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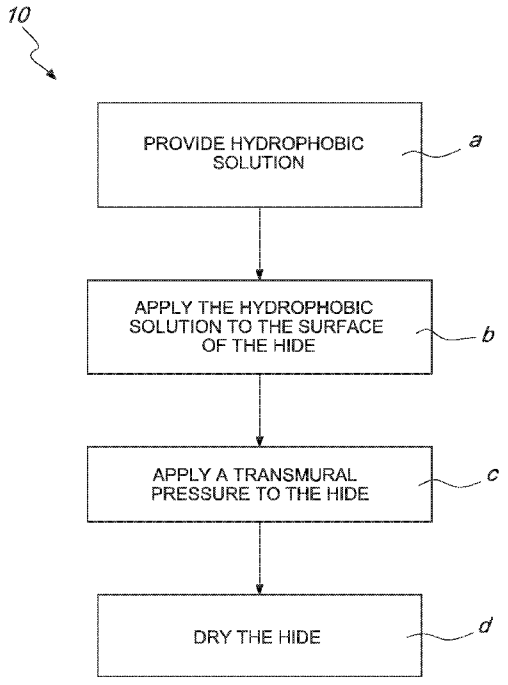

Fig. 1

(57) Abstract: A functionalizing treatment method for hides and the like that have a surface and an internal substrate that comprises interconnected fibers, which comprises a deposition step (10), in which a plurality of functionalizing elements comprising nanomolecules and/or nanoparticles are inserted into the internal substrate of the hide to be treated, between the individual fibers, so as to coat and functionalize the individual fibers.

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FUNCTIONALIZING TREATMENT METHOD FOR HIDES

The present invention relates to a functionalizing treatment method for hides and the like, which is useful and practical particularly, but not exclusively, in the field of the tanning industry.

In more detail, the treatment method according to the present invention is useful and practical in order to obtain in the hide, for example, one or more of the following functional enhancements: hydrophobization, increasing the mechanical properties, conferring tear-resistant and scratch-proof properties, electrification and/or integration of responsive electrical circuits.

Nowadays, various treatment methods for hides are known, which include the various techniques of tanning, retanning and finishing.

Among the known treatments, hydrophobization treatments are particularly important.

As is known, the wettability of a surface depends substantially on 2 factors: the surface energy and the roughness of the surface. Therefore the hydrophobization treatments of the known type are aimed at modifying these two factors.

A first known type of method for increasing the hydrophobicity of a hide or fabric consists of reducing the surface energy by way of chemical modifications.

A second known method for increasing the hydrophobicity of a hide or fabric, which can be used in addition to the chemically controlling the surface energy, consists of modifying the roughness of the surface by way of physical techniques like imprinting, abrasion or by way of deposition of particles.

These methods of the known type clearly have the limitation of being designed to and having their effect only on the surface portion of the fabrics and of the hides, and therefore the effect is normally limited over time

owing to the inevitable deterioration of the surface layer of the treated hide or fabric.

In general, the functionalizing treatments of the known type mainly affect the surface layer of the hide or fabric and therefore they have the same limitations on duration, as well as limited performance levels.

The aim of the present invention is to overcome the limitations of the known art described above, by devising a functionalizing treatment method for hides and the like that makes it possible to obtain a functionalization that lasts longer over time.

Within this aim, an object of the present invention is to provide a functionalizing treatment method for hides and the like that makes it possible to obtain a functionalization with improved performance levels compared to the background art.

Another object of the invention consists of conceiving of a functionalizing treatment method for hides and the like that can be inserted in the standard production lines and processes used currently in the tanning sector.

Another object of the invention consists of conceiving of a functionalizing treatment method for hides and the like that is highly reliable, easily and practically implemented, and low cost when compared to the known art.

This aim and these and other objects which will become better apparent hereinafter are achieved by a functionalizing treatment method for hides and the like that have a surface and an internal substrate that comprises interconnected fibers, characterized in that it comprises a deposition step, in which a plurality of functionalizing elements comprising nanomolecules and/or nanoparticles are inserted into the internal substrate of the hide to be treated, between the individual fibers, so as to coat and functionalize the individual fibers.

Further characteristics and advantages of the invention will become

better apparent from the description of some preferred, but not exclusive, embodiments of a functionalizing treatment method for hides and the like, which are illustrated by way of non-limiting example with the aid of the single accompanying Figure 1 which is a flow diagram of a possible embodiment of the method according to the invention.

The method consists of a functionalizing treatment for hides or the like, that is to say a treatment that provides the hide with additional functions and/or properties, for example, to provide one or more of the following functional enhancements: hydrophobization, increasing the mechanical properties, conferring tear-resistant and scratch-proof properties, electrification and/or integration of responsive electrical circuits.

The term hide or the like (hereinafter also referred to simply as "hide") means, entirely generally, any material of the type of hide, cloth or fabric that comprises an outer surface and an internal substrate (or internal thickness) that comprises mutually interconnected fibers. In particular, in animal hide, the internal substrate is constituted by the dermis and the interconnected fibers are constituted by collagen fibrils.

According to the invention, the method comprises a deposition step 10, in which a plurality of functionalizing elements comprising at least nanomolecules and/or nanoparticles are inserted into the internal substrate of the hide to be treated, between the individual fibers, so as to coat and functionalize the individual fibers. In practice, according to the invention, at least some of the individual fibers are completely coated and individually functionalized.

In this manner, for animal hide, it is possible to modify the properties of the individual collagen fibril and therefore change/alter the properties of the final structure but without modifying the nature of the animal dermis.

Specifically the functionalizations developed with the method in question extend inside the entire dermal thickness because they make use of micro and nano molecules/particles that can interpenetrate in the individual

collagen fibers.

The term "nanomolecules" or "nano molecules" (or "nanoparticles" or "nano particles") means colloidal elements, or particles formed from atomic or molecular aggregates, with an average diameter comprised between 1 and 1000 nm, differently from "micromolecules" or "micro molecules" (or microparticles or "micro molecules") which are molecules (or particles) with micrometric dimensions, comprised between 3 and 800 μm.

In the preferred embodiments, in the deposition step, the nanomolecules or nanoparticles are comprised in a suspension or in a solution, preferably an aqueous solution or in a solvent.

Preferably the functionalizing elements also comprise macromolecules, even more preferably polymeric macromolecules.

Advantageously, the deposition step 10 can be carried out at different times in the entire process of treating the skins, which is broken down into tanning, retanning and finishing steps.

Optionally, the deposition step 10 is preceded by the steps of dehydrating the hide to be treated, and subsequently drying the hide, so as to have a substrate with high absorbent capacities.

In some embodiments adapted to hydrophobize the hide, in the deposition step, the functionalizing elements are comprised in a hydrophobic solution and comprise molecules of one or more silanes (SinH2n+2) and of one or more polydimethylsiloxanes ((C2H6OSi)n).

In more detail, again in the embodiments adapted to hydrophobize the hide, the functionalizing elements, in the deposition step, are applied to the hide and then conveyed inside the internal substrate by way of one or more of the following methods: suction, immersion and mechanical agitation spraying, vaporization, formation of stable mists.

In some of these embodiments, the functionalizing elements comprise one or more silicone polymers constituted by a prepolymer base and by a curing agent which facilitates the polymerization of the one or more silanes.

Preferably, such prepolymer base and such curing agent are present in a ratio comprised between 10:0.2 and 10:5 (that is to say, the base/curing agent ratio is comprised between 10:0.2 and 10:5).

Advantageously, in some of these embodiments, in the hydrophobic solution there are also:

- one or more fluorinating agents, each one of which comprises at least one fluorine group in the side chain; and

- one or more agents adapted to facilitate the polymerization of at least part of the functionalizing elements and their radication to the fibers of the internal substrate of the hide to be treated (in particular to the collagen fibers).

Optionally, the above mentioned hydrophobic solution also comprises silicon nanoparticles, which, by creating a roughness both on the surface and around the collagen fibers, favor the formation of the "lotus" effect that enables water droplets to slide on the surface of articles made of hide without being absorbed by the collagen fibers that constitute the animal dermis.

In a particular preferred embodiment adapted to hydrophobize the hide, shown in Figure 1, the deposition step 10 comprises the steps of:

a. providing a hydrophobic solution which comprises a uniform mixture of the functionalizing elements, comprising microparticles, nanoparticles and polymeric molecules, in an aqueous solution or in a solvent (for example isopropanol, ethanol, acetone);

b. applying the hydrophobic solution to the surface of the hide to be treated, preferably by way of spraying and/or by way of formation of stable mists that uniformly cover the surface of the hide;

c. applying a transmural pressure variation ΔP to the hide to be treated, so as to induce the previously applied hydrophobic solution to penetrate inside the internal substrate, so that the individual fibers will be functionalized.

Note that the functionalizing elements in this manner penetrate uniformly into the deepest layers of the internal substrate of the hide.

Optionally, after the above mentioned step c., there is a subsequent drying step d, in which the hide already subjected to the deposition step is dried inside an environment (for example a closed chamber) at a controlled temperature comprised between 20°C and 80°C, for a time comprised between 1 hour and 3 days, in order to allow the evaporation of any solvent, the complete polymerization of the silanes and the radication of the nanoparticles and of the micromolecules to the individual collagen fibers.

In the method according to the invention, the deposition step can be repeated multiple times and preferably for a number of cycles comprised between 1 and 30.

The number of cycles to be applied is correlated to the starting concentration of the hydrophobic solution, proceeding with dilutions thereof. The number of cycles can be increased further.

The concentration of the functionalizing elements in the hydrophobic solution can vary from 0.1% to 99.9%. The concentration of the functionalizing elements in the hydrophobic solution and the number of cycles can vary according to the degree of hydrorepellence that it is desired to confer on the article, and/or the origin animal of the hide, and/or the tanning, retanning and finishing treatments that have been carried out on the hide, or the stage of processing thereof.

The hide treated through the embodiments of the method, according to the invention, which are adapted to hydrophobize the hide, display angles of contact with water, of advancement and withdrawal, with values higher than 150°C, and therefore they can be considered superhydrophobic.

The superhydrophobicity conferred on articles made of hide subjected to these embodiments of the method, according to the invention, makes it possible to wash the garments in a washing machine without their losing their original color in terms of tone and intensity. Complete washing in a

washing machine furthermore does not result in the loss of the superhydrophobicity conferred on articles with the method according to the invention, because it does not affect only the surface layers of the hide and it is not a covering similar to a finish; in fact, the method according to the invention makes it possible to coat/varnish the individual collagen fibers, and therefore to fully functionalize, from the inside, the animal dermal structure and therefore the entire article.

Electron scanning microscope images show that the functionalization is not limited to only the surface of the grain side and to the surface of the flesh side, but clearly show the presence of fibers perfectly coated/varnished with hydrophobizing agents in the internal portions as well, therefore from both flesh and grain sides up to the central part.

Furthermore, it has been found that the hide subjected to these embodiments of the method can remain for over 48 hours immersed in water without evident penetration of water into the inner layers of the article, a finding that confirms the result of functionalizing from the inside the entire collagenic structure that makes up the animal dermis of articles made of hide.

Other embodiments of the method are adapted to provide the hide with responsive electrical circuits integrated therein.

In these embodiments, the functionalizing elements comprise conducting nanoparticles which, in the above mentioned deposition step, are inserted into predetermined portions of the internal substrate (or along predetermined paths inside the substrate) so as to render such predetermined portions (or such predetermined paths) electrically conducting; in more detail, such portions (or paths) are predetermined so as to create one or more electrical circuits inside the internal substrate.

Preferably, such conducting nanoparticles comprise nanoparticles of silver and/or graphene and/or graphite and/or copper and/or gallium. In some embodiments such conducting nanoparticles comprise nanotubes of

graphene and/or copper.

In more detail, in the deposition step, the conducting nanoparticles are comprised in a conducting ink which comprises an aqueous solution or solvent in which the aforementioned conducting nanoparticles are uniformly dispersed.

Conveniently, in a preliminary step of preparing the conducting ink, the conducting nanoparticles are uniformly dispersed in the aqueous solution or in solvent, preferably by way of ultrasound.

Optionally, in the conducting ink there are also polymers with variable viscosity which make it possible for the functionalizing elements to interpenetrate between the collagen fibers and also enable the radication and coating of those collagen fibers with the conducting ink.

In practice, with these embodiments of the method according to the invention, it is possible to print/transfer conductive circuits inside the animal dermis.

Advantageously, conductive circuits are created which are flexible and fold-resistant.

Note that the circuits are not printed on the surface of the hide, but are transferred and placed inside the three-dimensional structure of the collagen that constitutes the animal dermis. More specifically, the method according to the invention makes it possible to "write" the circuits precisely and locate them around the collagen fibers that constitute the animal dermis.

Using the method just described, it is possible in fact to make the conductive inks interpenetrate between the fibers or coat the individual collagen fibers. In this manner it is possible to write or print circuits precisely and locate them in the area of interest alone.

Ultimately, it is possible not only to write or print circuits inside articles made of hide, but also to convert the collagen fibers, coated by the conducting ink, to genuine conducting wires.

Advantageously, the circuits are integrated inside the article made of

hide so that they are imperceptible to the sight and touch of the user.

Advantageously, the hide or fabrics thus treated combine the natural advantages and comfort offered by natural hide, i.e. they are soft and pleasing to the touch and they are breathable, with new functions that until now have not been associated with these types of material.

Optionally, the method according to the invention makes it possible to integrate inside the hide, or fabrics, flexible electronics and electrical/optical devices, sensors, wires, or LEDs, which are inserted directly around and inside the collagen fibers that make up the structure of the animal dermis, sensors for monitoring the vital signs of the wearer, etc.

Fabrics thus treated can also be used for releasing moisturizing lotions or medicines for the skin, and/or they can be configured to control body temperature, or muscle vibration during athletic activities, and they can potentially change light and color and display images and video.

The hide or fabric thus functionalized, by way of any of the embodiments just described, can then be tanned and finished using the standard processes used daily in tanning companies. The substrate on which the dehydration and drying process is carried out forms part of the raw materials conventionally used in the tanning industry, which are typically pickled.

In practice it has been found that the functionalizing treatment method for hides and the like according to the present invention achieves the intended aim and objects in that it makes it possible to obtain a functionalization that lasts longer over time.

Another advantage of the method according to the invention consists in that it obtains a functionalization with improved performance levels compared to the background art.

Another advantage of the method according to the invention consists in that it can be inserted in the standard production lines and processes used currently in the tanning sector.

Another advantage of the method according to the invention consists in that it is highly reliable, easily and practically implemented, and low cost when compared to the known art.

The functionalizing treatment method for hides and the like, thus conceived, is susceptible of numerous modifications and variations, all of which are within the scope of the appended claims.

Moreover, all the details may be substituted by other, technically equivalent elements.

In practice the materials employed, and the contingent dimensions and shapes, may be any according to requirements and to the state of the art.

The disclosures in Italian Patent Application No. 102019000017942 from which this application claims priority are incorporated herein by reference.

Where the technical features mentioned in any claim are followed by reference numerals and/or signs, those reference numerals and/or signs have been included for the sole purpose of increasing the intelligibility of the claims and accordingly, such reference numerals and/or signs do not have any limiting effect on the interpretation of each element identified by way of example by such reference numerals and/or signs.

CLAIMS

1. A functionalizing treatment method for hides and the like that have a surface and an internal substrate that comprises interconnected fibers, characterized in that it comprises a deposition step (10), in which a plurality of functionalizing elements comprising nanomolecules and/or nanoparticles are inserted into the internal substrate of the hide to be treated, between the individual fibers, so as to coat and functionalize the individual fibers.

2. The method according to claim 1, characterized in that in said deposition step (10) said nanomolecules or nanoparticles are comprised in a suspension or in a solution.

3. The method according to one or more of the preceding claims, characterized in that said functionalizing elements also comprise macromolecules, preferably polymeric macromolecules.

4. The method according to one or more of the preceding claims, characterized in that said functionalizing elements, in said deposition step (10), are conveyed into said internal substrate by way of one or more of the following methods:

- suction;
- immersion and mechanical agitation;
- spraying;
- vaporization;
- formation of stable mists.

5. The method according to one or more of the preceding claims, characterized in that in said deposition step (10) said functionalizing elements are comprised in a hydrophobic solution and comprise molecules of one or more silanes (Si_nH_{2n+2}) and of one or more polydimethylsiloxanes ($(C_2H_6OSi)_n$).

6. The method according to claim 5, characterized in that said functionalizing elements comprise one or more silicone polymers constituted by a prepolymer base and by a curing agent which facilitates the

polymerization of said one or more silanes.

7. The method according to claim 6, characterized in that said prepolymer base and said curing agent are present in a ratio comprised between 10:0.2 and 10:5.

8. The method according to one or more of claims 5 to 7, characterized in that said hydrophobic solution also comprises:

- one or more fluorinating agents, each one of which comprises at least one fluorine group in the side chain; and

- one or more agents adapted to facilitate the polymerization of at least part of said functionalizing elements and their radication to said fibers of the internal substrate of the hide to be treated.

9. The method according to one or more of claims 5 to 8, characterized in that said hydrophobic solution also comprises silicon nanoparticles.

10. The method according to one or more of the preceding claims, characterized in that said deposition step comprises the steps of:

a. providing a hydrophobic solution which comprises a uniform mixture of said functionalizing elements, comprising microparticles, nanoparticles and polymeric molecules, in an aqueous solution or in a solvent;

b. applying said hydrophobic solution to the surface of the hide to be treated;

c. applying a transmural pressure variation to the hide to be treated, so as to induce said previously applied hydrophobic solution to penetrate inside said internal substrate.

11. The method according to claim 10, characterized in that it comprises a subsequent drying step (d), in which the hide already subjected to said deposition step is dried inside an environment at a controlled temperature comprised between 20°C and 80°C, for a time comprised between 1 hour and 3 days.

12. The method according to one or more of the preceding claims, characterized in that said functionalizing elements comprise conducting nanoparticles, which in said deposition step are inserted into predetermined portions of said internal substrate so as to render said predetermined portions electrically conducting; said portions being predetermined so as to create one or more electrical circuits inside said internal substrate.

13. The method according to claim 12, characterized in that said conducting nanoparticles comprise nanoparticles of silver and/or graphene and/or graphite and/or copper and/or gallium.

14. The method according to claim 12 or 13, characterized in that in said deposition step said conducting nanoparticles are comprised in a conducting ink which comprises an aqueous solution or solvent in which said conducting nanoparticles are uniformly dispersed.

15. The method according to claim 14, characterized in that it comprises a preliminary step of preparing said conducting ink, in which said conducting nanoparticles are uniformly dispersed in said aqueous solution or in solvent by way of ultrasound.

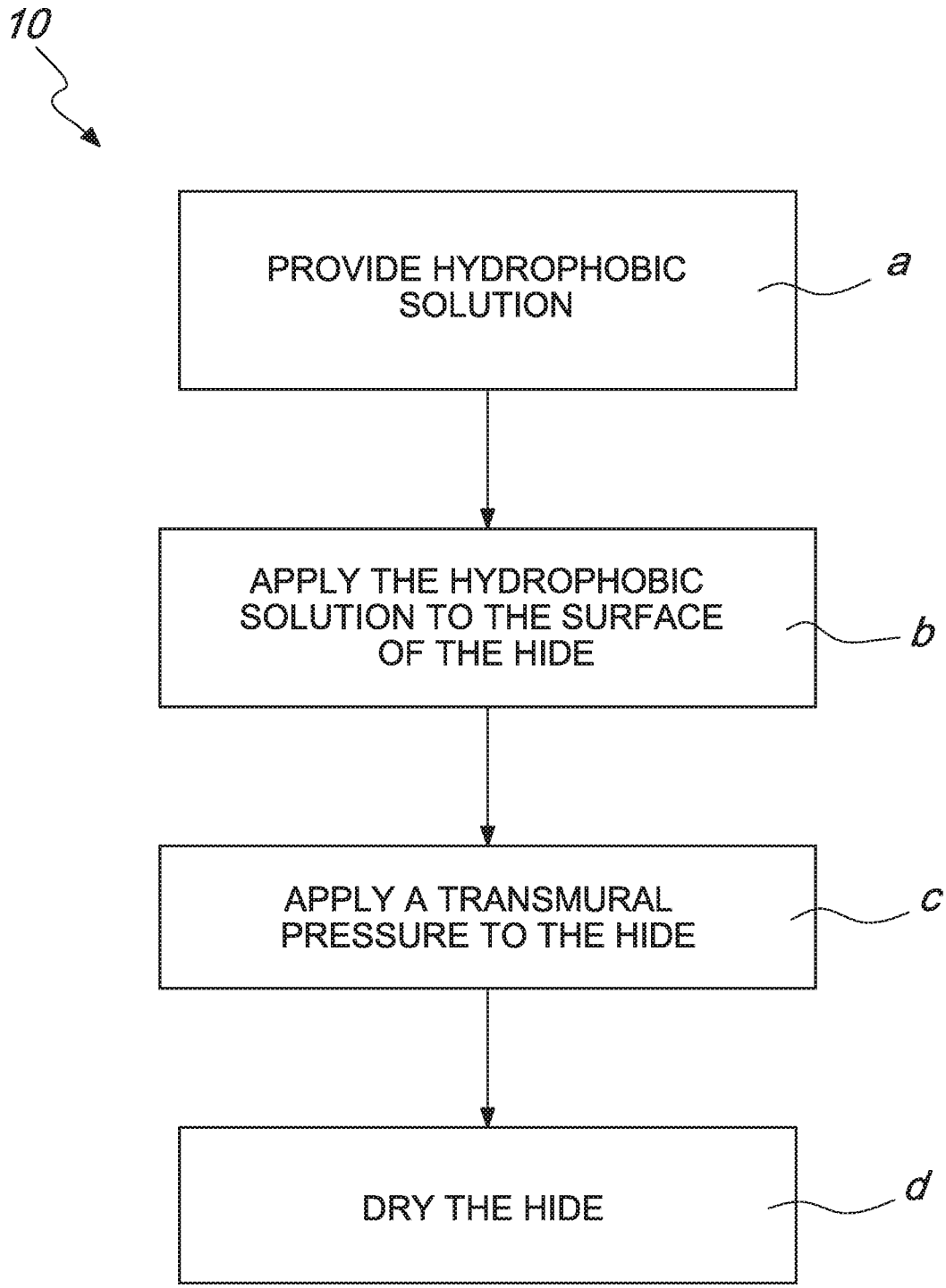
10
PROVIDE HYDROPHOBIC SOLUTION
a
APPLY THE HYDROPHOBIC SOLUTION TO THE SURFACE OF THE HIDE
b
APPLY A TRANSMURAL PRESSURE TO THE HIDE
c
DRY THE HIDE
d

Fig. 1

INTERNATIONAL SEARCH REPORT

International application No
PCT/IB2020/059256

A. CLASSIFICATION OF SUBJECT MATTER
INV. C14C9/00 C14C11/00 C14C13/02 C08G77/04 C08K3/22 C08K3/04
ADD.
According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
C14C C09J C08G C08K

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)
EPO-Internal, WPI Data

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	DATABASE WPI Week 201916 Thomson Scientific, London, GB; AN 2018-A0477V XP002799227, & CN 108 950 104 A (UNIV SHAANXI SCI & TECHNOLOGY) 7 December 2018 (2018-12-07)	1-4
A	abstract	5-15

X	DATABASE WPI Week 201930 Thomson Scientific, London, GB; AN 2019-13254T XP002799228, & CN 109 234 476 A (UNIV SICHUAN) 18 January 2019 (2019-01-18)	1-4
A	abstract	5-15

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[X] Further documents are listed in the continuation of Box C.

[X] See patent family annex.

* Special categories of cited documents :

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier application or patent but published on or after the international filing date

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"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art

"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
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INTERNATIONAL SEARCH REPORT

International application No
PCT/IB2020/059256

C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US 2019/017129 A1 (RENNER MANFRED [DE] ET AL) 17 January 2019 (2019-01-17) paragraphs [0013] - [0035]	1-15
A	EP 3 173 493 A1 (INST NAT DE CERCETARE-DEZVOLTARE PENTRU TEXTILE SI PIELARIE SUCURSALA) 31 May 2017 (2017-05-31) paragraphs [0006] - [0018]	1-15

INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No
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