

Description

STERILIZATION POUCH COMPRISING A POROUS NONWOVEN FABRIC

5 **Technical field**

The present invention relates to nonwoven materials used in the manufacture of sterilization items, and more particularly but not exclusively in the manufacture of preformed sterile barrier systems, notably sterilization pouches.

10 **Prior art**

To sterilize medical equipment (reusable or disposable), it is known practice to use preformed sterile barrier systems (pre-SBS) that meet certain standards, notably including ISO 11607 (Packaging for terminally sterilized medical devices) and NF EN 868, notably part 9, which describes the requirements and test methods for uncoated polyolefin-based nonwoven materials. These preformed sterile barrier systems may consist of a polymer film, notably a
15 multilayer film, typically based on polypropylene (PP) and polyethylene terephthalate (PET), and a porous nonwoven material, and produced by heat-sealing. One side of the pre-SBS is left unsealed so as to allow the insertion of the instrument to be sterilized.

Once the instrument has been inserted, the preformed sterile barrier system can be heat-
20 sealed at its opening, and the whole assembly placed in a sterilization chamber such as an autoclave or sterilization chamber, where a sterilizing agent is injected. This agent may be steam, formaldehyde, hydrogen peroxide (notably when excited with an electromagnetic field, in which case this is referred to as gas-plasma sterilization), or ethylene oxide. Sterilization can also be achieved via irradiation.

25 The porosity of the nonwoven fabric thus allows the sterilizing agent to penetrate the system to sterilize its contents.

When the instrument is to be used, medical staff open the system by separating the nonwoven fabric from the polymer film; during this operation, it is desirable that no fibers from the nonwoven fabric are lifted, in order to guarantee the asepsis of the packaging system.
30 Indeed, lifting fibers can lead to a risk of contamination of the surgical field.

The sealing strength between the polymer film and the nonwoven fabric must therefore be sufficient to guarantee the integrity of the sterile contents of the system and withstand the pressurizations during the sterilization cycles, but not too high so as to avoid the risk of tearing and fiber release when opened.

Patent application GB 2 449 418 describes the use of metallocene polypropylene (PP) for manufacturing a porous nonwoven fabric that can be used for making sterilizable packaging. No fillers other than fluorinated compounds, antistatic agents, colorants or antimicrobials are envisaged.

5 Patent application US 2018/0327948 A1 describes a stretchable SMMS-type nonwoven article comprising spunbonded layers containing metallocene polypropylene mixed with other polymers such as PE, PC and 0.25% by weight of TiO₂. Such a material is not suitable for making sterilization pouches, as it does not meet the standard NF EN 868-9, notably in terms of mechanical strength and aseptic opening. Said standard specifies in section 2.2.1 a tensile
10 strength in the machine direction (MD) of at least 2.5 kN/m and in the cross direction (CD) of at least 1.5 kN/m.

Patent application US 2022/0195645 A1 describes a nonwoven fabric comprising two-component fibers comprising polymers that may include metallocene PP, with a filler content of several percent by mass of CaCO₃ and TiO₂.

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Description of the invention

There is a need to further improve the porous nonwoven fabrics used in the manufacture of sterilization articles, notably preformed sterile barrier systems, in order to facilitate their opening by peeling while ensuring sufficient sealing strength, good quality of the imprint left
20 on the nonwoven fabric, and no tearing to guarantee asepsis.

Summary of the invention

The invention is directed toward meeting this need and achieves this by proposing a porous nonwoven fabric intended for manufacturing sterilization articles, notably preformed
25 sterile barrier systems, comprising fibers formed at least partially from metallocene polypropylene, and between 0.1% and 1.4% and preferably between 0.1% and 0.8% of titanium dioxide by mass relative to the total weight of the nonwoven fabric.

By virtue of the invention, the sealing forces can be adjusted. This improves the peelability and the user experience when opening the sterile barrier system. The opening forces
30 are constant and moderate, ensuring easy opening, no tearing of the nonwoven fabric, and maintenance of asepsis. The quality of the imprint formed on the nonwoven fabric when the sterile barrier system is opened is also improved (the imprint can be more uniform, whiter, without transparent areas (i.e. without transparency), and does not have any raised fibers).

Surprisingly, the claimed content makes it possible to adapt and improve the sealing performance, while at the same time maintaining good mechanical performance and preserving sterility.

In fact, a titanium dioxide content of greater than 1.4% by mass leads to a significant decrease in tear strength in the machine direction, and also a decrease in tensile strength in both the machine and cross directions.

A content of less than 0.1% leads to a transparent imprint on the porous nonwoven fabric, notably at sealing temperatures of between 150°C and 160°C. Furthermore, the sealing forces are not adjusted, which can cause tears in the nonwoven fabric or cause fibers to lift when the sterile barrier system is opened.

The nonwoven fabric according to the invention complies with the standard ISO 11607 and ISO 10993. It also complies with the standard DIN 58953-6 (the latter describes tests for the microbial barrier properties of medical devices to be sterilized), ASTM F2101 (measurement of bacterial filtration efficiency, BFE), and ASTM F1608 (measurement of the ability of a porous material to prevent the penetration of bacteria). According to the latter method, a completely impermeable control sample (bacterial penetration = 0) is exposed to one million cfu (colony-forming units). The number 10^6 has a log value of 6. If a sample subjected to the same test as the control sample allows the penetration of 10^1 cfu ($\log 10 = 1$), its LRV (Log Reduction Value) is 5 ($6 - 1 = 5$). Thus, the higher the LRV value, the more resistant the porous material will be to bacteria and microorganisms.

When the nonwoven fabric is used to make a sterilization pouch, the nonwoven material must meet the requirements of standard NF EN 868-9. In particular, the pouch must have an aseptic opening and the nonwoven fabric must have a tensile strength in the machine direction MD (measured according to EN ISO 1924-2) of at least 2.5 kN/m and in the cross direction CD (measured according to EN ISO 1924-2) of at least 1.5 kN/m according to this standard (section 2.2.1) for polypropylene-based materials.

According to one or more advantageous features of the invention, considered individually or in combination:

- the nonwoven fabric comprises between 0.1% and 1% by mass of titanium dioxide, notably more than 0.25% by mass of titanium dioxide, better still between 0.4% and 1% by mass of titanium dioxide, even better still between 0.4% and 0.6% by mass of titanium dioxide,
- the nonwoven fabric is of the spunbonded type,

the nonwoven fabric does not contain any mineral filler other than titanium dioxide, notably in an amount greater than or equal to 0.1% by mass, notably strictly less than 0.1% by mass, or even contains only titanium dioxide as a mineral filler, the absence of calcium carbonate as a filler being advantageous in that it avoids the creation of false positives in biocompatibility;

- the titanium dioxide has a number-average size of between 100 and 1000 nm,
- the fibers are formed entirely, with regard to the polymer material(s) of which they are composed, from metallocene polypropylene.

According to another of its aspects, the invention also relates to an article, notably a preformed sterile barrier system, notably a sterilization pouch, surgical drape, protective garment or pouch for transporting an active principle, comprising a nonwoven fabric according to the invention, as defined above.

Preferably, the article is a sterilization pouch.

According to another of its aspects, the invention also relates to a process for closing an article according to the invention, in which the nonwoven fabric is heat-sealed to a thermoplastic support.

The sealing is preferably performed at a temperature of between 135°C and 165°C, better still between 150°C and 160°C.

Brief description of the drawings

The invention may be understood more clearly on reading the detailed description that follows, of nonlimiting examples of its implementation, and on examining the attached drawing, in which:

- [Fig. 1] shows a front view, nonwoven side, of an example of a sterilization pouch according to the invention.
- [Fig. 2] is a partial schematic cross-section of the pouch,
- [Fig. 3] is a table illustrating the mechanical characteristics of the nonwovens obtained for different titanium dioxide contents,
- [Fig. 4] is a table illustrating the measured sealing forces and other findings for different sealing temperatures and different titanium dioxide contents.

Detailed description

Figures 1 and 2 show an example of a sterilization pouch 1 according to the invention, comprising a porous nonwoven fabric 2 heat-sealed to a support film 3 made of a non-porous thermoplastic material based on polypropylene PP and PET.

Figure 1 shows the pouch sealed along a weld line 4 extending along only three sides, thus providing an opening 5 for inserting an instrument to be sterilized. The nonwoven fabric is heat-sealed to the support along a closure line 6 after the instrument has been inserted, and the pouch is then sterilized.

On the side opposite the opening 5, the pouch 1 has an area 7 where the nonwoven fabric covers the support without being sealed thereto, so that the user can grasp them with their hands to pull them apart when opening the pouch by peeling it.

This sterile barrier system can be sterilized using various methods:

- with moist heat: the sterilizing agent is steam (temperature: 121°C–134°C for 18–30 minutes, under pressure),
- with ethylene oxide: temperature between 30 and 60°C, with a relative humidity above 30% for 16-18 hours with an ethylene oxide concentration of 200-800 mg/L). Ethylene oxide is a toxic agent, so a desorption period must be observed after sterilization,
- using ionizing radiation (gamma rays, beta sterilization or an electron beam),
- with formaldehyde: formaldehyde can be used in gaseous form,
- with gas plasma: use of hydrogen peroxide (H₂O₂) in gas form diffused into the chamber and converted into plasma by an electromagnetic field. This generates ions, free electrons, and numerous free radicals with very strong bactericidal activity. This method allows sterilization at low temperatures and does not require desorption.

Metallocene PP

Within the meaning of the invention, the term "polypropylene" covers a propylene homopolymer, propylene copolymers obtained by copolymerization of propylene with at least one alpha-olefin comonomer, and propylene homopolymer or propylene copolymer derivatives that are functionalized, notably by grafting a reactive function following interaction with maleic anhydride or acrylic acid, for instance maleated polypropylene. A copolymer may be block or random.

Polypropylene (regardless of its form) may be isotactic or syndiotactic.

In the context of the invention, the term “metallocene PP” refers to polypropylene obtained by metallocene catalysis.

Polypropylene is produced using metallocene catalysis rather than via the Ziegler-Natta process (the use of this type of catalysis for the production of polyolefins is well known to those skilled in the art, as described in patents US 5 571 619, US 5 322 728 and US 5 472 775).

The nonwoven fabric is preferably made from metallocene polypropylene (PP) in accordance with the teachings of patent GB 2 449 418.

Metallocene PP generally has a fairly narrow molecular weight distribution (MWD), preferably with an MWD value of less than 3, notably less than 2.5, and allows the production of fibers with a mean diameter of less than 25 microns, notably in the range of 10-20 microns.

The nonwoven fabric obtained is formed by a technique known as spunbonding, which is known *per se*. The formation of metallocene polypropylene fibers can be performed as described in patent application GB 2 449 418 (this process is also described in US 3 821 062, US 3 563 838, US 2021/0301425, and US 2019/145032). Titanium dioxide (TiO₂) is incorporated during the production of the polypropylene fibers by mixing it with the metallocene PP, notably by dry blending. The titanium dioxide is preferably in the form of a masterbatch.

The PP used may notably be Metocene HM562S with a melt flow index of 30 g/10 min measured at 230°C under a weight of 2.16 kg (according to the standard ASTM D1238).

According to the invention, the porous nonwoven fabric may undergo a calendering step. During this calendering step, several parameters may vary, such as temperature (typically about 130°C and 160°C), speed (typically greater than 10 m/min), and pressure (typically greater than 30 bar).

The mass content of titanium dioxide in the nonwoven fabric is between 0.1% and 1.4%, preferably about 0.5%.

Such a content affords advantageous properties for the manufacture of the pouch, as will be detailed with reference to the tables in Figures 3 and 4.

The fibers of the nonwoven fabric may have a mean diameter ranging from 10 to 25 microns.

The titanium dioxide may have a mean size ranging from 100 nm to 1 µm.

Comparative tests

Various samples were produced using porous nonwoven fabric composed of metallocene PP (Metocene HM562S) and TiO_2 (mean diameter of 300 nanometers), and a 12 μm laminated polymer film PET 12 μ /PP.

5 The nonwoven fabric has an average basis weight of 93 g/m^2 (measured according to the standard ISO 536).

Strips measuring 5 cm x 15 cm were prepared for each sample. The PP side of the PET/PP film was heat-sealed to the porous nonwoven fabric.

Film (PET/PP) and porous nonwoven fabric pairs were heat-sealed using a laboratory heat sealer, with the hot jaw pressed against the PET side. Different sealing temperatures were
10 tested: 150, 155, and 160°C, at a constant pressure of 5 bar, for a duration of 0.3 second.

The sealing forces were measured on 10 samples using a tensile testing machine in accordance with the standard ASTM F88/F88M-2021 (technique B).

Tensile tests are performed on a tensile testing machine according to the standard ASTM D882-18 on 10 samples.

15 Tear tests are performed on 10 samples using a tensile testing machine according to the standard ASTM D1938-19.

These film (PET/PP) and porous nonwoven fabric pairs, sealed on a laboratory heat sealer, also made it possible to study the quality of the imprint on opening.

Pouches such as those shown in Figure 1 were also produced on an FFS ("Form-Fill-Seal") machine. Three sealing temperatures were used: 150, 155, and 160°C, and the speed was
20 set at 15 pouches per minute. These pouches were used to evaluate the feel on opening and the quality of the imprint.

The table in Figure 3 shows that the tear strength is significantly reduced for tests with contents of 1.5% or more, and that it reaches a relative maximum at 0.5%.

25 The table in Figure 4 compares the sealing forces for different sealing temperatures and different titanium dioxide mass contents.

The results are also assessed in the form of comments in the last two columns of the table in Figure 4.

For a content of less than 0.1%, a lack of whiteness in the imprint can be observed, as
30 well as a transparent appearance, for sealing temperatures of 150, 155, and 160°C.

There is no "transparency" after peeling, a good sensation upon opening, while maintaining sterility, for titanium dioxide contents of 0.19%, 0.27%, 0.5% and 0.8%.

The sensation on opening is poor at a content of 1.5%.

The best results are obtained for titanium dioxide contents of 0.27% and 0.5% by weight, at 155°C and 160°C. In fact, a very good sensation on opening is observed, as well as a homogeneous and white imprint, while preserving the sterility, and also very good mechanical performance (notably tear strength).

5 Needless to say, the invention is not limited to the examples just described.

For example, the nonwoven fabric according to the invention can be used in the manufacture of preformed sterile barrier systems such as pouches, breathable tape bags, or hospital packaging sheaths, among other items.

10 The metallocene PP can be mixed with at least one other thermoplastic, for example grafted or non-grafted polyethylene (low density or linear low density), or polybutene, among other possibilities. The proportion of the other polymer(s) in the mixture is chosen to maintain the desired properties.

Claims

1. A sterilization pouch comprising a porous nonwoven fabric comprising fibers formed at least partially from metallocene polypropylene, and between 0.1% and 1.4% of titanium dioxide by mass relative to the total weight of the nonwoven fabric, the nonwoven fabric being free of any mineral filler other than titanium dioxide in an amount greater than or equal to 0.1% by mass.
2. The pouch as claimed in claim 1, comprising between 0.1% and 1% by mass of titanium dioxide.
3. The pouch as claimed in claim 1 or 2, comprising between 0.4% and 1% by mass of titanium dioxide.
4. The pouch as claimed in any one of claims 1 to 3, comprising between 0.4% and 0.6% by mass of titanium dioxide.
5. The pouch as claimed in any one of the preceding claims, being of the spunbonded type.
6. The pouch as claimed in any one of the preceding claims, the titanium dioxide having a number-average size of between 100 and 1000 nm.
7. The pouch as claimed in any one of the preceding claims, the fibers being formed entirely, with regard to the polymer material(s) constituting them, from metallocene polypropylene.
8. The pouch as claimed in any one of the preceding claims, the nonwoven fabric having a tensile strength in the machine direction MD (measured according to EN ISO 1924-2) of at least 2.5 kN/m and in the cross direction CD (measured according to EN ISO 1924-2) of at least 1.5 kN/m according to this standard (section 2.2.1).
9. A process for closing a pouch as claimed in any one of the preceding claims, wherein the nonwoven fabric is heat-sealed to a thermoplastic support.

10. The process as claimed in claim 9, the sealing being performed at a temperature of between 135°C and 165°C, better still between 150°C and 160°C.