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(54) **Polyolefin fibres loaded with polar, rigid and incompatible polymers**

(57) The present invention discloses fibres and filaments prepared from a composition of polypropylene and a polar, rigid and incompatible polymer selected from ar-

omatic or aliphatic polyester. It also discloses nonwoven and laminates prepared from these fibres and filaments.

**EP 2 096 198 A1**

**Description**

**[0001]** The present invention relates to a process for the production of polyolefin fibres with reduced smoothness and improved hydrophilicity. The present invention also relates to the fibres and nonwoven made with said process. Additionally it relates to composites and laminates comprising such fibres and nonwoven.

**[0002]** Polypropylene is one of the most widely used polymers in fibres and nonwoven. Due to its versatility and its good mechanical and chemical properties, polypropylene is well suited to fulfill requirements in many different applications. Polypropylene fibres and nonwoven are for example used in the construction and agricultural industries, sanitary and medical articles, carpets, and textiles.

**[0003]** The polypropylenes used for fibres and nonwoven have a melt flow that can range from 5 dg/min up to several thousands dg/min, depending upon the production method, final use etc.. For very strong high-tenacity fibers, the lower part of the range is preferred whereas for meltblown nonwoven, the higher part of the range is preferred. Typically, polypropylene used in fibre extrusion has a melt flow in the range of from 5 dg/min to about 40 dg/min. Polypropylene used for spunbond nonwoven typically has a melt flow index in the range of from 25 dg/min to 40 dg/min and is additionally characterised by a narrow molecular weight distribution (Polypropylene Handbook, ed. Nello Pasquini, 2nd edition, Hanser, 2005, p. 397).

**[0004]** Polypropylene is generally produced by the polymerisation of propylene and one or more optional comonomers in presence of a Ziegler-Natta catalyst, i.e. a transition metal coordination catalyst, specifically a titanium halide containing catalyst. These catalysts in general also contain internal electron donors, such as phthalates, diethers, or succinates. Polypropylene produced by Ziegler-Natta catalysis can be directly used without modification for the production of fibres. However, in order to improve the processability and the nonwoven properties in spunbond nonwoven the molecular weight distribution of the polypropylene needs to be narrowed, which can be done either thermally or chemically by post-reactor degradation.

**[0005]** Research Disclosure RD 36347, for example, discloses the use of a polypropylene degraded from a starting melt flow of 1 dg/min to a final melt flow of 20 dg/min in the production of a spunbond nonwoven. The degraded polypropylene has a molecular weight distribution in the range from 2.1 to 2.6.

**[0006]** Whilst not wishing to be bound by theory it is believed that under the processing conditions used in the production of a spunbond nonwoven, the narrowing of the molecular weight distribution also reduces the melt elasticity, which in turn results in a reduction of die swell and in reduced resistance to fibre drawing. Thus, the stability of the spinning process as well as the maximum spinning speed are increased. Additionally, a polypropylene of narrow molecular weight distribution is more likely to retain orientation and good mechanical properties of the nonwoven.

**[0007]** There remains however a constant demand for further improvement such as for example improved hydrophilicity while keeping good processing and good mechanical properties.

**[0008]** It is an aim of the present invention to prepare polypropylene fibres having improved hydrophilicity.

**[0009]** It is also an aim of the present invention to prepare polypropylene fibres having reduced smoothness.

**[0010]** It is another aim of the present invention to prepare polypropylene fibres having improved resilience.

**[0011]** It is yet another aim of the present invention to prepare polypropylene fibres comprising evenly dispersed non-migrating melt additives or migrating melt additives with controlled release.

**[0012]** It is a further aim of the present invention to use these fibres to prepare nonwoven material.

**[0013]** Any one of these aims is at least partially fulfilled by the present invention.

**[0014]** Accordingly, the present invention discloses fibres and filaments prepared from a composition comprising from 90 to 99.5 wt%, based on the total weight of the composition, of polypropylene and from 0.5 to 10 wt% of a polar, rigid and incompatible polymer selected from aromatic or aliphatic polyester.

**[0015]** The preferred polar, rigid and incompatible polymers according to the present invention are selected for example from polybutylene terephthalate (PBT), polyethylene terephthalate (PET) polytrimethylene terephthalate (PTT), or mixtures thereof. The most preferred incompatible polymer is PBT.

**[0016]** Preferably, the amount of aromatic or aliphatic polyester present in the blend is of from 0.5 to 5 wt%, more preferably, from 0.5 to 2 wt%.

**[0017]** The polypropylene used in the present invention is either a homopolymer or a random copolymer of propylene with one or more comonomers, said comonomer being ethylene or a C<sub>4</sub> - C<sub>10</sub> alpha-olefin, such a butene-1, pentene-1, hexene-1, octene-1, 4-methyl-pentene-1. The preferred comonomers are ethylene and butene-1. The most preferred comonomer is ethylene. The random copolymer of the present invention comprises at least 0.1 % by weight, more preferably at least 0.2 % by weight and most preferably at least 0.5 % by weight of comonomer. It comprises at most 6 % by weight, preferably at most 5 % by weight and most preferably at most 3 % by weight of comonomer.

**[0018]** The polypropylene used in the present invention is produced by a Ziegler-Natta or by a metallocene-based catalytic system. Such catalytic systems are commercially available and thus known to the person skilled in the art. Preferably it is a Ziegler-Natta catalyst system. A Ziegler-Natta catalyst system comprises a titanium compound having at least one titanium-halogen bond and an internal electron donor, both on a suitable support, such as for example on

a magnesium halide in active form. It further comprises an organoaluminium compound, such as for example an aluminium trialkyl, and an optional external donor, such as for example a silane or a diether compound.

**[0019]** The homo- or co-polymerisation of propylene with one or more optional comonomers can be carried out according to known techniques in one or more polymerisation reactors, for example in a slurry, bulk or gas phase process. In a slurry process the polymerisation is carried out in a diluent, such as an inert hydrocarbon. In a bulk process the polymerisation is carried out in liquid propylene as reactor medium.

**[0020]** The molecular weight of the polymer chains, and in consequence the melt flow of the polypropylene, is regulated by the addition of hydrogen to the polymerisation medium.

**[0021]** The polypropylene of the present invention is characterised by a melt flow index in the range from 1 to 2000 dg/min, as measured according to ISO 1133, condition L, at a temperature of 230°C under a load of 2.16 kg. When used for fibre spinning the melt flow of the polypropylene is in the range from 5 dg/min to 40 dg/min. When used in the spunbonding process the melt flow of the polypropylene is of at least 10 dg/min, preferably at least 12, 14, 16, 18 or 20 dg/min. When used in the spunbonding process the melt flow of the polypropylene is at most 300 dg/min, preferably at most 200 dg/min, more preferably at most 150 dg/min, even more preferably at most 100 dg/min and most preferably at most 60 dg/min. When used in the melt blown process the melt flow of the metallocene polypropylene is of at least 100 dg/min, preferably at least 150 dg/min, more preferably at least 200 dg/min, even more preferably at least 250 dg/min and most preferably at least 300 dg/min. When used in the melt blown process the melt flow of the polypropylene is of at most 2000 dg/min, preferably at most 1800 dg/min, more preferably at most 1600 dg/min, and most preferably at most 1400 dg/min.

**[0022]** The polypropylene fibres and filaments of the present invention are produced as-spun by methods well known to the skilled person. Polypropylene is melted in an extruder, preferably passed through a melt pump to ensure a constant feeding rate and then extruded through a number of fine capillaries of a spinneret. The still molten fibres and filaments are simultaneously cooled by air, drawn to a final diameter and collected. They are for example collected on a winder or other suitable collecting means. An optional drawing step may be conducted with the so-obtained solidified fibres and filaments.

**[0023]** The nonwovens of the present invention may be produced by any suitable method. The preferred methods are the spunbonding process and the melt blown process. Of these the spunbonding process is the most preferred. In the spunbonding process as well as in the melt blown process the extruded fibres and filaments are drawn in the molten state only. For the purpose of the present invention the fibres and filaments comprised in a spunbond nonwoven or a melt blown nonwoven are therefore considered to be as-spun fibres and filaments.

**[0024]** In the spunbonding process, the polypropylene composition is melted in an extruder, preferably first passed through a melt pump to ensure a constant feeding rate and then extruded from a number of fine, usually circular, capillaries of a spinneret, thus obtaining filaments. The filament formation can either be done by using one single spinneret with a large number of holes, typically several thousands, or by using several small spinnerets with a much lower number of holes per spinneret. After exiting from the spinneret, the still molten filaments are quenched by a current of air. The diameter of the filaments is then quickly reduced by a flow of high-pressure air. Air velocities in this drawdown step can range up to several thousand metres per minute.

**[0025]** The present invention also provides a process for the production of fibres and filaments, said process comprising the steps of:

- (a) providing a blend comprising from 90 to 99.5 wt%, based on the total weight of the blend, of polypropylene and from 0.5 to 10 wt% of a rigid and incompatible polymer selected from aromatic or aliphatic polyester,
- (b) feeding the blend of step (a) to an extruder,
- (c) subsequently melt-extruding the blend to obtain a molten polymer stream,
- (d) extruding the molten polymer stream of step (c) from a number of fine, usually circular, capillaries of a spinneret, thus obtaining filaments of molten polymer, and
- (e) subsequently rapidly reducing the diameter of the filaments obtained in the previous step to a final diameter.

**[0026]** After drawdown the filaments are collected on a support, for example a forming wire or a porous forming belt, thus first forming an unbonded web, which is then passed through compaction rolls and finally through a bonding step. Bonding of the fabric may be accomplished by thermobonding, hydroentanglement, needle punching, or chemical bonding.

**[0027]** In the melt blown process, the polypropylene composition is melted in an extruder, preferably first passed through a melt pump to ensure a constant feeding rate and then through the capillaries of a special melt blowing die. Usually melt blown dies have a single line of usually circular capillaries through which the molten polymer passes. After exiting from the die, the still molten filaments are first contacted with hot air at high speed, which rapidly draws the fibres. They are then contacted with cool air that solidifies the filaments. The nonwoven is formed by depositing the filaments directly onto a forming wire or a porous forming belt.

**[0028]** The fibres and filaments of the present invention may be multicomponent fibres or filaments. Preferably they are bicomponent fibres or filaments. Bi- or multi-component fibres or filaments are known in many different configurations, such as for example side-by-side, sheath-core, islands-in-the-sea, pie or stripe configurations. Bi- or multi-component fibres or filaments can be formed by co-extrusion of at least two different components into one fibre or filament. This is done by feeding the different components to a corresponding number of extruders and combining the different melts into a single fibre or filament. The resulting fibre or filament has at least two different essentially continuous polymer phases. Such fibres or filaments, their production as well as their forming a nonwoven are well known to the skilled person and are for example described in F. Fourné, Synthetische Fasern, Carl Hanser Verlag, 1995, chapter 5.2 or in B.C. Goswami et al., Textile Yarns, John Wiley & Sons, 1977, p. 371 - 376.

**[0029]** The present invention also discloses a process for the production of multicomponent as-spun fibres and filaments, said process comprising the steps of

- a) providing a first composition comprising from 90 to 99.5 wt%, based on the total weight of the composition, of polypropylene and from 0.5 to 10 wt% of a polar, rigid and incompatible polymer selected from aromatic or aliphatic polyester;
- b) providing at least one further blend comprising a thermoplastic polymer;
- c) feeding the blends of steps a) and b) to separate extruders;
- d) consecutively melt-extruding the blends to obtain a molten polymer stream for each blend;
- e) co-extruding the molten polymer streams of step d) from a number of fine capillaries of a spinneret, thus obtaining multicomponent filaments of molten polymer, and
- f) subsequently rapidly reducing the diameter of the filaments obtained in the previous step to a final diameter.

**[0030]** Composites may be formed from two or more nonwovens, of which at least one is made in accordance with the present invention. In particular, the composites comprise a spunbond nonwoven layer (S) according to the present invention or a melt blown nonwoven layer (M) according to the present invention. Composites in accordance with the present invention can for example be SS, SSS, SMS, SMMSS or any other combination of spunbond and melt blown nonwoven layers. In a preferred embodiment according to the present invention, the polypropylene/(aromatic or aliphatic polyester) blend is used in the sheath of a concentric bicomponent configuration.

**[0031]** A first nonwoven or composite, said first nonwoven or composite being in accordance with the present invention, and a film may be combined to form a laminate. The film preferably is a polyolefin film. The laminate is formed by bringing the first nonwoven or composite and the film together and laminating them to one another for example by passing them through a pair of lamination rolls. The laminates may further include a second nonwoven or composite, which can be, but need not be, according to the present invention, on the face of the film opposite to that of the first nonwoven or composite. In a preferred embodiment, the film of the laminate is a breathable polyolefin film, thus resulting in a laminate with breathable properties.

**[0032]** For the present invention it is essential that the polypropylene comprises a polar, rigid and incompatible polymer selected from aromatic or aliphatic polyester.

**[0033]** The polypropylene of the present invention may also contain additives such as, by way of example, antioxidants, light stabilisers, acid scavengers, lubricants, antistatic additives, and colorants.

**[0034]** The preferred aromatic or aliphatic polyester are selected from polybutylene terephthalate (PBT), polyethylene terephthalate (PET) or polytrimethylene terephthalate (PTT). More preferably, it is polybutylene terephthalate (PBT).

**[0035]** The blend is obtained either by dry-blending or by pre-compounding. Preferably, the polyethylene resin is prepared with a Ziegler-Natta and contains low levels of residual catalytic component. Alternatively, it is an isotactic polypropylene prepared with a metallocene catalyst system. These two alternatives are selected in order to prevent the degradation of polyester and to prevent extruder fouling.

**[0036]** The PP/(aromatic or aliphatic polyester) compound can further optionally be loaded with a polar melt additive having more affinity to the polyester phase than to the polypropylene phase, in order to create a controlled release system.

**[0037]** Because the aromatic or aliphatic polyester is incompatible with polypropylene, the spinning of PP/ (aromatic or aliphatic polyester) compositions achieves a microstructure wherein the polyester organises itself in incompatible micro-domains within the polypropylene matrix. The subsequent drawing of the fibres, either in the melt or in the solid state leads to the elongation of the polyester micro-domains into polyester micro-fibrils. Cavitation may occur at the PP/ (aromatic or aliphatic polyester) interface, leading to the formation of voids and consequently to a reduced fibre density. If the polyester loading is low, i. e. at most 5 wt%, the tenacity and elongation of the fibres are not altered. In addition, the size of the micro-domains can be controlled by playing on the ratio of polymer viscosities or by playing on their partial compatibilisation for example through amphiphilic compounds.

**[0038]** As the polyester micro-domain are homogeneously distributed within the fibres, additional loading with a melt additive having more affinity towards polyester than towards polypropylene results in delayed diffusion of said melt additive towards the fibre surface. Melt additives can be selected for example from glycerol monostearate (GMS) or

polyester wax. Delayed diffusion offers the advantage that it acts as a reservoir and thus provides a time-controlled release of melt additive. It must additionally be noted that some of the polyester micro-domains necessarily lie just underneath the surface and are thus able to generate micro-rugosity. This will result in improved hydrophilicity.

**[0039]** The bending moment of the fibres is also affected by the polyester fibrils, resulting in improved resilience.

### Examples.

**[0040]** A master batch was prepared with 80 wt% of commercial resin PPH 7059<sup>®</sup> sold by Total Petrochemicals and 20 wt% PBT in a twin screw extruder at a temperature of 230 °C.

Resin R1 was prepared from 95 wt% of resin PPH 7059 and 5 wt% of master batch.

Resin R2 was prepared from 94 wt% of resin PPH 7059, 5 wt% of master batch and 1 wt% of Irgasurf HL560<sup>®</sup>.

Resin R3 was prepared from 75 wt% of resin PPH 7059 and 25 wt% of master batch.

**[0041]** Fibres were spun on a Busschaert machine at a temperature of 280 °C, with a hole size of 0.8 mm and at a rate of 0.5 g/hole/min. 3 dtex as-spun fibres were prepared at a constant speed of 1700 m/min.

**[0042]** The mechanical properties of the fibres are given in Table I.

**TABLE I.**

	PPH 7059	R1	R2
Tenacity (cN/tex)	15.2	14.5	14.8
Elongation %	429	413	430

**[0043]** Resins R1 and R3 were spun on a Labline machine at a temperature of 255 °C, with a hole size of 0.5 mm and at a rate of 0.5 g/hole/min. The density decreased and the rugosity increased with increasing amounts of added PBT. With amounts of PBT in the resin of more than 2 wt% the spinnability and the tenacity decreased dramatically. It is thus preferable to prepare bi-component fibres when large amounts of PBT are mixed with polypropylene.

**[0044]** The mechanical properties of the fibres are given in Table II.

**TABLE II.**

	PPH 7059	R1	R3
dtex	2.95	2.91	3.49
Tenacity (cN/tex)	13.64	13.55	12.66
Elongation %	205.36	188.29	238.8
Calc. density g/cc	0.9122	0.9162	0.9322
Obs. density g/cc	0.9122	0.9091	0.9210

**[0045]** It can be seen that the observed density was substantially smaller than the calculated density, indicating the formation of cavities in the fibres.

### Claims

1. Fibres and filaments prepared from a composition comprising from 90 to 99.5 wt%, based on the total weight of the composition, of polypropylene and from 0.5 to 10 wt% of a polar, rigid and incompatible polymer selected from aromatic or aliphatic polyester.
2. The fibres and filaments of claim 1 wherein the composition comprises from 95 to 99.5 wt%, based on the total weight of the composition, of polypropylene and from 0.5 to 5 wt% of a polar, rigid and incompatible polymer selected from aromatic or aliphatic polyester.
3. The fibres and filaments of claim 1 or claim 2 wherein the aromatic or aliphatic polyester is selected from, polyethylene terephthalate or polytrimethylene terephthalate or polybutylene terephthalate or mixtures thereof.
4. The fibres and filaments of claim 3 wherein the aromatic or aliphatic polyester is polybutylene terephthalate.

5. Process for the production of fibres and filaments, said process comprising the steps of:

- a) providing a composition comprising polypropylene and a polar, rigid and incompatible polymer selected from aromatic or aliphatic polyester according to any one of claims 1 to 4;
- b) feeding the blend of step a) to an extruder;
- c) subsequently melt-extruding the blend to obtain a molten polymer stream;
- d) extruding the molten polymer stream of step c) from a number of fine, usually circular, capillaries of a spinneret, thus obtaining filaments of molten polymer; and
- e) subsequently rapidly reducing the diameter of the filaments obtained in the previous step to a final diameter.

6. Process for the production of multicomponent fibres and filaments, said process comprising the steps of:

- a) providing a first composition polypropylene a polar, rigid and incompatible polymer selected from aromatic or aliphatic polyester according to any one of claims 1 to 4;
- b) providing at least one further blend comprising a thermoplastic polymer;
- c) feeding the blends of steps a) and b) to separate extruders;
- d) consecutively melt-extruding the blends to obtain a molten polymer stream for each blend;
- e) co-extruding the molten polymer streams of step d) from a number of fine capillaries of a spinneret, thus obtaining multicomponent filaments of molten polymer, and
- f) subsequently rapidly reducing the diameter of the filaments obtained in the previous step to a final diameter.

7. The process of 5 or claim 6, further comprising the steps of:

- i) collecting the final mono- or multi-component filaments on a support; and
- ii) subsequently bonding the collected filaments to form a bonded nonwoven.

8. Process according to any one of claims 5 to 7, further comprising the step of:

- iii) laminating a film to the bonded nonwoven obtained in step ii).

9. Nonwoven comprising mono- or multi-component fibres and filaments prepared by the process of any one of claims 5 to 8.

10. Nonwoven according to claim 9, wherein the nonwoven is a spunbond nonwoven or a melt blown nonwoven, preferably a spunbond nonwoven.

11. Laminates comprising the nonwoven of claims 9 or claim 10.

12. Laminates according to claim 11, wherein the nonwoven of claims 9 or claim 10 is laminated to a polyolefin film, preferably a polyethylene film.

13. Hygiene and sanitary articles comprising the nonwoven of claim 9 or claim 10 or the laminate of claim 11 or claim 12.



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# EUROPEAN SEARCH REPORT

Application Number  
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The present search report has been drawn up for all claims			
Place of search The Hague		Date of completion of the search 1 August 2008	Examiner Malik, Jan
<p>CATEGORY OF CITED DOCUMENTS</p> <p>X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document</p> <p>T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons &amp; : member of the same patent family, corresponding document</p>			

10  
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# EUROPEAN SEARCH REPORT

Application Number  
EP 08 15 1944

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10

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**ANNEX TO THE EUROPEAN SEARCH REPORT  
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EP 08 15 1944

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