628-3, in the range from 185 to 325 cm³/g.

5. The cable according to claim 4, wherein the ratio of comonomer content of the soluble fraction to the comonomer content of the crystalline fraction is in the range of from 1.5 : 1.0 to 10.0 : 1.0; and/or
the ratio of the intrinsic viscosity of the soluble fraction to the intrinsic viscosity of the crystalline fraction (iV/SF)/iV(CF)) is in the range of from 0.35 : 1.00 to 0.90 : 1.00.

6. The cable according to any one of claims 1 to 5, wherein the polypropylene composition has a total intrinsic viscosity (iV (total)) determined according to ISO 1628-3, in the range from 175 to 300 cm³/g.

7. The cable according to any one of claims 1 to 6, wherein the polypropylene composition has one or more or all of the following properties:
- a Charpy notched impact strength at 23°C of from 5.0 to 30.0 kJ/m², determined according to ISO 179-1/1eA; and/or
- a Charpy notched impact strength at -20°C of from 0.8 to 5.0 kJ/m², determined according to ISO 179-1/1eA.

8. The cable according to any one of claims 1 to 7, wherein the polypropylene composition has one or more or all of the following properties:
- a melting temperature Tm of from 132 to 155°C, determined by to DSC analysis according to ISO 11357 / part 3 /method C2; and/or
- a crystallization temperature Tc of from 85 to 115°C, determined by to DSC analysis according to ISO 11357 / part 3 /method C2; and/or
- a difference of the melting temperature to the crystallization temperature Tm-Tc is preferably in the range of from 35 to 55°C.

9. The cable according to any one of claims 1 to 8, wherein the polypropylene composition is not subjected to vis-breaking.

10. The cable according to any one of claims 1 to 9, wherein the polypropylene composition is free of a dielectric fluid.

11. The cable according to any one of claims 1 to 10, wherein the copolymer of propylene and comonomer units selected from ethylene and alpha-olefins having from 4 to 12 carbon atoms is present in the polypropylene composition in an amount of from 95.0 to 100 wt.-%, based on the total weight of the polypropylene composition.

12. The cable according to any one of claims 1 to 11, wherein the polypropylene composition is present in the at least one layer in an amount of from 95.0 to 100 wt.-%, based on the total weight of the layer.

13. The cable according to any one of claims 1 to 12, wherein the at least one layer is an insulation layer.

14. The cable according to any one of claims 1 to 13 being a medium or high voltage cable.

15. The cable according to any one of claims 1 to 14 having an AC breakdown strength as expressed by a Weibull alpha-value of at least 55 kV/mm, and/or a Weibull beta-value of at least 15.0, measured on a 10 kV cable in agreement with CENELEC HD 605 5.4.15.3.4 for 6/10 kV cables.

INTERNATIONAL SEARCH REPORT

International application No
PCT/EP2024/076998

A. CLASSIFICATION OF SUBJECT MATTER

INV. C08L23/14 H01B3/44 C08F2/00 C08F210/06
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 C08F

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.
A	US 2012/220727 A1 (KLIMKE KATJA [AT] ET AL) 30 August 2012 (2012-08-30) cited in the application examples IE3-IE5; tables 2,3 claim 17 paragraphs [0002], [0011], [0012]	1-15
A	US 4 259 410 A (DITTMANN WALTER ET AL) 31 March 1981 (1981-03-31) cited in the application examples 1,2 claim 1 column 1, line 6 - line 8	1-15

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[x] Further documents are listed in the continuation of Box C.

[x] 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

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

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"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
16 November 2024	26/11/2024

Name and mailing address of the ISA/	Authorized officer
European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Fax: (+31-70) 340-3016	van Bergen, Marc

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INTERNATIONAL SEARCH REPORT

International application No
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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 2 602 287 A1 (BOREALIS AG [AT]) 12 June 2013 (2013-06-12) cited in the application claims 1,6 tables 1a,1b,2 paragraph [0129] -----	1-15

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INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No
PCT/EP2024/076998

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