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(54) **Polyolefin fibres**

(57) A fibre comprising a polyolefin composition wherein said composition comprises (per cent by weight):
a) from 50% to 85%, of a propylene polymer having an amount of isotactic pentads (mmmm), measured by ¹³C-MNR on the fraction insoluble in xylene at 25° C, higher than 97.5 molar %;
b) from 5% to 20%, of a copolymer of ethylene and propylene, the copolymer having an amount of recurring units deriving from ethylene ranging from 30 to 50%, and

being partially soluble in xylene at 25° C; the polymer fraction soluble in xylene at 25° C having an intrinsic viscosity value ranging from 1.5 to 4 dl/g; and
c) from 5% to 30%, of ethylene homopolymer; the sum a) + b) + c) being 100;

said composition having a value of melt flow rate ranging from 20 to 150 g/10 min.

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Description

[0001] The present invention relates to fibres comprising a polyolefin composition.

[0002] Within the definition of fibres are also included the manufactured products similar to fibres, such as fibrils and cut filaments (staple fibres).

[0003] The fibres of the present invention are particularly fit for use in applications where a good balance of tenacity and elongation at break is required. In particular, the fibres of the present invention are suitable for the manufacture of spunbonded non-woven fabrics.

[0004] According to the teaching of the prior art, it is possible to obtain polyolefin compositions endowed with an exceptional combination of excellent properties by properly adding rubbers and polyethylene to the polypropylene.

[0005] WO 2006/125720 relates to a polypropylene composition comprising (per cent by weight):

a) 65-77% of a crystalline propylene polymer having an amount of isotactic pentads (mmmm), measured by ¹³C-MNR on the fraction insoluble in xylene at 25° C, higher than 97.5 molar % and a polydispersity index ranging from 5 to 10;

b) 8 to less than 13% of an elastomeric copolymer of ethylene and propylene, the copolymer having an amount of recurring units deriving from ethylene ranging from 30 to 70%, and being partially soluble in xylene at ambient temperature; the polymer fraction soluble in xylene at ambient temperature having an intrinsic viscosity value ranging from 2 to 4 dl/g; and

c) 10-23% of polyethylene having an intrinsic viscosity value ranging from 1.5 to 4 dl/g and optionally containing recurring units derived from propylene in amounts lower than 10%.

[0006] WO 2006/067023 relates to a polypropylene composition comprising (per cent by weight):

a) 50-77% of a crystalline propylene polymer having an amount of isotactic pentads (mmmm), measured by ¹³CNMR on the fraction insoluble in xylene at 25°C, higher than 97.5 molar % and a polydispersity index ranging from 4 to 10;

b) 13-28% of an elastomeric copolymer of ethylene and propylene, the copolymer having an amount of recurring units deriving from ethylene ranging from 30 to 70%, and being partially soluble in xylene at ambient temperature, the polymer fraction soluble in xylene at ambient temperature having an intrinsic viscosity value ranging from 2 to 4 dl/g; and

c) 10-22% of polyethylene having an intrinsic viscosity value ranging from 1 to 3 dl/g and optionally containing recurring units deriving from propylene in amounts up to less than 10%.

[0007] The compositions of WO 2006/125720 and of WO 2006/067023 typically have a value of melt flow rate ranging from 0.50 to 10 g/10 min and from 10 to 30 g/10 min, respectively, which values are well below the melt flow rates of the compositions comprised in the fibres of the present invention.

[0008] Polyolefin fibres are generally known in the prior art.

[0009] EP 1264017 relates to multicomponent fibres and non-woven fabrics and fabric laminates which comprise the multicomponent fibres. In the example 1-B, a continuous filament spunbond fabric is produced from a blend comprising 76% isotactic polypropylene, 20% propylene copolymer, and 4% polyethylene, which values are different from those of the polyolefin composition comprised in the fibres of the invention.

[0010] There is a need in the art to provide fibres which can show a better balance of properties, especially in terms of elongation at break and elastic recovery but maintaining good values of tenacity, such fibres being suitable for spunbonding applications.

[0011] The applicant has now surprisingly found that fibres showing a very good balance of mechanical properties and that are suitable for spunbonding applications can be obtained from a polyolefin composition comprising (per cent by weight):

a) from 50% to 85%, preferably from 60% to 82%, more preferably from 72% to 82%, even more preferably from 74% to 80%, of a propylene polymer having an amount of isotactic pentads (mmmm), measured by ¹³C-MNR on the fraction insoluble in xylene at 25°C, higher than 97.5 molar %;

b) from 5% to 20%, preferably from 8% to 15%, more preferably from 9% to 14%, even more preferably from 10% to 12%, of a copolymer of ethylene and propylene, the copolymer having an amount of recurring units deriving from ethylene ranging from 30 wt% to 50 wt%, preferably from 35 wt% to 45 wt%, more preferably from 39 wt% to 42 wt% and being partially soluble in xylene at 25° C; the polymer fraction soluble in xylene at 25° C having an intrinsic viscosity value ranging from 1.5 to 4 dl/g; and

c) from 5% to 30%, preferably from 8% to 14%, more preferably from 9% to 13%, even more preferably from 10% to 12%, of ethylene homopolymer; the sum a) + b) + c) being 100;

said composition having a value of melt flow rate ranging from 20 to 150 g/10 min, preferably from 40 to 90 g/10 min, preferably from 50 to 85 g/10 min, more preferably from 55 to 70 g/10 min.

[0012] Preferably the composition has a content of component (b) plus component (c) in amounts comprised between 15 wt% and 25 wt%, preferably from 20 wt% and 25 wt%.

[0013] Typically, the composition of the present invention exhibits a flexural modulus value of at least 1200 MPa, preferably at least 1300 MPa, even more preferably the flexural modulus is comprised between 1400 MPa and 1700 MPa.

[0014] The fibres of the invention typically with a titre of 2.3 dtex exhibit a value of tenacity at least equal to or higher than 8 cN/tex, preferably higher than 9 cN/tex, and a value of elongation at break at least equal to or higher than 350%, preferably equal to or higher than 400%. Typically, the fibres according to the present invention have a titre ranging from 1 to 4 dtex, preferably from 1.5 to 2.5 dtex.

[0015] The fibres of the present invention can be efficiently spun at speeds that are typically higher than 2000 m/min, preferably higher than 2500 m/min.

[0016] The fibres of the present invention also exhibit an elastic recovery at 10% of at least 5%, preferably of at least 7%.

[0017] Crystalline propylene polymer (a) is selected from a propylene homopolymer and a copolymer of propylene containing at most 3 wt% of ethylene or a C₄-C₁₀ α -olefin or combination thereof. Particularly preferred is the propylene homopolymer.

[0018] Typically, crystalline propylene polymer (a) has a polydispersity index ranging from 4 to 10, preferably from 5 to 10.

[0019] The melt flow rate of crystalline propylene polymer (a) preferably ranges from 130 to 250 g/10 min, preferably from 140 to 220 g/10 min, more preferably from 150 to 210 g/10 min. The intrinsic viscosity of the soluble fraction in xylene at 25° C of the ethylene-propylene copolymer (b) is comprised between 1.5 and 4 dl/g; preferably between 2 and 3.5 dl/g; more preferably between 2.5 and 3.5 dl/g (calculated on the sum of xylene soluble fraction of a+b). The composition of the present invention is obtained by means of a sequential copolymerization process.

[0020] Therefore, the present invention is further directed to a process for the preparation of the polyolefin compositions as reported above, said process comprising at least three sequential polymerization stages with each subsequent polymerization being conducted in the presence of the polymeric material formed in the immediately preceding polymerization reaction, wherein the polymerization stage of propylene to the crystalline polymer (a) is carried out in at least one stage, then a copolymerization stage of mixtures of ethylene with propylene (and optionally a diene) to elastomeric polymer (b) and finally a polymerization stage of ethylene to polyethylene (c) are carried out. The polymerisation stages may be carried out in the presence of a stereospecific Ziegler-Natta catalyst.

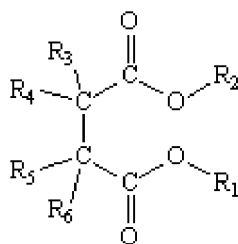
[0021] According to a preferred embodiment, all the polymerisation stages are carried out in the presence of a catalyst comprising a trialkylaluminium compound, optionally an electron donor, and a solid catalyst component comprising a halide or halogen-alcoholate of Ti and an electron-donor compound supported on anhydrous magnesium chloride. Catalysts having the above-mentioned characteristics are well known in the patent literature; particularly advantageous are the catalysts described in USP 4,399,054 and EP-A-45 977. Other examples can be found in USP 4,472,524.

[0022] Preferably the polymerisation catalyst is a Ziegler-Natta catalyst comprising a solid catalyst component comprising:

- a) Mg, Ti and halogen and an electron donor (internal donor),
- b) an alkylaluminum compound and, optionally (but preferably),
- c) one or more electron-donor compounds (external donor).

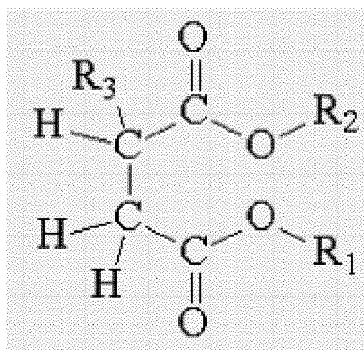
[0023] The internal donor is preferably selected from the esters of mono or dicarboxylic organic acids such as benzoates, malonates, phthalates and certain succinates. They are described in US patent 4522930, European patent 45977 and international patent applications WO 00/63261 and WO 01/57099, for example. Particularly suited are the phthalic acid esters and succinate acids esters. Alkylphthalates are preferred, such as diisobutyl, dioctyl and diphenyl phthalate and benzyl-butyl phthalate.

[0024] Among succinates, they are preferably selected from succinates of the formula (I):



(I)

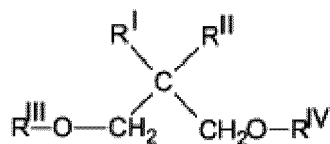
wherein the radicals R_1 and R_2 , equal to or different from each other, are a C_1 - C_{20} linear or branched alkyl, alkenyl, cycloalkyl, aryl, arylalkyl or alkylaryl group, optionally containing heteroatoms; the radicals R_3 to R_6 , equal to or different from each other, are hydrogen or a C_1 - C_{20} linear or branched alkyl, alkenyl, cycloalkyl, aryl, arylalkyl or alkylaryl group, optionally containing heteroatoms, and the radicals R_3 to R_6 which are joined to the same carbon atom can be linked together to form a cycle; with the proviso that when R_3 to R_5 are contemporaneously hydrogen, R_6 is a radical selected from primary branched, secondary or tertiary alkyl groups, cycloalkyl, aryl, arylalkyl or alkylaryl groups having from 3 to 20 carbon atoms; or of formula (II):



(II)

wherein the radicals R_1 and R_2 , equal to or different from each other, are a C_1 - C_{20} linear or branched alkyl, alkenyl, cycloalkyl, aryl, arylalkyl or alkylaryl group, optionally containing heteroatoms and the radical R_3 is a linear alkyl group having at least four carbon atoms optionally containing heteroatoms.

[0025] The Al-alkyl compounds used as co-catalysts comprise Al-trialkyls, such as Al-triethyl, Al-triisobutyl, Al-tri-n-butyl, and linear or cyclic Al-alkyl compounds containing two or more Al atoms bonded to each other by way of O or N atoms, or SO_4 or SO_3 groups. The Al-alkyl compound is generally used in such a quantity that the Al/Ti ratio be from 1 to 1000. External donor (c) can be of the same type or it can be different from the succinates of formula (I) or (II). Suitable external electron-donor compounds include silicon compounds, ethers, esters such as phthalates, benzoates, succinates also having a different structure from those of formula (I) or (II), amines, heterocyclic compounds and particularly 2,2,6,6-tetramethylpiperidine, ketones and the 1,3-diethers of the general formula (III):



(III)

wherein R^{I} and R^{II} are the same or different and are C_1 - C_{18} alkyl, C_3 - C_{18} cycloalkyl or C_7 - C_{18} aryl radicals; R^{III} and R^{IV} are the same or different and are C_1 - C_4 alkyl radicals; or the 1,3-diethers in which the carbon atom in position 2 belongs

to a cyclic or polycyclic structure made up of 5, 6 or 7 carbon atoms and containing two or three unsaturations.

[0026] Ethers of this type are described in published European patent applications 361493 and 728769.

[0027] Preferred electron-donor compounds that can be used as external donors include aromatic silicon compounds containing at least one Si-OR bond, where R is a hydrocarbon radical. A particularly preferred class of external donor compounds is that of silicon compounds of formula $R_a^7R_b^8Si(OR^9)_c$, where a and b are integer from 0 to 2, c is an integer from 1 to 3 and the sum (a+b+c) is 4; R^7 , R^8 , and R^9 , are C_1 - C_{18} hydrocarbon groups optionally containing heteroatoms. Particularly preferred are the silicon compounds in which a is 1, b is 1, c is 2, at least one of R^7 and R^8 is selected from branched alkyl, alkenyl, alkylene, cycloalkyl or aryl groups with 3-10 carbon atoms optionally containing heteroatoms and R^9 is a C_1 - C_{10} alkyl group, in particular methyl. Examples of such preferred silicon compounds are cyclohexyltrimethoxysilane, t-butyltrimethoxysilane, t-hexyltrimethoxysilane, cyclohexylmethyldimethoxysilane, 3,3,3-trifluoropropyl-2-ethylpiperidyl-dimethoxysilane, diphenyldimethoxysilane, methyl-t-butyl dimethoxysilane, dicyclopentyl dimethoxysilane, 2-ethylpiperidinyl-2-t-butyl dimethoxysilane, (1,1,1-trifluoro-2-propyl)-methyldimethoxysilane and (1,1,1-trifluoro-2-propyl)-2-ethylpiperidinyl dimethoxysilane. Moreover, are also preferred the silicon compounds in which a is 0, c is 3, R^8 is a branched alkyl or cycloalkyl group, optionally containing heteroatoms, and R^9 is methyl. Particularly preferred specific examples of silicon compounds are (tert-butyl) $_2Si(OCH_3)_2$, (cyclohexyl)(methyl) $Si(OCH_3)_2$, (phenyl) $_2Si(OCH_3)_2$ and (cyclopentyl) $_2Si(OCH_3)_2$.

[0028] Preferably electron donor compound (c) is used in such an amount to give a molar ratio between the organoaluminum compound and said electron donor compound (c) of from 0.1 to 500, more preferably from 1 to 300 and in particular from 3 to 100.

[0029] As explained above, the solid catalyst component comprises, in addition to the above electron donors, Ti, Mg and halogen. In particular, the catalyst component comprises a titanium compound, having at least a Ti-halogen bond, and the above mentioned electron donor compounds supported on a Mg halide. The magnesium halide is preferably $MgCl_2$ in active form, which is widely known from the patent literature as a support for Ziegler-Natta catalysts. Patents USP 4,298,718 and USP 4,495,338 were the first to describe the use of these compounds in Ziegler-Natta catalysis. It is known from these patents that the magnesium dihalides in active form used as support or co-support in components of catalysts for the polymerisation of olefins are characterized by X-ray spectra in which the most intense diffraction line that appears in the spectrum of the non-active halide is diminished in intensity and is replaced by a halo whose maximum intensity is displaced towards lower angles relative to that of the more intense line.

[0030] The preferred titanium compounds are $TiCl_4$ and $TiCl_3$; furthermore, also Ti-haloalcoholates of formula $Ti(OR)_n-yXy$ can be used, where n is the valence of titanium, y is a number between 1 and n, X is halogen and R is a hydrocarbon radical having from 1 to 10 carbon atoms.

[0031] The preparation of the solid catalyst component can be carried out according to several methods, well known and described in the art.

[0032] According to a preferred method, the solid catalyst component can be prepared by reacting a titanium compound of formula $Ti(OR)_n-yXy$, where n is the valence of titanium and y is a number between 1 and n, preferably $TiCl_4$, with a magnesium chloride deriving from an adduct of formula $MgCl_2 \cdot pROH$, where p is a number between 0.1 and 6, preferably from 2 to 3.5, and R is a hydrocarbon radical having 1-18 carbon atoms. The adduct can be suitably prepared in spherical form by mixing alcohol and magnesium chloride in the presence of an inert hydrocarbon immiscible with the adduct, operating under stirring conditions at the melting temperature of the adduct (100-130° C). Then, the emulsion is quickly quenched, thereby causing the solidification of the adduct in form of spherical particles.

[0033] Examples of spherical adducts prepared according to this procedure are described in USP 4,399,054 and USP 4,469,648. The so obtained adduct can be directly reacted with the Ti compound or it can be previously subjected to thermally controlled dealcoholation (80-130° C) so as to obtain an adduct in which the number of moles of alcohol is generally lower than 3, preferably between 0.1 and 2.5. The reaction with the Ti compound can be carried out by suspending the adduct (dealcoholated or as such) in cold $TiCl_4$ (generally 0° C); the mixture is heated up to 80-130° C and kept at this temperature for 0.5-2 hours. The treatment with $TiCl_4$ can be carried out one or more times. The electron donor compound(s) can be added during the treatment with $TiCl_4$.

[0034] Regardless of the preparation method used, the final amount of the electron donor compound(s) is preferably such that the molar ratio with respect to the $MgCl_2$ is from 0.01 to 1, more preferably from 0.05 to 0.5.

[0035] The said catalyst components and catalysts are described in WO 00/63261 and WO 01/57099. The catalysts may be precontacted with small quantities of olefin (prepolymerisation), maintaining the catalyst in suspension in a hydrocarbon solvent, and polymerising at temperatures from 25° C to 60° C, thus producing a quantity of polymer from 0.5 to 3 times the weight of the catalyst. The operation can also take place in liquid monomer, producing, in this case, a quantity of polymer 1000 times the weight of the catalyst.

[0036] By using the above mentioned catalysts, the polyolefin compositions are obtained in spheroidal particle form, the particles having an average diameter from about 250 to 7,000 microns, a flowability of less than 30 seconds and a bulk density (compacted) greater than 0.4 g/ml.

[0037] The polymerisation stages may occur in liquid phase, in gas phase or liquid-gas phase. Preferably, the polym-

erisation of crystalline polymer (a) is carried out in liquid monomer (e.g. using liquid propylene as diluent), while the copolymerisation stages of elastomeric copolymer (b) and polyethylene (c) are carried out in gas phase. Alternatively, all the three sequential polymerisation stages can be carried out in gas phase.

[0038] The reaction temperature in the polymerisation stage for the preparation of crystalline polymer (a) and in the preparation of elastomeric copolymer (b) and polyethylene (c) be the same or different, and is preferably from 40 to 100° C; more preferably, the reaction temperature ranges from 50 to 80° C in the preparation of polymer (a), and from 70 to 100° C for the preparation of polymer components (b) and (c).

[0039] The pressure of the polymerisation stage to prepare polymer (a), if carried out in liquid monomer, is the one which competes with the vapor pressure of the liquid propylene at the operating temperature used, and it may be modified by the vapor pressure of the small quantity of inert diluent used to feed the catalyst mixture, by the overpressure of optional monomers and by the hydrogen used as molecular weight regulator.

[0040] The polymerisation pressure preferably ranges from 33 to 43 bar, if done in liquid phase, and from 5 to 30 bar if done in gas phase. The residence times relative to the two stages depend on the desired ratio between polymers (a) and (b) and (c), and can usually range from 15 minutes to 8 hours. Conventional molecular weight regulators known in the art, such as chain transfer agents (e.g. hydrogen or ZnEt_2), may be used.

[0041] Conventional additives, fillers and pigments, commonly used in olefin polymers, may be added, such as nucleating agents, extension oils, mineral fillers, and other organic and inorganic pigments. In particular, the addition of inorganic fillers, such as talc, calcium carbonate and mineral fillers, also brings about an improvement to some mechanical properties, such as flexural modulus and HDT. Talc can also have a nucleating effect.

[0042] The nucleating agents are added to the compositions of the present invention in quantities ranging from 0.05 to 2% by weight, more preferably from 0.1 to 1% by weight, with respect to the total weight, for example.

[0043] The fibres of the invention are suitable for the manufacture of spunbonded non-woven fabrics. The particulars are given in the following examples, which are given to illustrate, without limiting, the present invention.

Examples

[0044] The following analytical methods have been used to determine the properties reported in the detailed description and in the examples. - Ethylene (C2): By IR spectroscopy.

[0045] The spectrum of a pressed film of the polymer is recorded in absorbance vs. wavenumbers (cm^{-1}). The following measurements are used to calculate C2 content:

a) **Area (A_1)** of the combination absorption bands between 4482 and 3950 cm^{-1} which is used for spectrometric normalization of film thickness.

b) **Area (A_{C2})** of the absorption band due to methylenic sequences (CH_2 rocking vibration) after a proper digital subtraction of an isotactic polypropylene (IPP) reference spectrum. The range 660 to 790 cm^{-1} is used for both heterophasic and/or random copolymers

- Fractions soluble and insoluble in xylene at 25 °C: 2.5 g of polymer are dissolved in 250 mL of xylene at 135° C under agitation. After 20 minutes the solution is allowed to cool to 25° C, still under agitation, and then allowed to settle for 30 minutes. The precipitate is filtered with filter paper, the solution evaporated in nitrogen flow, and the residue dried under vacuum at 80° C until constant weight is reached. Thus one calculates the percent by weight of polymer soluble and insoluble at 25° C.

- Intrinsic Viscosity $[\eta]$: Measured in tetrahydronaphthalene at 135° C.

- Molecular weight ($\overline{M}_n$, $\overline{M}_w$): Measured by way of gel permeation chromatography (GPC) in 1,2,4-trichlorobenzene.

- Determination of isotactic pentads content: 50 mg of each xylene insoluble fraction were dissolved in 0.5 mL of $\text{C}_2\text{D}_2\text{Cl}_4$

[0046] The ^{13}C NMR spectra were acquired on a Bruker DPX-400 (100.61 Mhz, 90° pulse, 12s delay between pulses). About 3000 transients were stored for each spectrum; mmmm pentad peak (21.8 ppm) was used as reference.

[0047] The microstructure analysis was carried out as described in literature (Polymer, 1984, 25, 1640, by Inoue Y. et Al. and Polymer, 1994, 35, 339, by Chujo R. et Al.).

- Polydispersity index: Measurement of molecular weight distribution of the polymer. To determine the PI value, the modulus separation at loss modulus value, e.g. 500 Pa, is determined at a temperature of 200° C by using a RMS-800 parallel plates rheometer model marketed by Rheometrics (USA), operating at an oscillation frequency which increases from 0.01 rad/second to 100 rad/second. From the modulus separation value, the PI can be derived using the following equation:

$$PI = 54.6 \times (\text{modulus separation})^{-1.76}$$

wherein the modulus separation (MS) is defined as:

$$MS = (\text{frequency at } G' = 500 \text{ Pa}) / (\text{frequency at } G'' = 500 \text{ Pa})$$

wherein G' is the storage modulus and G'' is the loss modulus.

- Melting temperature: Determined by differential scanning calorimetry (DSC). A sample weighting 6 ± 1 mg, is heated to $220 \pm 1^\circ \text{C}$ at a rate of 20°C/min and kept at $220 \pm 1^\circ \text{C}$ for 2 minutes in nitrogen stream and it is thereafter cooled at a rate of 20°C/min to $40 \pm 2^\circ \text{C}$, thereby kept at this temperature for 2 min to crystallise the sample. Then, the sample is again fused at a temperature rise rate of 20°C/min up to $220^\circ \text{C} \pm 1$. The melting scan is recorded, a thermogram is obtained, and, from this, temperatures corresponding to peaks are read.
- Titre of filaments
From a 10 cm long roving, 50 fibers are randomly chosen and weighed. The total weight of the 50 fibers, expressed in mg, is multiplied by 2, thereby obtaining the titre in dtex.
- Tenacity and Elongation at break of filaments
From a 500 m roving a 100 mm-long segment is cut and single fibers randomly chosen. Each single fiber is fixed to the clamps of a Dynamometer and tensioned to break with a traction speed of 20 mm/min for elongations lower than 100% and 50 mm/min for elongations greater than 100%, the initial distance between the clamps being of 20 mm. The ultimate strength (load at break) and the elongation at break are determined in machine (MD) direction.

[0048] The tenacity is calculated by way of the following equation:

$$\text{Tenacity} = \text{Ultimate strength (cN)} \times 10 / \text{Titre (dtex)}.$$

- Melt flow rate: Determined according to ISO method 1133 (230°C and 2.16 kg).
- Flexural modulus: Determined according to ISO method 178.
- Elastic recovery 10%: measured with a dynamometer at 23°C . The spun fibre samples were prepared according to extrusion/spinning conditions specified in the examples. Single fibres were randomly chosen as above said from a 500 m roving.

[0049] Two deformations cycles were applied to the samples under the following conditions: First cycle:

- Initial Cross head distance 200 mm,
- cross head speed 3.3 mm/s,
- maximum cross head distance at maximum deformation 220 mm
- relaxation time at maximum cross head distance 60 seconds
- reverse cross head speed 3.3 mm/second
- cross head time at the end of the deformation cycle (cross head distance 200 mm) 180 seconds. Second cycle:
- Initial Cross head distance 200 mm,
- cross head speed 3.3 mm/s,

[0050] Percent of residual deformation: it is the percentage value of the deformation recorded when the load cell starts to record a tensional force in the second cycle corresponding to the formula Percent of residual deformation = $100 \times (\text{current crosshead distance} - \text{initial crosshead distance}) / (\text{initial crosshead distance})$.

- End of test

[0051] Elastic recovery (%) is the percentage value of the maximum deformation (220 mm, corresponding to 10% deformation with respect to the initial cross head distance of 200 mm) minus the percent of residual deformation as above defined.

Examples 1-2

[0052] In a plant operating continuously according to the mixed liquid-gas polymerization technique, runs were carried out under the conditions specified in Table 1.

[0053] The polymerization was carried out in the presence of a catalyst system in a series of three reactors equipped with devices to transfer the product from one reactor to the one immediately next to it.

Preparation of the solid catalyst component

[0054] Into a 500 ml four-necked round flask, purged with nitrogen, 250 ml of TiCl_4 are introduced at 0°C . While stirring, 10.0 g of microspheroidal $\text{MgCl}_2 \cdot 1.9\text{C}_2\text{H}_5\text{OH}$ (prepared according to the method described in ex.2 of USP 4,399,054 but operating at 3000 rpm instead of 10000 rpm) and 9.1 mmol of diethyl 2,3-(diisopropyl)succinate are added. The temperature is raised to 100°C and maintained for 120 min. Then, the stirring is discontinued, the solid product was allowed to settle and the supernatant liquid is siphoned off. Then 250 ml of fresh TiCl_4 are added. The mixture is reacted at 120°C for 60 min and, then, the supernatant liquid is siphoned off. The solid is washed six times with anhydrous hexane ($6 \times 100 \text{ ml}$) at 60°C .

Catalyst system and prepolymerization treatment

[0055] The solid catalyst component described above was contacted at 12°C for 24 minutes with aluminium triethyl (TEAL) and dicyclopentylmethoxysilane (DCPMS) as outside-electron-donor component. The weight ratio between TEAL and the solid catalyst component and the weight ratio between TEAL and DCPMS are specified in Table 1.

[0056] The catalyst system is then subjected to prepolymerization by maintaining it in suspension in liquid propylene at 20°C for about 5 minutes before introducing it into the first polymerization reactor.

Polymerization

[0057] The polymerisation run is conducted in continuous in a series of three reactors equipped with devices to transfer the product from one reactor to the one immediately next to it. The first reactor is a liquid phase reactor, and the second and third reactors are fluid bed gas phase reactors. Polymer (a) is prepared in the first reactor, while polymers (b) and (c) are prepared in the second and third reactor, respectively.

[0058] Temperature and pressure are maintained constant throughout the course of the reaction. Hydrogen is used as molecular weight regulator.

[0059] The gas phase (propylene, ethylene and hydrogen) is continuously analysed via gaschromatography.

[0060] At the end of the run the powder is discharged and dried under a nitrogen flow. The polymerization conditions are reported in table 2.

[0061] The propylene compositions were added with the following additives: calcium stearate 500 ppm, Irganox B215 1500 ppm.

[0062] Then the polymer particles are introduced in a twin screw extruder Leistritz (L/D=27).

[0063] Temperature of the feeding section: $180\text{-}210^\circ \text{C}$

[0064] Melt temperature: 210°C

[0065] Temperature of the die section: 200°C

[0066] Flow rate: 15 Kg/h

[0067] Rotational speed: 250 rpm

[0068] The features of the polymer have been reported on Table 3.

Preparation of the fibres

[0069] After extrusion the polyolefin compositions are spun in a Leonard 25 spinning pilot line with screw L/D ratio of 25, screw diameter of 25 mm and compression ratio of 1:3. The line is marketed by Costruzioni Meccaniche Leonard-Sumirago (VA). The operative spinning conditions and properties of the filaments are reported in Table 4.

Table 1- preparation of fibres

		Ex 1	Ex 2
<i>Operative conditions</i>			
Hole diameter	mm	0,6	

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(continued)

		Ex 1	Ex 2
<i>Operative conditions</i>			
Output per hole	g/min	0.6	
Hole number in the die	/	37	
Die temperature	°C	250	
Melt temperature	°C	258	

[0070] The maximum spinning speed gives indication of the spinnability of the polyolefin composition of the invention. The value corresponds to the highest spinning rate that can be maintained for 30 minutes with no filament break.

Table 2 - Polymerization

Example		1	2
TEAL/solid catalyst component weight ratio		14	11
TEAL/DCPMS weight ratio		5	5
Liquid phase reactor			
Polymerisation temperature	°C	70	70
Pressure	Barg	39	39
Residence time	min	70	70
H ₂ bulk	Mol ppm	7000	6950
1st gas phase reactor			
Polymerisation temperature	°C	80	80
Pressure	Barg	14	14
Residence time	min	13	15
C ₂ /(C ₂ +C ₃)	Mol ratio	0.26	0.28
H ₂ /C ₂	Mol ratio	0.049	0.051
2nd gas phase reactor			
Polymerisation temperature	°C	100	100
Pressure	Barg	13	13
Residence time	min	13	14
C ₂ /(C ₂ +C ₃)	Mol ratio	0.97	0.97
H ₂ /C ₂	Mol ratio	0.15	0.15
H ₂ bulk = hydrogen concentration in the liquid monomer; C ₂ = ethylene ; C ₃ = propylene			

Table 3 - Composition Analysis

Example		1	2
Component a)			
Crystalline propylene homopolymer			
Homopolymer content	%wt	77	70
MFR	g/10 min	170	150

(continued)

Example		1	2
Component a)			
Crystalline propylene homopolymer			
Xylene soluble fraction	%wt	2.5	2.5
Pentad content of the xylene insoluble fraction	Molar%	> 97.5	> 97.5
Component b)			
Propylene-ethylene copolymer			
Copolymer content	%wt	11	15
Ethylene content	%wt	40	38
Intrinsic viscosity xylene soluble fraction*	dl/g	3.2	3.1
Component c)			
Polyethylene			
Polyethylene content	%wt	12	15
Ethylene content (by calculation)	%wt	100	100
Properties of the composition			
Xylene-soluble fraction	dl/g	11.9	15
MFR	g/10 min	57	45
Flexural Modulus	MPa	1521	1280
Melting Temperature	°C	161.1	160.6
* calculated on the xylene soluble fraction of a) + b)			

Table 4 - Properties of Fibres

Examples	1	2
Titre, dtex	2.3	2.3
Tenacity, cN/tex	9.6	9.6
Elongation at Break, %	410	345
Elastic recovery 10%, %	8	1.1

[0071] The data in Table 4 show that the fibres according to the present invention exhibit an enhanced balance of mechanical properties.

Claims

1. A fibre comprising a polyolefin composition wherein said composition comprises (per cent by weight):

- a) from 50% to 85%, of a propylene polymer having an amount of isotactic pentads (mmmm), measured by ¹³C-MNR on the fraction insoluble in xylene at 25° C, higher than 97.5 molar %;
- b) from 5% to 20%, of a copolymer of ethylene and propylene, the copolymer having an amount of recurring units deriving from ethylene ranging from 30 to 50%, and being partially soluble in xylene at 25° C; the polymer fraction soluble in xylene at 25° C having an intrinsic viscosity value ranging from 1.5 to 4 dl/g; and
- c) from 5% to 30%, of ethylene homopolymer; the sum a) + b) + c) being 100; said composition having a value of melt flow rate ranging from 20 to 150 g/10 min.

2. The fibre according to claim 1 wherein the polyolefin composition comprises (per cent by weight):

- a) from 70 % to 84% of a crystalline propylene polymer having an amount of isotactic pentads (mmmm), measured by ¹³C-MNR on the fraction insoluble in xylene at 25° C, higher than 97.5 molar %;
- b) from 8% to 15% of a copolymer of ethylene and propylene, the copolymer having an amount of recurring units deriving from ethylene ranging from 30 to 50%, and being partially soluble in xylene at 25° C; the polymer fraction soluble in xylene at 25° C having an intrinsic viscosity value ranging from 1.5 to 4 dl/g; and
- c) from 8% to 14% of ethylene homopolymer,

3. The fibre according to claims 1 or 2 wherein the polyolefin composition comprises from 72wt% to 82wt% of the propylene (a), from 9 wt% to 14 wt% of the copolymer (b) and from 9 wt% to 13 wt% of the polyethylene (c).

4. The fibre according to anyone of claims 1-3 wherein the composition has a content of component (b) plus component (c) in amounts comprised between 20 wt% and 25 wt%.

5. The fibre according to any of claims 1-4 wherein the composition has a value of melt flow rate ranging from 40 to 90 g/10 min.

6. A spunbonded non-woven fabric comprising the fibres of any of claims 1-5.



EUROPEAN SEARCH REPORT

Application Number
EP 11 19 1869

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