COMMONWEALTH of AUSTRALIA
Patents Act 1952

APPLICATION FOR A STANDARD PATENT

I/We

Freudenberg Spunweb S.A.

of

25 Quai Paul Doumer, Courbevoie, 92400, France

632216

hereby apply for the grant of a Standard Patent for an invention entitled:

Nonwovens of continuous polyester filaments their manufacture and their use

which is described in the accompanying complete specification.

Details of basic application(s):–

Number	Convention Country	Date
90 01708	France	8 February 1990

The address for service is care of DAVIES & COLLISON, Patent Attorneys, of 1 Little Collins Street, Melbourne, in the State of Victoria, Commonwealth of Australia.

DATED this EIGHTH day of FEBRUARY 1991

To: THE COMMISSIONER OF PATENTS

.....
a member of the firm of
DAVIES & COLLISON for
and on behalf of the
applicant(s)

Davies & Collison, Melbourne

M 024821 080291

COMMONWEALTH OF AUSTRALIA

PATENTS ACT 1952–1973

DECLARATION IN SUPPORT OF CONVENTION OR NON-CONVENTION APPLICATION FOR A PATENT OR PATENT OF ADDITION

Insert title of invention.

In support of the Application made for a ~~patent of addition~~ patent for an invention entitled: Nonwovens of Continuous Polyester Filaments their manufacture and their use

Insert full name(s) and address(es) of declarant(s) being the applicant(s) or person(s) authorized to sign on behalf of an applicant company.

I / ~~We~~ Daniel Braconnier, of
FREUDENBERG SPUNWEB S A
a French body corporate, of:-
25 Quai Paul Doumer 92400 Courbevoie
France

Cross out whichever of paragraphs 1(a) or 1(b) does not apply

1(a) relates to application made by individual(s)
1(b) relates to application made by company; insert name of applicant company.

do solemnly and sincerely declare as follows :-

1. (a) ~~I am~~ / ~~We are~~ ~~the applicant for the patent of addition~~

or (b) I am authorized by

FREUDENBERG SPUNWEB S.A.

the applicant for the ~~patent of addition~~ patent to make this declaration on ~~their~~ its behalf.

Cross out whichever of paragraphs 2(a) or 2(b) does not apply

2(a) relates to application made by inventor(s)
2(b) relates to application made by company(s) or person(s) who are not inventor(s); insert full name(s) and address(es) of inventors.

2. (a) I am / ~~We are~~ ~~the actual inventor of the invention~~

or (b)

Jean BARAVIAN
22 rue du Vesinet 78290 CROISSY/SEINE France
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1 rue du Platre 69002 LYON France
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31 avenue Clemenceau 68000 COLMAR France

~~is~~ / are the actual inventors of the invention and the facts upon which the applicant

~~is~~ / are entitled to make the application are as follows :-

State manner in which applicant(s) derive title from inventor(s)

This applicant would, if a patent were granted upon an application made by the inventors, be entitled to have the patent assigned to it. Rhone Poulenc Fibres assigned the right to claim priority from the basic applicant to Production Chimiques S.A. (now known as FREUDENBERG SPUNWEB S.A.

Cross out paragraphs 3 and 4 for non-convention applications. For convention applications, insert basic country(s) followed by date(s) and basic applicant(s).

3. The basic application as defined by Section 141 of the Act ~~was~~ / were made

in France - (90 01708) on the 8 Feb 90

by ~~xxxxxxxx~~ RHONE POULENC FIBRES

in ..... on the .....

by .....

in ..... on the .....

by .....

4. The basic application referred to in paragraph 3 of this Declaration ~~was~~ / were the first application made in a Convention country in respect of the invention the subject of the application.

Insert place and date of signature.

Declared at St Fons this 21 day of JANUARY 1991

Signature of declarant(s) (no attestation required)

FREUDENBERG SPUNWEB S.A,
The Mandatary;

Daniel BRACONNIER

Note: Initial all alterations.

(12) PATENT ABRIDGMENT (11) Document No. AU-B-70891/91
(19) AUSTRALIAN PATENT OFFICE (10) Acceptance No. 632216

(54) Title
NONWOVENS OF CONTINUOUS POLYESTER FILAMENTS THEIR MANUFACTURE AND THEIR USE

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(56) Prior Art Documents
US 3940302
US 3193516
EP 242037

(57) Claim

1. A nonwoven of continuous melt-extruded polyester filaments, at least 50% by weight of which contain 0.5 to 3% by weight of a polyorganosiloxane oil introduced into the polyester during the melt extrusion.

7. Process for the manufacture of a nonwoven as claimed in Claim 1, which comprises introducing, into a polyester melt in an extruder, by injection into the nozzle of the extruder after the melting of the polyester or by injection into the body of the extruder during the melting of the polyester, a polyorganosiloxane oil in a proportion of 0.5 to 3% by weight relative to the polyester, and extruding the polymer melt to produce the filaments by passage through orifices of a die.

632216

COMMONWEALTH OF AUSTRALIA

PATENTS ACT 1952

COMPLETE SPECIFICATION

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COMPLETE SPECIFICATION FOR THE INVENTION ENTITLED:

Nonwovens of continuous polyester filaments their manufacture and their use

The following statement is a full description of this invention, including the best method of performing it known to me/us:-

The present invention relates to nonwovens of synthetic continuous filaments, to their manufacture and to their use. The invention relates more particularly to nonwovens of continuous filaments which can be employed as backings for floor coverings or carpets with stitched or tufted pile.

The use of nonwovens of continuous filaments, generally made of polypropylene or polyester, as backings or supports for pile stitching is well known.

Polyester supports are employed in particular when it is desired to produce particularly stable carpets such as carpet tiles or printed carpets in strips with print patterns capable of being matched from one strip to another. Such uses require supports of great dimensional stability both during conversion (stitching, dyeing, printing, back-coating, etc.) and after they have been laid. Polyester supports are also employed for the manufacture of mouldable tufted carpets for motor vehicles because of their good elongation at break, their tearing strength and their good thermal behaviour.

However, the stitching of threads into such supports presents problems. In fact, the entry of the needles gives rise to breakages of strands and filaments, weakening the mechanical properties of the backing and, consequently, resulting in perturbations during the manufacture and faults in the back of the article, which

are detrimental to its robustness and hence to its subsequent use under normal conditions.

The most frequently proposed solution for overcoming this technical problem consists in spraying the nonwoven, or the filaments from which it is made, with a lubricating product when it is being manufactured. When lubricated in this way, the nonwoven can be entered by the needles without damage when the pile is stitched. Such processes are described, for example, in French patent applications published under Nos. 2,174,290 and 2,245,807, in which a product of polysiloxane type is employed as lubricant. For spraying the lubricating product, these processes require, during the manufacture of the nonwoven or afterwards, the use of additional plant, resulting in additional complications and handling costs.

The present invention provides a new nonwoven and a process for its manufacture, which offer a simple, economic answer to the problem discussed above, of maintaining the qualities of nonwovens when they are entered by needles when the pile is stitched to them.

The subject of the present invention is a nonwoven of continuous melt-extruded polyester filaments at least 50 % by weight of which contain from 0.5 to 3 %, preferably from 0.6 to 1.5 %, of a polyorganosiloxane oil introduced into the said filaments during the melt

extrusion.

Another subject of the present invention is a process for the manufacture of the above nonwoven, which comprises introducing into a polyester melt in an extruder, by injection into the nozzle of the extruder after the melting of the polyester or by injection into the body of the extruder during the melting of the polyester, a polyorganosiloxane oil in a proportion of 0.5 to 3%, by weight relative to the polyester, and extruding the polymer melt to produce the filaments by passage through orifices of a die.

The new nonwovens are more particularly useful as backings for floor coverings with stitched pile.

The filaments may be made of any synthetic polyester. Such polymers may be employed by themselves or in combinations to produce bilaminar filaments of the core-sheath or side/side type. Polymers of the same chemical nature but of different appearance may also be employed. Polyesters such as polyethylene terephthalate and polybutylene terephthalate may more particularly be used.

The polyorganosiloxane employed is preferably a polydiorganosiloxane oil and, preferably, a polydimethylsiloxane with a molecular weight from 1000 to 250,000, and preferably between 7,500 and 70,000, corresponding to a dynamic viscosity of 50 to 10,000

mPa s respectively. These oils are nonfunctionalised polydimethylsiloxanes. They are chemically and thermally stable at the polymer melting/spinning temperatures. The proportion in which the silicone oil is introduced in relation to the polymer may vary between 0.5 and 3 %, preferably 0.6 to 1.5 %.

It has been found that the quantity of polyorganosiloxane which is introduced into the melt has virtually no perturbing effect on the extrusion, transfer, spinning or filament-drawing conditions. The flow of the melt through the die orifices is made easier because of the lubrication brought about by the polyorganosiloxane contained in the melt, part of which later exudes on the surface of the filaments, forming a stable film.

The incorporation of the polyorganosiloxane into the polyester may be performed during or after the melting of the polyester in the body or nozzle of the extruder, and in general in any region of transfer of the molten polyester before it is converted into filaments.

The polyorganosiloxane may be introduced into the molten polyester in the extruder by means of a volumetric metering pump. Mass-based metering is completely suitable.

The process which is employed for the production of the continuous-filament nonwoven is of known type; it

is possible, for example, to employ that described in the French patent specifications published under No. 1,601,049 or No. 2,299,438 by the Applicant Company, the bonding of the sheet being performed by needling or heat-bonding with or without resin.

The polyorganosiloxane may be introduced into all the polymer(s) employed or into only a proportion of the latter, provided at least 50 % of the filaments contain the polyorganosiloxane. It is thus possible to extrude two different polymers in the form of separate or bilaminar filaments as mentioned before, with a proportion of the filaments in the first case or a single lamina in the second case containing the polyorganosiloxane.

To implement the present invention, use is preferably made of the nonwovens described in the Applicant's French Patent specification published under No. 2,546,537, in which polyethylene terephthalate and polybutylene terephthalate are employed.

The nonwovens of the present invention can be employed for all the usual textile or technical applications of nonwovens either by themselves or more particularly as supports for nonwovens with stitched pile. They can be coupled with other nonwovens and, if appropriate, reinforced with threads which are mutually parallel, preferably in the length direction. They may

be subjected to any embossing, forming, moulding, impregnating, coating or similar handling operation.

Various tests described below are employed for differentiating the nonwovens of the invention from nonwovens not containing the polyorganosiloxane in the bulk of the filaments.

Measurement of the needle entry force:

a) **In laboratory**

The measurement of the force of entry of a row of tufting needles into a sheet which has been consolidated by heat-bonding is performed as follows:

3 small bars of Singer tufting needles series No. 82,753, each comprising 12 needles are welded side by side on the same line, forming a 9-cm wide row of needles. This assembly is attached to the movable jaw of a tensometer and enters (at a speed of 1000 m/min) the fixed nonwoven surface stretched on a suitable device at right angles to the movement of the needles.

b) **On tufting machine**

The test is performed on a Singer-Cobble machine with 1/10"-gauge needles, the pile thread employed being a curled multifilament continuous yarn of 1100 dtex/88 strands Z torsion 150 • 2S 150 45, 40-point tension.

Determination of the polyorganosiloxane introduced into the bulk on filaments present in the nonwoven:

a) **Determination of the total % of silicone** (core

+ surface of the filaments)

By ethoxylation and gas phase chromatography: digestion of the nonwoven in an alkaline medium. This method makes it possible to separate each silicon atom, leaving it with its initial chemical environment. Analysis is then carried out using gas phase chromatography.

b) <u>Determination of % of silicone at the surface of the filaments</u>

By extraction with cyclohexane at 30°C, followed by weighing the residue after evaporation of the solvent. The determination of the polyorganosiloxane in this extract is then carried out, corresponding to the various products present at the surface of the filament, chiefly polyorganosiloxane and oligomers.

The following Examples illustrate the present invention:

<u>Example 1</u>:

As described in the Applicant's French patent specification published under No. 2,546,537, a nonwoven sheet of 120 g/m^2 is produced from continuous filaments of 2 types, polyethylene glycol terephthalate filaments (2GT) (85 %) and polybutylene glycol terephthalate filaments (4GT) (15 %) of 6 and 4 dtex counts respectively. The filaments are deposited by the travelling process which is the subject of the

Applicant's French patent specification published under No. 2,299,438.

1 % relative to the weight of the 2GT granules of Rhodorsil 47 V 350 silicone oil (polydimethylsiloxane with a viscosity of 350 mPa s from Rhône-Poulenc) is introduced using a metering pump, upstream of the 2GT extruder.

The sheet is needled on a single-stroke needler at a rate of 50 perforations/cm^2 with Singer needles of 40-Rb gauge, 15-mm entry.

The sheet is then heat-bonded by running "S"-fashion over a calender with two heating rolls at 230°C. The pressure between the rolls is 12 daN/cm and the total time of contact between the sheet and the hot rolls is 9 seconds. The sheet is then cooled and wound.

The determination of the proportion of the silicone oil which has exuded at the surface of the filaments gives a silicone content of 0.05 % relative to the weight of the sheet, the total quantity of silicone relative to the weight of the sheet being 1 %. The force of entry of the needles is, according to the laboratory test, 23.6 daN in the case of the sheet without silicone and 15.7 daN in the case of the sheet obtained as described above, showing unambiguously the difference in the entry forces and the advantage of employing the silicone product.

On a tufting machine, the sheet without silicone is impossible to run, because of breakage of filaments, and complete deterioration of the support after tearing, whereas the silicone-treated sheet results in an excellent tufting.

Tests were carried out according to previous methods using full-bath fulling in an aqueous silicone dispersion so as to have a dry deposit of 0.5 % of silicone relative to the weight of the nonwoven: the results are of the same order as those obtained with the sheet containing a silicone product in the bulk. The economy of the process, however, argues in favour of the present invention.

The tufted carpet has mechanical properties and a sufficient stability for being converted, after coating with bitumen on the reverse side, into tiles for floor covering.

Example 2:

A nonwoven sheet of 120 g/cm^2 of polyethylene terephthalate continuous filaments with a count of 7 dtex is produced using the process outlined in Example 1, care having been taken to introduce into the polymer 0.5 % of silicone oil (polydimethylsiloxane with a viscosity of 1000 mPa s) relative to the weight of polymer.

The sheet is then needled with Singer 40 RB needles at a rate of 80 perforations/cm^2 and 13 mm of

entry on 1 single face.

A heat treatment is then carried out by hot calendering between 2 rolls heated to a temperature of 235°C with a pressure of 25 daN/cm and a contact time of 8 seconds.

This support is tufted without any difficulty and exhibits all the toughness and deformability properties required for the production of a mouldable floor carpet for a motor vehicle.

Example 3:

A nonwoven sheet of 115 g/m^2 of continuous filaments containing 2 types of filaments, namely 2GT 88 % and 4GT 12 % with counts of 9 and 5 dtex respectively, is produced using the process outlined in Example 1.

1.5 % of Rhodorsil 47V2000 silicone oil (polydimethylsiloxane with a viscosity of 2000 mPa s) relative to the weight of 2GT granules is introduced using a metering pump, before the 2GT extruder.

The sheet is needled on a single-stroke needler at a rate of 60 perforations per cm^2 with Singer 40-Rb gauge needles with a depth of entry of 14 mm. The sheet is then heat-bonded by running over a perforated drum with air passing through at a temperature of 242°C with a contact time of 15 seconds, and is then sized with the aid of a calender with metal rolls heated to 230°C, with a gap between the rolls which are preset so as to set the

density of the heat-bonded sheet to 0.19, that is to say a thickness of 0.6 mm. This sheet exhibits all the tuftability properties, mechanical properties and dimensional stability for making a support for a floor covering with stitched pile as a strip and capable of being printed with patterns which can be matched from one strip to the next.

Example 4:

A nonwoven sheet composed of 125 g/m^2 continuous coaxial filaments, having a core of polyethylene terephthalate and a sheath of polybutylene terephthalate, is produced using the process outlined in Example 1. The core represents 80 % of the mass of the coaxial filament and the sheath 20 %.

Care is taken to introduce 0.8 % of a 47V1200 silicone oil (polydimethylsiloxane with a viscosity of 1200 mPa s) relative to the weight of polyester into the 4GT polymer constituting the sheath, this being done at the nozzle of the extruder using a volumetric metering device. The count of the coextruded filaments is 10 dtex.

The nonwoven sheet is then consolidated under the conditions of Example 3. The heat-bonded support thus obtained has the appearance, mechanical performance, tuftability and stability properties needed for the production of a velvet carpet presented in tile form.

The claims defining the invention are as follows:

1. A nonwoven of continuous melt-extruded polyester filaments, at least 50% by weight of which contain 0.5 to 3% by weight of a polyorganosiloxane oil introduced into the polyester during the melt extrusion.

2. A nonwoven according to Claim 1 in which the said filaments contain 0.6 to 1.5% by weight of the polyorganosiloxane oil.

3. A nonwoven according to Claim 1 or 2 in which the polyorganosiloxane oil used is a polydiorganosiloxane oil with a molecular weight of between 1000 and 250,000, corresponding to a dynamic viscosity of 50 to 10,000 mPa s.

4. A nonwoven according to Claim 3 in which the polydiorganosiloxane oil used has a molecular weight between 7,500 and 70,000.

5. A nonwoven according to any one of Claims 1 to 4, in which the polyester filaments consist of more than 50% of polyethylene terephthalate filaments containing 0.6 to 3% of polyorganosiloxane oil and of less than 50% of polybutylene terephthalate filaments.

6. A nonwoven according to Claim 1 substantially as described in any one of Examples 1 to 4.

7. Process for the manufacture of a nonwoven as claimed in Claim 1, which comprises introducing, into a

polyester melt in an extruder, by injection into the nozzle of the extruder after the melting of the polyester or by injection into the body of the extruder during the melting of the polyester, a polyorganosiloxane oil in a proportion of 0.5 to 3% by weight relative to the polyester, and extruding the polymer melt to produce the filaments by passage through orifices of a die.

8. Process according to Claim 7 in which the proportion of the polyorganosiloxane oil is 0.6 to 1.5% by weight relative to the polyester.

9. Process according to Claim 7 or 8, in which a plurality of polyesters are separately extruded and the polyorganosiloxane oil is introduced into at least one of them.

10. Process according to Claim 7 substantially as hereinbefore described.

11. A nonwoven when produced by the process of any of Claims 7 to 10.

12. A floor covering with stitched pile comprising, as a support, a nonwoven as claimed in any one of Claims 1 to 6 or 11.

~~13. The hereinbefore described invention in all its new and useful aspects.~~

DATED this EIGHTH day of FEBRUARY 1991

Freudenberg Spunweb S.A.

by DAVIES & COLLISON
Patent Attorneys for the Applicants