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(54) MATERIAL COMPRISING A NONWOVEN LAYER OF FIBERS

MATERIAL MIT EINER VLIESSCHICHT AUS FASERN

MATÉRIAUX COMPRENANT UNE COUCHE NON TISSÉE DE FIBRES

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(56) References cited:

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Description

[0001] The invention pertains to a material comprising a nonwoven layer of fibers, to a process for providing a material comprising a nonwoven layer of fibers, and to fibers comprised in a material comprising a nonwoven layer of fibers.

[0002] Materials comprising a nonwoven layer of fibers find their way as substrates or carrier materials in many applications. Materials comprising a nonwoven layer of fibers may for example be used as a substrate or carrier material for bitumen membranes for waterproofing in roofing, sealing and sarking applications, wherein the carrier material is impregnated with bitumen to provide a bitumen membrane.

[0003] A carrier material for bitumen membranes may for example be provided as a spunbonded nonwoven manufactured by extruding molten polyester polymer into polyester filaments, collecting the polyester filaments on a moving conveyor belt to form a nonwoven web, and consolidating the nonwoven web into a spunbonded nonwoven material by mechanical needling and chemical bonding.

[0004] Manufacturing of a material for a carrier material for bitumen membranes comprising or consisting of a nonwoven may include the step of melting recycled polyester collected from polyethylene terephthalate (PET) bottles and extruding the molten polyester polymer through orifices in a spinneret to form fibers. The recycling of polyester bottles has been well organized over the last decades, thereby providing a polyester source for nonwoven production which has a relatively constant quality and a low amount of contamination by other polymers, in particular by polyolefin polymers. The polyethylene terephthalate (PET) bottles are washed and grinded into bottle flakes, which can then be fed to an extruder where the bottle flakes are melted and subsequently extruded through orifices in a spinneret.

[0005] Extrusion of molten polyester through orifices in a spinneret to form polyester filaments, in particular in spunbond processes, requires that the contamination of the polyester polymer with other polymers, such as polyolefins, is at a low level, generally well below 2000 ppm, to avoid filament breakage and to prevent that the tenacity of the filaments is too low for the intended application in nonwoven carrier material for bitumen membranes.

[0006] However, there is an increased effort to recycle polyethylene terephthalate (PET) bottles directly into new polyester bottles again, which means that it becomes increasingly difficult to source sufficient amounts of recycled polyethylene terephthalate bottles for spinning polyester filaments in the manufacture of nonwovens. Other sources of recycled polyester, in particular of recycled polyethylene terephthalate, may contain higher levels of contamination with other polymers, including polyolefins, such as polyethylene and/or polypropylene. These higher levels of contamination of the polyethylene terephthalate to be recycled result in increased filament breakage and lower tenacity of the filaments during manufacturing of nonwovens for carrier materials.

[0007] EP 2 455 523 discloses a PET spunbond for use as a reinforcing substrate for bituminous membranes.

[0008] It is an object of the invention to prevent, or at least reduce, the disadvantages of using recycled polyester, in particular of polyethylene terephthalate, containing high levels of contamination with polyolefin polymer(s) for providing of nonwovens for materials to be used as carrier material for bitumen membranes.

[0009] The object of the invention is achieved by the process according to claim 1.

[0010] A process for providing a material comprising a nonwoven layer of fibers comprising the steps of providing a material composition comprising 90 to 98.7 wt.% of a recycled polyester polymer, 1 to 5 wt.% of a polyolefin polymer and 0.3 to 5 wt.% of a compatibilizer based on the total weight of the recycled polyester polymer, the polyolefin polymer and the compatibilizer, melting the material composition, extruding the molten material composition through spinning holes in a spinneret to form extruded fibers, supplying the extruded fibers onto a moving surface to form a nonwoven web of fibers and consolidating the nonwoven web to obtain a material comprising a nonwoven layer of fibers enables to use recycled polyester from sources comprising higher levels of contamination with polyolefins, such as polyethylene and/or polypropylene.

[0011] The process enables to use recycled polyester from sources comprising high levels of contamination with polyolefins, such as polyethylene and/or polypropylene, without the need of chemical recycling of the polyester for removing contaminants. The process preferably excludes a step of chemical recycling of the polyester, wherein the polyester is depolymerized into monomers, wherein the monomers are purified by at least a part of the contaminants, and re-polymerized into polyester.

[0012] The fibers comprised in the nonwoven layer of fibers provided by the process may have an average tenacity of at least 2.4 cN/dtex as determined according to ISO 5079:2020. The fibers comprised in the nonwoven layer of fibers may have an average tenacity of at least 2.5 cN/dtex, or at least 2.7 cN/dtex, or of at least 3.0 cN/dtex, or of at least 3.5 cN/dtex, or of at least 4.0 cN/dtex. The material composition provided in the process enables to obtain extruded fibers having an average tenacity of at least 2.4 cN/dtex without being confronted with a too high level of fiber breakage during extrusion of the molten material composition.

[0013] The fibers comprised in the nonwoven layer of fibers provided by the process may have an elongation at break of at least 50%, preferably of at least 60%, more preferably of at least 70%, as determined according to ISO 5079:2020, using an MTS Exceed E42 dynamometer.

[0014] The process enables to provide fibers in the nonwoven layer of fibers which do not comprise voids within the

fibers, as debonding between the polyester polymer and the polyolefin polymer is prevented by the material composition.

[0015] The process may comprise a step of solidifying the extruded fibers before the fibers are supplied on a moving surface, for example by blowing air of ambient temperature against the extruded fibers.

[0016] The process may comprise a step of drawing the extruded fibers to further improve the properties of the fibers, in particular the tenacity of the fibers. The step of drawing the extruded fibers may comprise drawing by an air flow and/or by drawing by two godets having different circumferential speeds. The material composition provided in the process enables to draw extruded, solidified fibers without being confronted with a too high level of fiber breakage during drawing.

[0017] The process comprises the step of supplying the extruded fibers to form a nonwoven web of fibers, and consolidating the nonwoven web to obtain a material comprising a nonwoven layer of fibers. The fibers may be supplied onto any suitable surface to form a nonwoven web of fibers.

[0018] The fibers may be supplied onto a moving surface, for example a rotating drum or a conveyor belt.

[0019] The fibers may be supplied onto a moving surface which is air-permeable to enable applying a vacuum below the moving surface to fixate the nonwoven web of fibers on the moving surface until the nonwoven web of fibers is being consolidated into a nonwoven layer of fibers. The moving surface may advantageously be an air permeable conveyor belt.

[0020] The process may comprise the step of consolidating the nonwoven web of fibers into a nonwoven layer of fibers by any known suitable consolidating process or any combination of consolidating processes. The step of consolidating the nonwoven web of fibers into a nonwoven layer of fibers may include a mechanical bonding process, such as mechanical needling and/or fluid entanglement, for example hydroentanglement. The step of consolidating the nonwoven web of fibers into a nonwoven layer of fibers may include a chemical bonding process, wherein the nonwoven web of fibers or the nonwoven layer of fibers is impregnated with a chemical binder composition, for example a latex binder composition, and curing the binder composition. The step of consolidating the nonwoven web of fibers into a nonwoven layer of fibers may include a thermal bonding process, such as hot air bonding and/or calendaring.

[0021] The recycled polyester polymer provided in the material composition in the process preferably is a recycled polyethylene terephthalate enabling to obtain high breaking strength and high elongation at break in the fibers and/or in the nonwoven layer of fibers.

[0022] The polyolefin polymer provided in the material composition in the process may be a polyethylene, preferably a recycled polyethylene.

[0023] The compatibilizer provided in the material composition in the process may be a polyolefin polymer grafted with maleic anhydride, a polyolefin co-polymer grafted with maleic anhydride or a mixture thereof. Preferably, the fibers of the nonwoven layer of fibers comprise less than 4 wt.%, more preferably less than 3 wt.%, more preferably less than 2 wt.%, even more preferably less than 1 wt.%, of a polyolefin polymer grafted with maleic anhydride, a polyolefin co-polymer grafted with maleic anhydride or a mixture thereof.

[0024] The compatibilizer provided in the material composition in the process may be an ethylene-methyl-acrylate copolymer, an ethylene-methyl-acrylate copolymer grafted with maleic anhydride or a mixture thereof. Preferably, the material composition comprises less than 4 wt.%, more preferably less than 3 wt.%, more preferably less than 2 wt.%, even more preferably less than 1 wt.%, most preferably less than 0.5 wt.%, of an ethylene-methyl-acrylate copolymer, an ethylene-methyl-acrylate copolymer grafted with maleic anhydride or a mixture thereof.

[0025] The compatibilizer provided in the material composition in the process may be a polymer comprising a glycidyl-methacrylate monomer, for example an ethylene-butyl acrylate - glycidyl methacrylate reactive terpolymer. Preferably, the fibers of the nonwoven layer of fibers comprise less than 4 wt.%, more preferably less than 3 wt.%, more preferably less than 2.5 wt.%, even more preferably less than 1.5 wt.%, of a polymer comprising a glycidyl-methacrylate monomer, for example an ethylene-butyl acrylate - glycidyl methacrylate reactive terpolymer.

[0026] The process may exclude a step of polycondensation of the recycled polyester for increasing the viscosity of the recycled polyester, as the material composition provided in the process enables extruding the material composition through spinning holes in a spinneret to form extruded fibers having high average tenacity, preferably of at least 2.4 cN/dtex, although the viscosity of the compound comprising the recycled polyester polymer and the polyolefin polymer is relatively low.

[0027] The material composition in the process may have a viscosity of at least 1100 Poise (110 Pa.s), or at least 1200 Poise (120 Pa.s), or at least 1250 Poise (125 Pa.s), calculated from the melt flow rate determined with a melt flow tester at a temperature of 290°C and a weight of 2.16 kg and a humidity level in the material composition of 91 ppm, enabling to improve the strength of the fibers.

[0028] The process may include a step of providing threads extending in the longitudinal direction of the material, in particular of high modulus threads comprising fibers, filaments or yarns, such as for example based on glass, polyester, carbon, cellulose or metal, to enable that the material can withstand elevated temperatures and/or high tensions which are often encountered in production processes wherein carrier materials are used. Preferably, the threads are based on glass.

[0029] The layer of threads extending in the longitudinal direction of the material may be provided in the process as a layer of unidirectional threads spaced at a distance from one another, preferably spaced at a constant distance from one another. The distance between two neighbouring threads extending in the longitudinal direction of the material may be at

least 1 mm, preferably at least 2 mm, more preferably at least 5 mm.

[0030] The layer of threads extending in the longitudinal direction of the material may be provided in the process as a scrim. As is well known to the person skilled in the art, a scrim is an open lattice structure composed of at least two sets of parallel threads, wherein the first group of parallel threads is oriented at an angle, generally at a 90° angle, to the second group of parallel threads. The first group of parallel threads may be connected to the second group of parallel threads by chemical bonding and/or the first group of parallel threads may be interwoven with the second group of parallel threads to form a woven scrim. Preferably, the openings in the scrim have at least one dimension in the plane of the carrier material being at least 1 mm, preferably at least 2 mm, more preferably at least 5 mm. More preferably, the openings in the scrim have two dimensions in the plane of the carrier material being at least 1 mm, preferably at least 2 mm, more preferably at least 5 mm.

[0031] A nonwoven layer of fibers is understood to be a sheet of fibers, continuous filaments, or chopped yarns of any nature or origin, that have been formed into a web by any means, and bonded together by any means, with the exception of weaving or knitting, and felts obtained by wet milling are not nonwovens. In the nonwoven layer of fibers, the continuous filaments, or chopped yarns are randomly laid and do not follow any specific orientation.

[0032] The nonwoven layer of fibers comprised in the material may be any type of nonwoven, such as for example staple fiber nonwovens produced by well-known processes, such as for example carding processes, wet-laid processes or air-laid processes or any combination thereof. The nonwoven layer of fibers may also be a nonwoven composed of filaments produced by well-known spunbonding processes wherein filaments are extruded from a spinneret and subsequently laid down on a conveyor belt as a web of filaments and subsequently consolidating the web to form a nonwoven layer of fibers, or by a two-step process wherein filaments are spun and wound on bobbins, preferably in the form of multifilament yarns, followed by the step of unwinding the yarns and/or multifilament yarns from the bobbins, optionally opening the multi-filament yarns into essentially individual filaments and/or filaments groups comprising 2 to 50 filaments, preferably 2 to 25 filaments, more preferably 2 to 10 filaments, and laying the filaments and/or filament groups down on a moving conveyor belt as a web of filaments and bonding the web to form a nonwoven layer of fibers.

[0033] Within the scope of the present invention, it is understood that the term fibers refers to both staple fibers and filaments. Staple fibers are fibers which have a specified, relatively short length in the range of 2 to 200 mm. Filaments are fibers having a length of more than 200 mm, preferably more than 500 mm, more preferably more than 1000 mm. Filaments may even be virtually endless, for example when formed by continuous extrusion and spinning of a filament through a spinning hole in a spinneret.

[0034] Preferably, the fibers in the nonwoven layer of fibers are filaments in order to provide higher tensile strength and/or higher tear strength to the material useable as a carrier material for bitumen membranes.

[0035] The fibers in the nonwoven layer of fibers may have a linear density in the range of 1 to 25 dtex, preferably in the range of 1 to 20 dtex, more preferably in the range of 1.5 to 15 dtex, most preferably in the range of 2 to 10 dtex to provide high tensile strength and/or tear strength in combination with good mass regularity to the carrier material while maintaining sufficient structure openness for impregnation, for example with bitumen into the material. The unit dtex defines the fineness of the fibers as their weight in grams per 10000 meter, and can be determined in accordance with ISO 1973, using a Lenzing Vibroskope 400.

[0036] The fibers in the nonwoven layer of fibers may have any cross-sectional shape, including round, trilobal, multi-lobal or rectangular.

[0037] The fibers in the nonwoven layer of fibers may be mono-component fibers, bicomponent fibers, such as bicomponent fibers having a core-sheath, a side-by-side or a segmented pie configuration, or multi-component fibers.

[0038] A material comprising a nonwoven layer of fibers is provided wherein the nonwoven layer of fibers comprises fibers comprising 90 to 98.7 wt.% of a recycled polyester polymer, 1 to 5 wt.% of a polyolefin polymer and a 0.3 to 5 wt.% of a compatibilizer based on the total weight of the recycled polyester polymer, the polyolefin polymer and the compatibilizer.

[0039] The fibers comprised in the nonwoven layer of fibers may have an average tenacity of at least 2.4 cN/dtex as determined according to ISO 5079:2020. The fibers comprised in the nonwoven layer of fibers may have an average tenacity of at least 2.5 cN/dtex, or at least 2.7 cN/dtex, or of at least 3.0 cN/dtex, or of at least 3.5 cN/dtex, or of at least 4.0 cN/dtex.

[0040] The fibers comprised in the nonwoven layer of fibers may have an elongation at break of at least 50%, preferably of at least 60%, more preferably of at least 70%.

[0041] The nonwoven layer of fibers comprised in the material may be consolidated by any known suitable consolidating process or any combination of consolidating processes. The nonwoven layer of fibers comprised in the material may be consolidated by mechanical bonding processes, such as mechanical needling and/or fluid entanglement, for example hydroentanglement, by chemical bonding processes, wherein the nonwoven web of fibers or the nonwoven layer of fibers is impregnated with a chemical binder composition, for example a latex binder composition, and curing the binder composition, and/or by thermal bonding processes, such as hot air bonding and/or calendaring.

[0042] The recycled polyester polymer comprised in the fibers of the nonwoven layer of fibers preferably is a recycled polyethylene terephthalate enabling to obtain high breaking strength and high elongation at break in the fibers and/or in the

nonwoven layer of fibers.

[0043] The polyolefin polymer comprised in the fibers of the nonwoven layer of fibers may be a polyethylene, preferably a recycled polyethylene.

[0044] The compatibilizer comprised in the fibers of the nonwoven layer of fibers may be a polyolefin polymer grafted with maleic anhydride, a polyolefin co-polymer grafted with maleic anhydride or a mixture thereof. Preferably, the fibers of the nonwoven layer of fibers comprise less than 4 wt.%, more preferably less than 3 wt.%, more preferably less than 2 wt.%, even more preferably less than 1 wt.%, of a polyolefin polymer grafted with maleic anhydride, a polyolefin co-polymer grafted with maleic anhydride or a mixture thereof.

[0045] The compatibilizer comprised in the fibers of the nonwoven layer of fibers may be an ethylene-methyl-acrylate copolymer, an ethylene-methyl-acrylate copolymer grafted with maleic anhydride or a mixture thereof. Preferably, the fibers of the nonwoven layer of fibers comprise less than 4 wt.%, more preferably less than 3 wt.%, more preferably less than 2 wt.%, even more preferably less than 1 wt.%, most preferably less than 0.5 wt.%, of an ethylene-methyl-acrylate copolymer, an ethylene-methyl-acrylate copolymer grafted with maleic anhydride or a mixture thereof.

[0046] The compatibilizer provided in the material composition in the process may be a polymer comprising a glycidyl-methacrylate monomer, for example an ethylene-butyl acrylate - glycidyl methacrylate reactive terpolymer. Preferably, the fibers of the nonwoven layer of fibers comprise less than 4 wt.%, more preferably less than 3 wt.%, more preferably less than 2.5 wt.%, even more preferably less than 1.5 wt.%, of a polymer comprising a glycidyl-methacrylate monomer, for example an ethylene-butyl acrylate - glycidyl methacrylate reactive terpolymer.

[0047] The material comprising a nonwoven layer of fibers may further comprise threads extending in the longitudinal direction of the material, in particular of high modulus threads comprising fibers, filaments or yarns, such as for example based on glass, polyester, carbon, cellulose or metal, to enable that the material can withstand elevated temperatures and/or high tensions which are often encountered in production processes wherein carrier materials are used. Preferably, the threads are based on glass.

[0048] The layer of threads extending in the longitudinal direction of the material may be provided as a layer of unidirectional threads spaced at a distance from one another, preferably spaced at a constant distance from one another. The distance between two neighbouring threads extending in the longitudinal direction of the material may be at least 1 mm, preferably at least 2 mm, more preferably at least 5 mm.

[0049] The layer of threads extending in the longitudinal direction of the material may be provided as a scrim. As is well known to the person skilled in the art, a scrim is an open lattice structure composed of at least two sets of parallel threads, wherein the first group of parallel threads is oriented at an angle, generally at a 90° angle, to the second group of parallel threads. The first group of parallel threads may be connected to the second group of parallel threads by chemical bonding and/or the first group of parallel threads may be interwoven with the second group of parallel threads to form a woven scrim. Preferably, the openings in the scrim have at least one dimension in the plane of the carrier material being at least 1 mm, preferably at least 2 mm, more preferably at least 5 mm. More preferably, the openings in the scrim have two dimensions in the plane of the carrier material being at least 1 mm, preferably at least 2 mm, more preferably at least 5 mm.

[0050] The nonwoven layer of fibers comprised in the material may be any type of nonwoven, such as for example staple fiber nonwovens produced by well-known processes, such as for example carding processes, wet-laid processes or air-laid processes or any combination thereof. The nonwoven layer of fibers may also be a nonwoven composed of filaments produced by well-known spunbonding processes wherein filaments are extruded from a spinneret and subsequently laid down on a conveyor belt as a web of filaments and subsequently consolidating the web to form a nonwoven layer of fibers, or by a two-step process wherein filaments are spun and wound on bobbins, preferably in the form of multifilament yarns, followed by the step of unwinding the yarns and/or multifilament yarns from the bobbins, optionally opening the multi-filament yarns into essentially individual filaments and/or filaments groups comprising 2 to 50 filaments, preferably 2 to 25 filaments, more preferably 2 to 10 filaments, and laying the filaments and/or filament groups down on a moving conveyor belt as a web of filaments and bonding the web to form a nonwoven layer of fibers.

[0051] Preferably, the fibers in the nonwoven layer of fibers are filaments in order to provide higher tensile strength and/or higher tear strength to the material useable as a carrier material for bitumen membranes.

[0052] The fibers in the nonwoven layer of fibers may have a linear density in the range of 1 to 25 dtex, preferably in the range of 1 to 20 dtex, more preferably in the range of 1.5 to 15 dtex, most preferably in the range of 2 to 10 dtex to provide high tensile strength and/or tear strength in combination with good mass regularity to the carrier material while maintaining sufficient structure openness for impregnation, for example with bitumen into the material.

[0053] The fibers in the nonwoven layer of fibers may have any cross-sectional shape, including round, trilobal, multi-loval or rectangular.

[0054] The fibers in the nonwoven layer of fibers may be mono-component fibers, bicomponent fibers, such as bicomponent fibers having a core-sheath, a side-by-side or a segmented pie configuration, or multi-component fibers.

[0055] The material comprising a nonwoven layer of fibers comprising fibers comprising 90 to 98.7 wt.% of a recycled polyester polymer, 1 to 5 wt.% of a polyolefin polymer and 0.3 to 5 wt.% of a compatibilizer based on the total weight of the recycled polyester polymer, the polyolefin polymer and the compatibilizer, may be used as a carrier material in various

applications, including as a carrier material for bitumen membranes for waterproofing, for example in roofing applications, as a primary backing or secondary backing for tufted carpets, as a carrier material for vinyl flooring, and as carrier material for filter media.

[0056] A fiber for a nonwoven layer of fibers is provided, the fiber comprising 90 to 98.7 wt.% of a recycled polyester polymer, 1 to 5 wt.% of a polyolefin polymer and a 0.3 to 5 wt.% of a compatibilizer based on the total weight of the recycled polyester polymer, the polyolefin polymer and the compatibilizer.

[0057] The fiber for a nonwoven layer of fibers may have an average tenacity of at least 2.4 cN/dtex as determined according to ISO 5079:2020. The fiber for a nonwoven layer of fibers may have an average tenacity of at least 2.5 cN/dtex, or at least 2.7 cN/dtex, or of at least 3.0 cN/dtex, or of at least 3.5 cN/dtex, or of at least 4.0 cN/dtex.

[0058] The fiber for a nonwoven layer of fibers may have an elongation at break of at least 50%, preferably of at least 60%, more preferably of at least 70%.

[0059] The recycled polyester polymer comprised in the fibers for a nonwoven layer of fibers preferably is a recycled polyethylene terephthalate enabling to obtain high breaking strength and high elongation at break in the fiber.

[0060] The polyolefin polymer comprised in the fiber for a nonwoven layer of fibers may be a polyethylene, preferably a recycled polyethylene.

[0061] The compatibilizer comprised in the fiber for a nonwoven layer of fibers may be a polyolefin polymer grafted with maleic anhydride, a polyolefin co-polymer grafted with maleic anhydride or a mixture thereof. Preferably, the fiber for a nonwoven layer of fibers comprises less than 4 wt.%, more preferably less than 3 wt.%, more preferably less than 2 wt.%, even more preferably less than 1 wt.%, of a polyolefin polymer grafted with maleic anhydride, a polyolefin co-polymer grafted with maleic anhydride or a mixture thereof.

[0062] The compatibilizer comprised in the fiber for a nonwoven layer of fibers may be an ethylene-methyl-acrylate copolymer, an ethylene-methyl-acrylate copolymer grafted with maleic anhydride or a mixture thereof. Preferably, the fiber for a nonwoven layer of fibers comprises less than 4 wt.%, more preferably less than 3 wt.%, more preferably less than 2 wt.%, even more preferably less than 1 wt.%, most preferably less than 0.5 wt.%, of an ethylene-methyl-acrylate copolymer, an ethylene-methyl-acrylate copolymer grafted with maleic anhydride or a mixture thereof.

[0063] The compatibilizer provided in the material composition in the process may be a polymer comprising a glycidyl-methacrylate monomer, for example an ethylene-butyl acrylate - glycidyl methacrylate reactive terpolymer. Preferably, the fibers of the nonwoven layer of fibers comprise less than 4 wt.%, more preferably less than 3 wt.%, more preferably less than 2.5 wt.%, even more preferably less than 1.5 wt.%, of a polymer comprising a glycidyl-methacrylate monomer, for example an ethylene-butyl acrylate - glycidyl methacrylate reactive terpolymer.

[0064] The fiber for a nonwoven layer of fibers may have a linear density in the range of 1 to 25 dtex, preferably in the range of 1 to 20 dtex, more preferably in the range of 1.5 to 15 dtex, most preferably in the range of 2 to 10 dtex to provide high tensile strength and/or tear strength to the nonwoven layer of fibers while enabling to maintain sufficient structure openness for impregnation, for example with bitumen into the material.

[0065] The fiber for a nonwoven layer of fibers may have any cross-sectional shape, including round, trilobal, multi-lobal or rectangular.

[0066] The fiber for a nonwoven layer of fibers may be a mono-component fiber, a bicomponent fiber, such as a bicomponent fiber having a core-sheath, a side-by-side or a segmented pie configuration, or a multi-component fiber.

40 Examples.

[0067] Fibers are formed by provided a material composition, melting the material composition in an extruder at 285°C, extruding the molten material composition at a throughput of 2.2 g/min/spinning hole and 2.5 g/min/spinning hole through the spinning holes of a spinneret comprising four spinning holes to form extruded fibers, and winding the fibers.

45 Example 1

[0068] The material composition in example 1 was basically composed of 70 wt.% of recycled polyethylene terephthalate from PET bottles and 30 wt.% of a recycled polyester comprising 5.3 wt.% of polyethylene, but additionally comprised 1.25 wt.% of a compatibilizer, being a polymer comprising a glycidyl-methacrylate monomer, based on the total weight of the recycled polyester polymer, the polyolefin polymer and the compatibilizer. The compatibilizer was an ethylene-butyl acrylate - glycidyl methacrylate reactive terpolymer having a melt flow rate of 12 g/10 min according to ISO1133 at a temperature of 190°C and a weight of 2.16 kg. Fibers were spun at a throughput of 2.2 g/min/hole (example 1a) and 2.5 g/min/hole (example 1b).

55 Example 2

[0069] The material composition in example 2 was basically composed of 70 wt.% of recycled polyethylene terephtha-

late from PET bottles and 30 wt.% of a recycled polyester comprising 5.3 wt.% of polyethylene, but additionally comprised 2.5 wt.% of a compatibilizer, being a polyolefin grafted maleic anhydride, based on the total weight of the recycled polyester polymer, the polyolefin polymer and the compatibilizer. The compatibilizer was a statistic copolymer based on polypropylene and polyethylene grafted with maleic anhydride having a maleic anhydride content of 1.7% and a melt flow rate of 30 g/10 min according to ISO1133 at a temperature of 190°C and a weight of 2.16 kg. The material composition had a viscosity of 1280 Poise (128 Pa.s) calculated from the melt flow rate determined with a melt flow tester at a temperature of 290°C and a weight of 2.16 kg and a humidity level in the material composition of 91 ppm. Fibers were spun at a throughput of 2.2 g/min/hole (example 2a) and 2.5 g/min/hole (example 2b).

10 Example 3

[0070] The material composition in example 3 was basically composed of 70 wt.% of recycled polyethylene terephthalate from PET bottles and 30 wt.% of a recycled polyester comprising 5.3 wt.% of polyethylene, but additionally comprised 1.25 wt.% of a compatibilizer, being an ethylene-methyl-acrylate random copolymer, based on the total weight of the recycled polyester polymer, the polyolefin polymer and the compatibilizer. The compatibilizer was an ethylene-methyl-acrylate copolymer having a methyl-acrylate content of 24 wt.% and a melt index of 10.0 g/10 min according to D1238 at a temperature of 190°C and a weight of 2.16 kg. Fibers were spun at a throughput of 2.2 g/min/hole (example 3a) and 2.5 g/min/hole (example 3b).

20 Example 4

[0071] The material composition in example 4 was basically composed of 70 wt.% of recycled polyethylene terephthalate from PET bottles and 30 wt.% of a recycled polyester comprising 5.3 wt.% of polyethylene, but additionally comprised 1.25 wt.% of a compatibilizer, being an ethylene-methyl-acrylate block copolymer, based on the total weight of the recycled polyester polymer, the polyolefin polymer and the compatibilizer. The compatibilizer was an ethylene methyl acrylate copolymer having a methyl-acrylate content of 22 wt.% and a melt index of 2.0 g/10 min according to D1238 at a temperature of 190°C and a weight of 2.16 kg. Fibers were spun at a throughput of 2.2 g/min/hole (example 4a) and 2.5 g/min/hole (example 4b).

30 Example 5

[0072] The material composition in example 5 was basically composed of 70 wt.% of recycled polyethylene terephthalate from PET bottles and 30 wt.% of a recycled polyester comprising 5.3 wt.% of polyethylene, but additionally comprised 1.25 wt.% of a compatibilizer, being an ethylene-methyl-acrylate random copolymer grafted with maleic anhydride, based on the total weight of the recycled polyester polymer, the polyolefin polymer and the compatibilizer. The compatibilizer was a maleic anhydride modified ethylene methyl acrylate copolymer having a methyl-acrylate content of 24 wt.% and a melt index of 2.7 g/10 min according to D1238. Fibers were spun at a throughput of 2.2 g/min/hole (example 5a) and 2.5 g/min/hole (example 5b).

40 Comparative Example 1

[0073] The material composition in comparative example 1 consisted for 100% of recycled polyethylene terephthalate from PET bottles. Fibers were spun at a throughput of 2.2 g/min/hole (comparative example 1a) and 2.5 g/min/hole (comparative example 1b). The material composition had a viscosity of 1401 Poise (140.1 Pa.s) calculated from the melt flow rate determined with a melt flow tester at a temperature of 290°C and a weight of 2.16 kg and a humidity level in the material composition of 91 ppm. Fibers were spun at a throughput of 2.2 g/min/hole (Comp. 1a) and 2.5 g/min/hole (Comp. 1b).

50 Comparative Example 2

[0074] The material composition in comparative example 2 consisted for 70 wt.% of recycled polyethylene terephthalate from PET bottles and 30 wt.% of a recycled polyester comprising 5.3 wt.% of polyethylene. Fibers were spun at a throughput of 2.2 g/min/hole and 2.5 g/min/hole. The material composition had a viscosity of 1083 Poise (108.3 Pa.s) calculated from the melt flow rate determined with a melt flow tester at a temperature of 290°C and a weight of 2.16 kg and a humidity level in the material composition of 91 ppm. Fibers were spun at a throughput of 2.2 g/min/hole (Comp. 2a) and 2.5 g/min/hole (Comp. 2b).

Table 1: Fiber properties

Example	Throughput (g/m in/hole)	Linear density (dtex)	Tenacity (cN/dtex)	Elongation (%)	Stable spinning	Broken fibers
5 1a	2.2	4.92	2.4	77.0	Yes	Few
	1b	2.5	5.62	2.4	85.4	Few
10 2a	2.2	6.21	2.7	76.8	Yes	Few
	2b	2.5	6.10	3.0	66.9	Few
15 3a	2.2	4.83	3.0	55.2	Yes	Few
	3b	2.5	4.96	3.2	54.1	Few
20 4a	2.2	4.44	3.4	51.7	Yes	Few
	4b	2.5	4.96	3.0	61.0	Few
25 5a	2.2	5.04	3.0	61.3	Yes	Few
	5b	2.5	5.73	2.8	69.9	Few
30 Comp. 1a	2.2	5.21	3.2	61.1	Yes	Few
	Comp. 1b	2.5	5.54	3.2	65.4	Few
35 Comp. 2a	2.2	5.79	2.6	76.7	No	Many
	Comp. 2b	2.5	5.65	2.6	69.3	Many

[0075] Due to the presence of polyethylene contamination, the fibers formed from a material composition which consisted for 70 wt.% of recycled polyethylene terephthalate from PET bottles and 30 wt.% of a recycled polyester comprising 5.3 wt.% of polyethylene contamination, of comparative example 2a and 2b, have a lower tenacity and a higher elongation than the fibers formed from a material composition which consisted for 100% of recycled polyethylene terephthalate from PET bottles of comparative example 1a and 1b. Fiber spinning was very unstable with a large number of broken filaments.

[0076] Due to the fact that the material composition of example 1 comprised a polymer comprising a glycidyl-methacrylate monomer, the fiber spinning became stable and the number of broken filaments was reduced, as compared to fibers made from the material composition of comparative example 2.

[0077] Due to the fact that the material composition of example 2 comprised a polyolefin grafted maleic anhydride, the fiber spinning became stable and the number of broken filaments was reduced, as compared to fibers made from the material composition of comparative example 2. The tenacity of the fibers was increased as compared to fibers made from the material composition of comparative example 2.

[0078] Due to the fact that the material composition of example 3 comprised an ethylene-methyl-acrylate random copolymer, the fiber spinning became stable and the number of broken filaments was reduced, as compared to fibers made from the material composition of comparative example 2. The tenacity of the fibers was increased as compared to fibers made from the material composition of comparative example 2. The tenacity of the fibers at a throughput of 2.5 g/min/spinning hole was even at the same level as fibers made from the material composition of comparative example 1, i.e. from 100% of recycled polyethylene terephthalate from PET bottles.

[0079] Due to the fact that the material composition of example 4 comprised an ethylene-methyl-acrylate block copolymer, the fiber spinning became stable and the number of broken filaments was reduced, as compared to fibers made from the material composition of comparative example 2. The tenacity of the fibers was increased as compared to fibers made from the material composition of comparative example 2. The tenacity of the fibers at a throughput of 2.2 g/min/spinning hole was even at the higher level as fibers made from the material composition of comparative example 1, i.e. from 100% of recycled polyethylene terephthalate from PET bottles.

[0080] Due to the fact that the material composition of example 5 comprised an ethylene-methyl-acrylate random copolymer grafted with maleic anhydride, the fiber spinning became stable and the number of broken filaments was reduced, as compared to fibers made from the material composition of comparative example 2. The tenacity of the fibers was increased as compared to fibers made from the material composition of comparative example 2.

Claims

1. A process for providing a material comprising a nonwoven layer of fibers comprising the steps of providing a material composition comprising 90 to 98.7 wt.% of a recycled polyester polymer, 1 to 5 wt.% of a polyolefin polymer and 0.3 to 5 wt.% of a compatibilizer based on the total weight of the recycled polyester polymer, the polyolefin polymer and the compatibilizer, melting the material composition, extruding the molten material composition through spinning holes in a spinneret to form extruded fibers, supplying the extruded fibers to form a nonwoven web of fibers and consolidating the nonwoven web to obtain a material comprising a nonwoven layer of fibers.
- 10 2. The process according to claim 1 wherein the fibers have an average tenacity of at least 2.4 cN/dtex determined according to ISO 5079:2020.
- 15 3. The process according to any one of claims 1 to 2 wherein the recycled polyester polymer is a recycled polyethylene terephthalate.
4. The process according to any one of claims 1 to 3 wherein the polyolefin polymer is a polyethylene, preferably a recycled polyethylene.
- 20 5. The process according to any one of claims 1 to 4 wherein the compatibilizer is a polyolefin polymer grafted with maleic anhydride, a polyolefin co-polymer grafted with maleic anhydride or a mixture thereof.
6. The process according to any one of claims 1 to 4 wherein the compatibilizer is an ethylene-methyl-acrylate copolymer, an ethylene-methyl-acrylate copolymer grafted with maleic anhydride or a mixture thereof.
- 25 7. The process according to any one of claims 1 to 4 wherein the compatibilizer is a polymer comprising a glycidyl-methacrylate monomer.
8. A material comprising a nonwoven layer of fibers wherein the nonwoven layer of fibers comprises fibers comprising 90 to 98.7 wt.% of a recycled polyester polymer, 1 to 5 wt.% of a polyolefin polymer and a 0.3 to 5 wt.% of a compatibilizer based on the total weight of the recycled polyester polymer, the polyolefin polymer and the compatibilizer.
- 30 9. The material comprising a nonwoven layer of fibers according to claim 8 wherein the fibers have an average tenacity of at least 2.4 cN/dtex determined according to ISO 5079:2020.
- 35 10. The material according to any one of claims 8 to 9 wherein the polyester is a polyethylene terephthalate.
11. The material according to any one of claims 8 to 10 wherein the polyolefin is a polyethylene, preferably a recycled polyethylene.
- 40 12. The material according to any one of claims 8 to 11 wherein the compatibilizer is a polyolefin polymer grafted with maleic anhydride, a polyolefin co-polymer grafted with maleic anhydride or a mixture thereof.
13. The material according to any one of claims 8 to 11 wherein the compatibilizer is an ethylene-methyl-acrylate copolymer, an ethylene-methyl-acrylate copolymer grafted with maleic anhydride or a mixture thereof.
- 45 14. A fiber for a nonwoven layer of fibers comprising 90 to 98.7 wt.% of a recycled polyester, 1 to 5 wt.% of a polyolefin and a 0.3 to 5 wt.% of a compatibilizer based on the total weight of the recycled polyester, the polyolefin and the compatibilizer.
- 50 15. The fiber according to claim 14 wherein the fiber has an average tenacity of at least 2.4 cN/dtex determined according to ISO 5079:2020.

Patentansprüche

1. Verfahren zur Bereitstellung eines Materials, das eine Faservliesschicht umfasst, umfassend die Schritte der Bereitstellung einer Materialzusammensetzung umfassend 90 bis 98,7 Gew.-% an einem recycelten Polyesterpolymer, 1 bis 5 Gew.-% an einem Polyolefinpolymer und 0,3 bis 5 Gew.-% an einem Verträglichmacher bezogen auf das

Gesamtgewicht des recycelten Polyesterpolymers, des Polyolefinpolymers und des Verträglichmachers, Schmelzen der Materialzusammensetzung, Extrudieren der geschmolzenen Materialzusammensetzung durch Spinnlöcher in einer Spinndüse, um extrudierte Fasern zu bilden, Zuführen der extrudierten Fasern, um eine Faservliesbahn zu bilden, und Konsolidieren der Vliesbahn, um ein Material zu erhalten, das eine Faservliesschicht umfasst.

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2. Verfahren gemäß Anspruch 1, wobei die Fasern eine mittlere Festigkeit von wenigstens 2,4 cN/dtex, bestimmt gemäß ISO 5079:2020, aufweisen.
- 10 3. Verfahren gemäß einem der Ansprüche 1 bis 2, wobei das recycelte Polyesterpolymer ein recyceltes Polyethylen-terephthalat ist.
4. Verfahren gemäß einem der Ansprüche 1 bis 3, wobei das Polyolefinpolymer ein Polyethylen, vorzugsweise ein recyceltes Polyethylen, ist.
- 15 5. Verfahren gemäß einem der Ansprüche 1 bis 4, wobei der Verträglichmacher ein mit Maleinsäureanhydrid gepfropftes Polyolefinpolymer, ein mit Maleinsäureanhydrid gepfropftes Polyolefin-Copolymer oder ein Gemisch davon ist.
- 20 6. Verfahren gemäß einem der Ansprüche 1 bis 4, wobei der Verträglichmacher ein Ethylen-Methylacrylat-Copolymer, ein mit Maleinsäureanhydrid gepfropftes Ethylen-Methylacrylat-Copolymer oder ein Gemisch davon ist.
7. Verfahren gemäß einem der Ansprüche 1 bis 4, wobei der Verträglichmacher ein Polymer ist, das ein Glycidylmethacrylat-Monomer umfasst.
- 25 8. Material umfassend eine Faservliesschicht, wobei die Faservliesschicht Fasern umfasst, die 90 bis 98,7 Gew.-% an einem recycelten Polyesterpolymer, 1 bis 5 Gew.-% an einem Polyolefinpolymer und 0,3 bis 5 Gew.-% an einem Verträglichmacher bezogen auf das Gesamtgewicht des recycelten Polyesterpolymers, des Polyolefinpolymers und des Verträglichmachers umfassen.
- 30 9. Material umfassend eine Faservliesschicht gemäß Anspruch 8, wobei die Fasern eine mittlere Festigkeit von wenigstens 2,4 cN/dtex, bestimmt gemäß ISO 5079:2020, aufweisen.
10. Material gemäß einem der Ansprüche 8 bis 9, wobei der Polyester ein Polyethylenterephthalat ist.
- 35 11. Material gemäß einem der Ansprüche 8 bis 10, wobei das Polyolefin ein Polyethylen, vorzugsweise ein recyceltes Polyethylen, ist.
12. Material gemäß einem der Ansprüche 8 bis 11, wobei der Verträglichmacher ein mit Maleinsäureanhydrid gepfropftes Polyolefinpolymer, ein mit Maleinsäureanhydrid gepfropftes Polyolefin-Copolymer oder ein Gemisch davon ist.
- 40 13. Material gemäß einem der Ansprüche 8 bis 11, wobei der Verträglichmacher ein Ethylen-Methylacrylat-Copolymer, ein mit Maleinsäureanhydrid gepfropftes Ethylen-Methylacrylat-Copolymer oder ein Gemisch davon ist.
14. Faser für eine Faservliesschicht, umfassend 90 bis 98,7 Gew.-% an einem recycelten Polyester, 1 bis 5 Gew.-% an einem Polyolefin und 0,3 bis 5 Gew.-% an einem Verträglichmacher bezogen auf das Gesamtgewicht des recycelten Polyester, des Polyolefins und des Verträglichmachers.
- 45 15. Faser gemäß Anspruch 14, wobei die Faser eine mittlere Festigkeit von wenigstens 2,4 cN/dtex, bestimmt gemäß ISO 5079:2020, aufweist.

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Revendications

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1. Procédé pour la fourniture d'un matériau comprenant une couche non-tissée de fibres comprenant les étapes de fourniture d'une composition de matériau comprenant 90 à 98,7 % en poids d'un polymère de type polyester recyclé, 1 à 5 % en poids d'un polymère de type polyoléfine et 0,3 à 5 % en poids d'un agent de compatibilité sur la base du poids total du polymère de type polyester recyclé, du polymère de type polyoléfine et de l'agent de compatibilité, fusion de la composition de matériau, extrusion de la composition de matériau fondue à travers des trous de filage dans une filière

pour former des fibres extrudées, apport des fibres extrudées pour former une toile non-tissée de fibres et consolidation de la toile non-tissée pour obtenir un matériau comprenant une couche non-tissée de fibres.

2. Procédé selon la revendication 1, les fibres ayant une ténacité moyenne d'au moins 2,4 cN/dtex déterminée selon la norme ISO 5079:2020.
3. Procédé selon l'une quelconque des revendications 1 à 2, le polymère de type polyester recyclé étant un poly(té-réphthalate d'éthylène) recyclé.
- 10 4. Procédé selon l'une quelconque des revendications 1 à 3, le polymère de type polyoléfine étant un polyéthylène, préféablement un polyéthylène recyclé.
- 15 5. Procédé selon l'une quelconque des revendications 1 à 4, l'agent de compatibilité étant un polymère de type polyoléfine greffé avec de l'anhydride maléique, un copolymère de type polyoléfine greffé avec de l'anhydride maléique ou un mélange correspondant.
- 20 6. Procédé selon l'une quelconque des revendications 1 à 4, l'agent de compatibilité étant un copolymère d'éthylène-acrylate de méthyle, un copolymère d'éthylène-acrylate de méthyle greffé avec de l'anhydride maléique ou un mélange correspondant.
7. Procédé selon l'une quelconque des revendications 1 à 4, l'agent de compatibilité étant un polymère comprenant un monomère de méthacrylate de glycidyle.
- 25 8. Matériau comprenant une couche non-tissée de fibres, la couche non-tissée de fibres comprenant des fibres comprenant 90 à 98,7 % en poids d'un polymère de type polyester recyclé, 1 à 5 % en poids d'un polymère de type polyoléfine et 0,3 à 5 % en poids d'un agent de compatibilité sur la base du poids total du polymère de type polyester recyclé, du polymère de type polyoléfine et de l'agent de compatibilité.
- 30 9. Matériau comprenant une couche non-tissée de fibres selon la revendication 8, les fibres ayant une ténacité moyenne d'au moins 2,4 cN/dtex déterminée selon la norme ISO 5079:2020.
10. Matériau selon l'une quelconque des revendications 8 à 9, le polyester étant un poly(téréphthalate d'éthylène).
- 35 11. Matériau selon l'une quelconque des revendications 8 à 10, la polyoléfine étant un polyéthylène, préféablement un polyéthylène recyclé.
12. Matériau selon l'une quelconque des revendications 8 à 11, l'agent de compatibilité étant un polymère de type polyoléfine greffé avec de l'anhydride maléique, un copolymère de type polyoléfine greffé avec de l'anhydride maléique ou un mélange correspondant.
- 40 13. Matériau selon l'une quelconque des revendications 8 à 11, l'agent de compatibilité étant un copolymère d'éthylène-acrylate de méthyle, un copolymère d'éthylène-acrylate de méthyle greffé avec de l'anhydride maléique ou un mélange correspondant.
- 45 14. Fibre pour une couche non-tissée de fibres comprenant 90 à 98,7 % en poids d'un polyester recyclé, 1 à 5 % en poids d'une polyoléfine et 0,3 à 5 % en poids d'un agent de compatibilité sur la base du poids total du polyester recyclé, de la polyoléfine et de l'agent de compatibilité.
- 50 15. Fibre selon la revendication 14, la fibre ayant une ténacité moyenne d'au moins 2,4 cN/dtex déterminée selon la norme ISO 5079:2020.

REFERENCES CITED IN THE DESCRIPTION

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Patent documents cited in the description

- EP 2455523 A [0007]