



(43) International Publication Date
12 November 2015 (12.11.2015)

(51) International Patent Classification:

D04H 11/00 (2006.01) *D06N 3/14* (2006.01)
D04B 21/02 (2006.01) *D04H 1/541* (2012.01)

(21) International Application Number:

PCT/IB2015/053345

(22) International Filing Date:

7 May 2015 (07.05.2015)

(25) Filing Language:

Italian

(26) Publication Language:

English

(30) Priority Data:

MI2014A000847 9 May 2014 (09.05.2014) IT

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(81) Designated States (unless otherwise indicated, for every kind of national protection available): AE, AG, AL, AM, AO, AT, AU, AZ, BA, BB, BG, BH, BN, BR, BW, BY, BZ, CA, CH, CL, CN, CO, CR, CU, CZ, DE, DK, DM, DO, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, GT, HN, HR, HU, ID, IL, IN, IR, IS, JP, KE, KG, KN, KP, KR, KZ, LA, LC, LK, LR, LS, LU, LY, MA, MD, ME, MG, MK, MN, MW, MX, MY, MZ, NA, NG, NI, NO, NZ, OM, PA, PE, PG, PH, PL, PT, QA, RO, RS, RU, RW, SA, SC, SD, SE, SG, SK, SL, SM, ST, SV, SY, TH, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, ZA, ZM, ZW.

(84) Designated States (unless otherwise indicated, for every kind of regional protection available): ARIPO (BW, GH, GM, KE, LR, LS, MW, MZ, NA, RW, SD, SL, ST, SZ, TZ, UG, ZM, ZW), Eurasian (AM, AZ, BY, KG, KZ, RU, TJ, TM), European (AL, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HR, HU, IE, IS, IT, LT, LU, LV, MC, MK, MT, NL, NO, PL, PT, RO, RS, SE, SI, SK, SM, TR), OAPI (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, KM, ML, MR, NE, SN, TD, TG).

Published:

— with international search report (Art. 21(3))

(54) Title: FLOCKED MATERIAL AND PROCESS TO PRODUCE IT

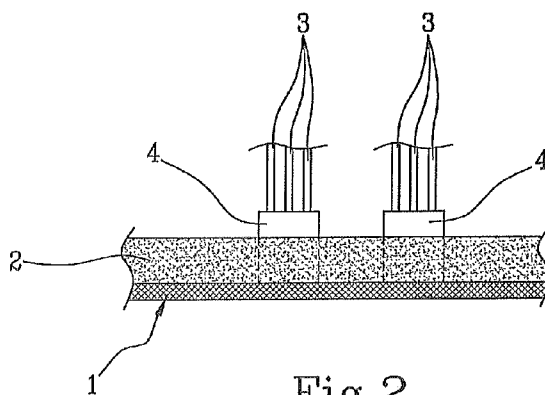


Fig.2

(57) Abstract: The present invention concerns a process for the preparation of a flocked material for coating surfaces and structures such as the interiors of automobiles, objects for interior decorating (walls, sofas, armchairs, etc.), handbags, suitcases or other accessories, covers or cases for weapons, musical instruments or electronic devices, or to make carpets and/or rugs, and that comprises the use of bicomponent fibres of the "island-in-the-sea" type of limited length (microfibres). The process of the invention comprises a step of partial and selective dissolution of the sea component by an alkaline removal agent, which makes it possible to obtain a flocked material characterized by improved resistance to abrasion and a more appealing appearance compared to flocked products of the prior art.

FLOCKED MATERIAL AND PROCESS TO PRODUCE IT

DESCRIPTION

The object of the present invention is a process for the preparation of a flocked material starting from bicomponent fibres of a sea-island type, as well as a flocked material obtained from this process. The flocked material that is obtainable by means of this process can be used for various applications, for example to coat surfaces and structures such as the interiors of motor vehicles, objects for interior decorating (walls, sofas, armchairs, etc.), handbags, suitcases or other accessories, covers or cases for weapons, musical instruments or electronic devices, or to make carpets and/or rugs.

Electrostatic flocking is a particular process that makes it possible to obtain a napped type of effect on various types of surfaces (fabrics, paper, plastic, metal, wood, etc.).

Flocking can be carried out on two-dimensional, rolled materials such as paper or fabrics, as well as on three-dimensional objects (e.g. eyeglass frames, clothes hangers, containers, interior components for automobiles, etc.).

In the flocking process, the synthetic fibres, which are normally already coloured, are applied to a surface that has been previously treated with the application of a specific glue. By means of an electrostatic field, the fibres penetrate the layer of glue, orienting themselves perpendicularly to the surface to be napped.

In order for the fibres to orient themselves in the electrostatic field, they must be cut uniformly, the length thereof normally being related to the size of fibre. The smaller the diameter of the fibre, the shorter its length will be.

Prior to flocking, the fibres require pre-treatment for "activation", which is aimed at predisposing the fibre to electrical conductivity. In general, solutions of metal salts are used; as they coat the fibre surface, they facilitate the orientation of the fibre in the electrostatic field.

Prior to or together with the activation step, a process of dyeing the flock can also be carried out.

The flocking process thus requires the following steps:

- cutting the fibre
- optional dyeing
- activation of the fibres
- application of the glue on the backing layer
- laying the cut fibres in an electrostatic booth, which correctly orients the fibres
- drying and possible reticulation of the glue
- brushing with compressed air to remove excess fibres.

It is extremely difficult to obtain a flocked material realized with ultrafine fibres, given that such fibres would have to be cut to a length that is too short, which is not possible in an industrial process, and they would be extremely unstable and difficult to work with in the activation process. Moreover, ultrafine fibres, which can be classified as microfibres, would encounter more difficulty in penetrating the adhesive backing layer, thereby jeopardizing the abrasion resistance of the flocked material.

However, the problem of using very short fibres for flocking surfaces has already been addressed in the sector. In fact, flocked materials prepared starting from bicomponent fibres of the sea-island type that are limited in length and utilizing an electrostatic flocking process as described above are already known in the sector. Following the flocking of the fibres on the substrate and after the glue has dried, the sea component of the sea-island fibres is normally removed by means of the use of solvents such as trichloroethylene or basic solutions.

For example, examples 15 and 16 in patent no. GB1300268 discloses the preparation of a flocked material consisting of a base made of nylon taffeta coated with a polyurethane adhesive, on which "islands-in-a-sea" type of composite fibres are flocked by means of an electrostatic process; the island component of the composite fibres is nylon 6,6 and the sea component is polystyrene. Prior to the electrostatic process, the fibres are cut at a length of 3 mm and pre-treated with sodium silicate and ammonium chloride. The flocked taffeta is dried and then immersed in a bath of trichloroethylene at a temperature of 50-60°C to dissolve the sea component. Lastly, it is washed with methanol and left to dry.

US patent no. 4574018 discloses a process for preparing a flocked material in which short sea-island bicomponent fibres are flocked by means of an electrostatic procedure, over a base of various type covered with an adhesive (e.g. a polyurethane adhesive). After reticulation of the adhesive at high temperature, the sea component is removed partially by means of immersion in trichloroethylene or in a 3% NaOH solution.

The issue of the workability of microfibres to create flocked materials has been addressed in the prior art through the use of "sea-island" types of fibres. The fibres are activated and laid on the backing layer prior to removal of the sea component.

However, the processes of the prior art have a major drawback concerning the sea component removal step. In fact, the Applicant has found that when using solvents similar to those known in the prior art, for example a basic solution of NaOH, the sea component is completely removed, that is, even in the portion underlying the layer of adhesive. In this regard, see Figure 1A, which shows the results of a removal step, similar to those applied in the prior art, using solutions containing a solvent or even basic solutions. The number 1 indicates the backing layer for the flocked fibres and that can be of various types, whereas

the number 2 indicates the layer of adhesive (of various types) that is applied to the backing layer 1 prior to flocking. During the electrostatic flocking procedure, the sea-island fibres (number 3 indicates the island component and number 4 indicates the sea component), which have been previously cut to a suitable length and pre-treated with inorganic salts, are oriented perpendicularly with respect to the backing layer and a given portion of their length penetrates within the adhesive layer.

Once the adhesive has been reticulated, if the flocked material is subjected to removal of the sea component by means of immersion or treatment in an organic solvent (e.g. trichloroethylene) or in a basic or acid solution, the problem shown in Figure 1B is encountered: the sea component is removed completely, even in the portion inserted in the adhesive layer. This is the cause of major problems with the structural resistance and strength of the material, as substantially empty spaces are created between the adhesive and the island component of the undissolved fibre. The fibres thus tend to become detached from the substrate, as they are no longer "submersed" within the layer of adhesive, resulting in "exfoliation" of the flocked material. See Example 7.0, which was carried out by the Applicant, in which the removal step was performed by immersing the flocked material in a bath containing 8% NaOH. The result is total removal of the sea component, even in the part immersed in the adhesive, with the result that the remaining fibre is no longer securely anchored to the backing layer and it can be easily removed by abrasion, to the extent that in the next dyeing step in a jet dyeing machine at the temperature of 120°C with disperse dyes, and resulting reduction, the microfibre is completely removed from the adhesive layer.

This drawback concerning the step for dissolution of the sea component encountered in the prior art processes has been resolved by the Applicant through the development of a process according to the present invention.

The invention concerns a process for the preparation of the flocked material starting from sea-island types of bicomponent fibres, comprising a step for selective removal of the sea component carried out using a removal agent that has a viscosity ranging between 300 mPa.s and 100.000 mPa.s, preferably between 400 and 64.000 mPa.s. The removal agent is preferably in the form of a paste having a viscosity within the ranges indicated above and that is spread on the fibres following flocking of the fibres by means of an electrostatic process and drying of the adhesive layer.

Owing to its viscosity, the removal agent is unable to penetrate within the adhesive layer where the sea-island fibres are partially immersed and therefore it is unable to affect that part of the sea component that is immersed in the layer of adhesive. Therefore, the removal of the sea component is a selective process and it makes it possible to obtain a

flocked material that offers good abrasion resistance and resistance to fibre removal, unlike the flocked products known in the sector that are obtained starting from sea-island fibres and have the drawback of offering poor resistance to fibre removal by abrasion, as those same fibres are not securely anchored to the layer of adhesive.

In a preferred embodiment of the process of the invention, a further measure can be adopted to avoid corrosion of the sea component in the lower part of the fibres and even more so in the part immersed in the adhesive layer; this measure consists in protecting the above-mentioned portion of fibres by using a removable resin applied by spreading, for example by air-spraying, prior to applying the removal agent. The removable resin can have the same formulation as the removal agent, but without the corrosive agent (which can be a caustic agent, acid or selective solvent). For example, the removable resin can consist of a solution of polyvinyl alcohol or an aqueous solution of a thickening agent in general, preferably compatible with the sea component with which it comes into contact. The viscosity of the removable resin is greater than that of the corrosive paste so as to prevent permeation between them.

Controlling the amount of removable resin deposited allows for finer modulation of the fibre fraction to be dissolved.

Referring now to Figure 2, it can be noted that the selective removal step of the invention makes it possible to obtain a flocked material in which the sea component 4 has been partially removed from the portion of fibre that is not immersed in the adhesive layer. This constitutes a substantial structural difference between the flocked material of the invention and the flocked material of the prior art, as can be seen by comparing Figure 2 and Figure 1B, or Figures 5 and 6.

The invention thus also concerns a flocked material that can be obtained by means of the process described hereinabove, in which the sea component of the sea-island bicomponent fibre is partially removed from the portion of fibre that is not immersed in the adhesive layer (Figure 2).

Keeping an undissolved portion of the sea component outside of the adhesive layer also makes it possible to keep the fibre more localized at the flocking point and this gives rise to a product with a more appealing appearance (the nap is perceived as being more uniform).

The invention shall now be explained in detail below with reference also to the attached figures, of which:

- Figure 1A shows the flocked material of the invention prior to removal of the sea component;

- Figure 1B shows the flocked material with sea-island bicomponent fibres following removal with a solvent solution and/or base solution of the prior art;
- Figure 2 shows the flocked material of the invention after selective removal of the sea component by means of the removal agent of the invention;
- Figure 3 is a SEM (Scanning Electron Microscope) image of the flocked material of the invention prior to removal of the sea component;
- Figure 4 is a SEM image of the flocked material following the selective superficial removal of the sea component;
- Figure 5 is a detail from Figure 4 where the intact structure is evident at the base of the emerging flocked fibers, whereas the sea component has been removed from the upper part by selective dissolution;
- Figure 6 is a SEM image of the flocked material following non-selective removal of the sea component by immersion in a bath of NaOH;
- Figures 7 and 8 are details from Figure 6 in which it is possible to see that the microfibers with the sea component removed are not anchored to the layer of glue in some points; the effect is particularly evident on the surface (Figure 8), where craters can be seen on the layer of glue, left by the fibers that have lost their adhesion following dissolution of the sea component.

The present invention concerns a process for the preparation of a flocked material starting from sea-island bicomponent fibres, comprising the steps of:

- spinning a bicomponent fibre of the "island-in-the-sea" type;
- cutting the fibre to a length ranging between 0.1 mm and 3 mm, preferably between 0.3 and 1.25 mm;
- optionally, dyeing fibres with colouring agents, preferably with disperse dyes;
- activating the fibre by application of an aqueous solution of inorganic salts;
- applying an adhesive agent onto a backing layer made of fabric or non-woven fabric;
- laying the cut fibre on the backing layer coated with the adhesive agent by means of electrostatic deposition, which correctly orients the fibres;
- drying and possibly reticulating the adhesive agent;
- optionally, removing excess fibres;
- optionally, applying a layer of removable resin at the base of the fibres to protect the base of the flocked fibres;

- selectively and partially removing the sea component of the fibres by applying a removal agent that has a viscosity ranging between 300 mPa.s and 100.000 mPa.s, preferably between 400 and 64.000 mPa.s;
- optionally, proceeding with dyeing the flocked material.

The viscosity of the removal agent and of every other fluid has been measured as reported in Example 8.0 unless specified otherwise.

The spinning of the sea-island bicomponent fibres can be carried out according to prior-art techniques, which comprise the feeding of two pure polymers or two mixtures of polymers to a spinneret so that one of the two polymeric components ("sea") completely surrounds the other component constituted by various polymeric filaments (preferably 16 microfilaments of circular shape and equal diameter) that form the various "islands". In this regard, the island component can be chosen from among: modified polyesters, cationic polyesters, nylon or other types of polyamides (PA), polyethylene (PE), polypropylene (PP), polytrimethylene terephthalate (PTT), polybutylene terephthalate (PBT) and polyethylene terephthalate (PET), the latter being particularly preferred.

The island component can be solution-dyed, that is, dyed prior to spinning with the aid of specific colouring agents or pigments added during the spinning process.

The sea component can be chosen from among: nylon 6,6, co-polyester (called co-PES or TLAS) with a different content of monomer soluble in alkali, modified polyolefins with insertion of polar monomers in the chain, the polar monomers preferably being selected from among vinyl alcohol, vinyl acetate or maleic anhydride (called co-PS). Depending upon the content of monomer soluble in alkali, the co-PES and co-PS can be easily removed by adding a solution of alkali (low monomer content) or also in hot water (high monomer content).

Both the sea and island components can be used in a mixture with added components selected from among inorganic pigments for the island component, and incompatible polymers for the sea component. Among the added inorganic pigments for the island component, carbon black is particularly preferred. It makes it possible to achieve a coloured "flock" in shades of colour ranging from grey to black, with a colouring that proves to be particularly resistant to UV degradation, even by adding only small amounts of the colouring agent.

Among the added incompatible components for the sea component, polyvinyl alcohol for the co-PES and polyethylene glycols with average molecular weights between 10,000 and 20,000 g/mol for the co-PS, are particularly preferred.

In a particularly preferred embodiment, the fibre used in the invention is made up of an island component made of PET and a sea component made of PA 6,6 or co-PES.

The ratio of the island component to the sea component in the bicomponent fibre is such as to enable spinning of the two components by means of a spinneret rapidly and efficiently. Said island/sea ratio is preferably within the range of 20/80 to 80/20, more preferably within the range of 50/50 to 80/20. Seen in section, the fibre exhibits a number of island components ranging from 8 to 96, preferably 8 to 40.

Following the spinning step, the bicomponent fibre thus obtained is subjected to a drawing process so as to reduce the titre thereof from a range of 6.5 to 19.4 dtex, preferably within the range of 9.2 to 17 dtex, to a titre ranging between 3 and 5 dtex. Drawing is carried out preferably with drawing ratios generally varying in the range of 4-1, preferably in the range of 3-1, more preferably in the range of 2.5-1.

Following the drawing step, the "tow" thus obtained is collected in bins and undergoes cutting of the continuous fibre to a length ranging between 0.1 mm and 3.0 mm. The length of the fibre preferably ranges between 0.3 and 1.25 mm.

The cut fibre is subjected to an activation step preferably by means of immersion in a bath of aqueous solution comprising inorganic salts, for example aluminium sulphate and/or calcium chloride. Fibre that has been cut and activated is defined as "flock". The length/diameter ratio in the flock must be within the range of 10:1 and 100:1, preferably between 20:1 and 50:1.

This activation step makes the cut fibre more sensitive to the electrostatic field applied during the electrostatic deposition step and thus enables a more precise orientation of the fibres in a direction perpendicular to the substrate and to the adhesive layer.

Preferably, the flock is left to dry and after drying it has an inorganic salt content within the range of 0.5% and 2% of the initial weight.

The backing layer on which a layer of adhesive agent is applied can be an orthogonal fabric or a knit fabric, or a non-woven fabric such as a spun-bonded non-woven nylon or polypropylene fabric, or a non-woven elastomeric composite fabric (hereinafter identified as "EVN base"), for example polyester microfiber in a polyurethane matrix. The backing layers indicated above can be utilized as is or they can undergo a surface coating treatment to eliminate the porosity thereof, which would alter the thickness of the applied adhesive agent on the surface; the surface coating treatment can also facilitate fixing of the adhesive agent to the surface of the backing layer. Alternatively, the backing layer can be a film made of a polyolefin, such as polypropylene pre-treated with plasma to make the surface hydrophilic and thus easily wettable with the adhesive agent, or it can be paper.

The backing layer has a unit weight ranging between 40 g/m² and 500 g/m², preferably between 80 g/m² and 350 g/m², and a thickness ranging between 0.10 mm and 2.0 mm, preferably between 0.20 mm and 1.10 mm.

The adhesive agent placed on the backing layer is preferably chosen from among: a polyurethane adhesive (in a solvent or water), a water-based acrylic adhesive, and a silicone glue; silicone glues and polyurethane adhesives are particularly preferred.

A pigment - and/or additives capable of facilitating adhesion with the bicomponent fibre - can be added to the adhesive agent for the purpose of giving the final flocked material specific shades of colour, preferably a conductive pigment capable of facilitating the subsequent flocking process (making the backing layer conductive makes it possible to neutralize the charges transported from the flock and thus keep the electrostatic field applied constant). These additives, which are also called adhesion promoters, are molecules possessing functional groups compatible with the adhesive agent (or that react with the functional groups of the latter) and functional groups compatible with the sea component (or that react with the functional groups of the latter) of the bicomponent fibre that is made to penetrate into the glue. The layer of adhesive agent can be applied by coating the entire surface of the backing layer (full application) or only part of it (patterned application), for a thickness within the range of 0.05 mm to 0.50 mm, preferably 0.10 mm to 0.35 mm. In the case of patterned application, the flock is stably deposited only in areas with the adhesive agent, thus realizing patterns on the surface of the backing layer.

The electrostatic flocking of the flock on the adhesive agent layer preferably takes place in an environment with a controlled and constant level of humidity ranging between 60% and 90%, preferably between 70% and 80%. The electrostatic field applied is preferably within the range of 20 to 50 kV, preferably 20 to 40 kV.

The amount of flock deposited ranges between 50 and 250 g/cm², preferably between 140 and 190 g/cm², relative to the area in which the adhesive agent is present in the case of patterned deposition.

The titre of the sea-island fibre ranges from 1.5 dtex to 10 dtex, preferably within the range of 3.0 dtex to 7 dtex.

The deposition rate is between 2 and 7 m/min, preferably between 2 and 4 m/min.

The flock fibres penetrate within the adhesive layer to a depth ranging from 40 microns to the entire thickness of the layer of adhesive agent, depending upon the adhesive agent used, its viscosity and the applied electrical field.

Upon completion of deposition, the material is placed in an oven to harden and fix the adhesive agent, for a period of time ranging from 2 to 10 minutes, preferably 3 to 5 minutes. The oven temperature is preferably in the range of 110°C and 200°C, preferably 120 to 190°C.

Selective removal of the sea component takes place by applying a removal agent on the flocked fibres, preferably in the form of a paste and having the viscosity indicated

hereinabove. The removal agent comprises a base, for example NaOH, or an acid, for example formic acid, preferably mixed with a polysaccharide, preferably a polysaccharide such as xanthan. Alternatively, the removal agent can also comprise a selective solvent for the sea component. Examples of solvents suitable for dissolving a co-PS-based sea component consist of halogenated solvents such as trichloroethylene, perchloroethylene, chloroform, hydrocarbon solvents such as toluene, xylene, ethylbenzene, cyclohexane, and other polar solvents such as N,N-dimethylformamide, acetone, dioxane, tetrahydrofuran, methyl ethyl ketone, acetonitrile, dimethyl sulphoxide, methanol and ethanol.

The base or acid concentration may range between 1.5% and 20% by weight, preferably between 4% and 18% by weight. The polysaccharide, and particularly the xanthan, is preferably contained in the paste in an amount ranging from 0.5% to 7% by weight.

The removal agent is applied on the flocked fibres in an amount ranging between 80 and 150 g/m².

In a preferred embodiment of the invention, prior to applying the removal agent, a removable resin can be applied by coating, as a further measure to avoid corrosion of the sea component in the lower part of the fibres and even more so in the part immersed in the adhesive layer. The removable resin can have the same formulation as the removal agent, but without the corrosive agent (which can be a caustic agent, acid or a selective solvent). For example, the removable resin can consist of a solution of polyvinyl alcohol or an aqueous solution of a thickening agent in general, preferably compatible with the sea component with which it comes into contact. The viscosity of the removable resin is preferably greater than that of the removal agent so as to prevent permeation between them. The viscosity of the removable resin preferably ranges between 1.000 mPa.s and 150.000 mPa.s.

Controlling the amount of removable resin deposited allows for finer modulation of the flock fraction to be dissolved.

Following application of the removal agent, the material can be treated with a saturated vapour current, with radio frequencies, with microwaves or thermally treated with hot air, so as to facilitate dissolution of the sea component. Following this optional treatment, which can last 5 to 15 minutes (at a temperature of 70-100°C in the case of thermal treatment), the removal agent and any removable resin can be removed by washing with water. In this manner, selective and partial removal of the sea component is brought about: only the portion of the sea component that is not immersed in the layer of adhesive agent is removed, while the part immersed in the adhesive layer remains and allows for maintaining secure anchoring of the flock fibers to the substrate (see Figures 3-5, which

show the absence of empty spaces between the island fibres and the adhesive layer).

According to a variant of the procedure, following application of the removal agent, the material treated with a saturated vapour current, radio frequencies or microwaves is further treated with hot air in an oven for the purpose of creating a protective barrier on the surface of the removal agent before washing with water.

In this manner it is possible to roll up the flocked material, for example in order to transport it safely to the washing line, without jeopardizing the quality of the final flocked material.

The fraction of fibre that has undergone removal of the sea component reveals the microfibrils constituting the island component, each having a titre in the range of 0.04 dtex to 0.30 dtex. In this manner, the flocked material becomes more resistant to possible abrasions which could cause removal of the fibres and the microfibre proves to be more localized at the flocking point based on the fraction of corroded fibre, thus giving rise to a product with a more appealing appearance (the nap is perceived as being more uniform).

Moreover, the flocked material can also be dyed in a jet dyeing machine and the excess dye then removed without risking a loss of flocked fibre. Alternatively, to produce a flocked material without a mottled effect (flat appearance), it can be dyed in a machine for garment dyeing or more generally for "open-width dyeing", which permits pressurized dyeing of particularly delicate materials without subjecting them to significant mechanical stress (the material remains spread open for its entire width without forming lengthwise or crosswise folds).

Therefore, the flocked material that is obtained using the process of the invention differs from the materials known in the *prior art* in that it does not exhibit empty spaces between the fibres and the layer of adhesive, which could contribute to the poor resistance of the prior art materials in the subsequent dyeing steps and poor resistance to abrasion in general. The flocked material of the invention presents itself as a finer material compared to those of the prior art and offering greater durability over time.

Therefore, an object of the invention is a flocked material obtainable using the process of the invention.

The flocked material of the invention comprises:

- a backing layer, preferably made of fabric or non-woven fabric;
- an adhesive layer applied onto the backing layer;
- a fibrous layer comprising a plurality of sea-island fibres that are preferably oriented in a direction perpendicular to the backing layer, partially immersed in the adhesive layer and having the sea component still present in the portion of fibres immersed within the adhesive layer. The sea component is instead totally or partially absent in the section of fibres that emerges from the adhesive layer.

The adhesive layer is present on the entire surface of the backing layer or on a portion thereof (patterned flocking).

The adhesive layer is of a thickness ranging between 0.05 mm and 0.50 mm, preferably between 0.10 mm and 0.35 mm.

The plurality of sea-island fibres are included within the adhesive layer for a depth that ranges from 40 microns to the entire thickness of the adhesive layer.

The island component of the sea-island fibres has a titre in the range of 0.04 to 0.30 dtex.

The flocked material of the invention can be used in the field of automobiles, furnishings and consumer electronics, replacing all parts currently coated with fabrics, non-wovens or leathers.

In particular, the flocked material of the invention can be used for various applications, for example to coat surfaces and structures such as interiors of motor vehicles, objects for interior decorating (walls, sofas, armchairs, etc.), handbags, suitcases or other accessories, covers or cases for weapons, musical instruments or electronic devices, or to make carpets and/or rugs.

Examples of embodiments and comparative examples

Example 0.1 White flock with PET/TLAS bicomponent fibre – 1.0 mm

An "island-in-the-sea" type of bicomponent fibre flock is realized, in which the island component is realized in PET and the sea component is realized in TLAS (co-polyester soluble in alkali). The ratio of the island component to the sea component in the fibre is 57:43.

The section of the fibre reveals 16 PET microfilaments of circular shape and equal diameter.

The flock is obtained by means of the subsequent procedures of drawing, collecting the tow in bins and cutting the continuous sea-island fibre to the desired length.

The characteristics of the fibre and the flock are as follows:

- 1- drawing ratio 2.5 / 1
- 2- titre 4.3 dtex
- 3- length 1.0 mm

The flock thus defined undergoes activation by means of immersion in a bath of an aqueous solution of aluminium sulphate; after drying, the content of aluminium sulphate in the flock is equal to 1% of the initial weight.

The flock thus realized is called **Thread 1**.

Example 0.1.1 Black flock with (PET+disperse dyes)/TLAS bicomponent fibre

The flock is realized with the same procedure used for **Thread 1**, with the variant that prior to the activation process the flock is dyed with black dye dispersed in water at a temperature of 120°C in accordance with the prior art.

Subsequent activation leaves 1% aluminium sulphate by weight on the flock. The flock thus realized is called **Thread 2**.

Example 0.2 Black flock with solution-dyed PET/TLAS bicomponent fibre – 0.3 mm

An "island-in-the-sea" type of bicomponent fibre flock is realized, in which the island component is realized in PET with added Carbon Black in the amount of 7% and the sea component is realized in TLAS (co-polyester soluble in alkali). The ratio of the island component to the sea component in the fibre is 57:43.

The section of the fibre reveals 16 PET microfilaments of circular shape and equal diameter.

The flock is obtained by means of the subsequent procedures of drawing, collecting the tow in bins and cutting the continuous sea-island fibre to the desired length.

The characteristics of the fibre and the flock are as follows:

- 1- drawing ratio 2.5 / 1
- 2- titre 4.3 dtex
- 3- length 0.3 mm

The flock thus defined undergoes activation by means of immersion in a bath of an aqueous solution of aluminium sulphate; after drying, the content of aluminium sulphate in the flock is equal to 1% of the initial weight.

The flock thus realized is called **Thread 3**.

Example 0.3 White flock with PET/PA6.6 bicomponent fibre – 0.5 mm

An "island-in-the-sea" type of bicomponent fibre flock is realized, in which the island component is realized in PET and the sea component is realized in PA 6,6. The ratio of the island component to the sea component in the fibre is 57:43.

The section of the fibre reveals 16 PET microfilaments of circular shape and equal diameter.

The flock is obtained by means of the subsequent procedures of drawing, collecting the tow in bins and cutting the continuous sea-island fibre to the desired length.

The characteristics of the fibre and the flock are as follows:

- 1- drawing ratio 3.0 / 1
- 2- titre 3.8 dtex
- 3- length 0.5 mm

The flock thus defined undergoes activation by means of immersion in a bath of an aqueous solution of aluminium sulphate; after drying, the content of aluminium sulphate in the flock is equal to 1% of the initial weight.

The flock thus realized is called **Thread 4**.

Example 0.4 White flock with PET/HWS bicomponent fibre – 1.0mm

An "island-in-the-sea" type of bicomponent fibre flock is realized, in which the island component is realized in PET and the sea component is realized in HWS polyester. The ratio of the island component to the sea component in the fibre is 57:43.

The section of the fibre reveals 16 PET microfilaments of circular shape and equal diameter.

The flock is obtained by means of the subsequent procedures of drawing, collecting the tow in bins and cutting the continuous sea-island fibre to the desired length.

The characteristics of the fibre and the flock are as follows:

- 1- drawing ratio 2.5 / 1
- 2- titre 4.3 dtex
- 3- length 1.0 mm

The flock thus defined undergoes activation by means of immersion in a bath of an aqueous solution of aluminium sulphate, in the presence of 0.5% calcium chloride; after drying, the aluminium flock undergoes a 1% increase in weight.

The flock thus realized is called **Thread 5**.

Example 0.5 White flock with PET/TLAS bicomponent fibre – 1.0 mm type A

An "island-in-the-sea" type of bicomponent fibre flock is realized and it is similar to the one reported in Example 0.1 with the exception that the bicomponent fibre utilized for realizing the flock has the following characteristics:

- 1- overall drawing ratio 3.5 /1
- 2- titre 3.1 dtex
- 3- length 0.5 mm
- 4- ratio of the island component to the sea component equal to 55:45
- 5- Section of the fibre containing 36 PET microfilaments of circular shape and equal diameter.

The flock thus obtained undergoes activation by means of immersion in a bath of an aqueous solution of aluminium sulphate, in the presence of 0.5% calcium chloride; after drying, the aluminium flock undergoes a 1% increase in weight.

The flock thus realized is called **Thread 6**.

Example 1.0 EVN base – silicone glue – 1.0 mm

A layer of bicomponent ALAPATEC 30340 adhesive (100% silicone glue supplied by CHT) having a thickness of 0.2 mm and a viscosity of 50.000 mPa.s is applied onto a backing layer realized in a composite material made of PET microfibre with 30% of a polyurethane matrix and having a thickness of 1.10 mm.

Electrostatic and mechanical flocking follows so as to deposit the flock indicated as **Thread 1**; on average the flock penetrates into the layer of glue by 60 microns.

Flocking takes place in an environment with a controlled and constant level of humidity at 65%, exposed to an electrostatic field of 30 kV so as to enable the deposition of 144 g/cm² of flock at a line rate of 3.0 m/min.

The intermediate product thus identified is placed in a convective oven to reticulate for 4 minutes at 150°C and it is called **FK 01.0**.

Example 1.1 EVN base - silicone glue – 1.0mm

An intermediate product similar to the one identified as **FK 01.0** (Example 1.0) is realized, using bicomponent TUBICOAT PROTECT LSR adhesive (100% silicone glue, supplied by CHT). The glue contains a black pigment and has a viscosity of 35.000 mPa.s, which is lower than that of ALPATEC 30340, to the extent that penetration of the flock indicated as **Thread 1** equals the thickness of 0.2 mm of the glue and the flock is therefore in contact with the surface of the backing layer.

The intermediate product is called **FK 01.1**.

Example 1.2 EVN base – polyurethane glue – 1.0 mm

An intermediate product similar to the one identified as **FK 01.0** (Example 1.0) is realized, using an aromatic, bicomponent, polyester-based polyurethane glue that can be reticulated by heating. The glue contains a black pigment and has a viscosity of 30.000 mPa.s, which is lower than that of the preceding examples, to the extent that penetration of the flock indicated as **Thread 1** equals the thickness of 0.2 mm of the glue and the flock is therefore in contact with the surface of the backing layer.

The intermediate product thus identified is placed in a convective oven to reticulate for 4 minutes at 150°C and it is called **FK 01.2**.

Example 1.3 EVN base - polyurethane glue – 0.5 mm

An intermediate product similar to the one identified as **FK 01.0** (Example 1.0) is realized, using the flock indicated as **Thread 6**, rather than Thread 1, and an aromatic,

bicomponent, polyester-based polyurethane glue that can be reticulated by heating. The glue has a viscosity of 29.000 mPa.s, and penetration of the flock indicated as **Thread 6** equals the thickness of 0.2 mm of the glue and the flock is therefore in contact with the surface of the backing layer.

The intermediate product thus identified is placed in a convective oven to reticulate for 4 minutes at 150°C and it is called **FK 01.3**.

Example 2.0 EVN base – silicone glue with adhesion promoter – 1.0 mm

An intermediate product similar to the one identified as **FK 01.1** (Example 1.1) is realized, using bicomponent TUBICOAT PROTECT LSR adhesive (100% silicone glue, supplied by CHT), which already contains within it a black pigment and an adhesion promoter specific for TLAS. The glue has a viscosity of 35.000 mPa.s, and penetration of the flock indicated as **Thread 3** equals the thickness of 0.2 mm of the glue (the flock is therefore in contact with the surface of the backing layer).

The intermediate product is called **FK 02.0**.

Example 3.0 fabric base – acrylic adhesive – 1.0 mm

A layer of TUBVINIL 401H adhesive of a thickness of 0.15 mm (water-based acrylic base, supplied by CHT) is placed on a 100% PET cloth backing layer having a unit weight of 82g/m² and electrostatic flocking is carried out with the flock indicated as **Thread 2**.

Flocking takes place in an environment with a controlled and constant level of humidity at 65%, exposed to an electrostatic field of 30 kV so as to enable the deposition of 165 g/cm² of flock at a line rate of 3.5 m/min.

Subsequently, the product is placed in a convection oven at 170°C for 3 minutes to dry and fix the adhesive.

The flock penetrates into the adhesive to the point of coming into contact with the underlying layer of fabric.

The intermediate product is called **FK 03.0**.

Example 4.0 removable base - silicone glue – 1.0 mm

A layer of bicomponent TUBICOAT PROTECT LSR adhesive (100% silicone glue, supplied by CHT) containing black pigment (see Example 1.1) and having a thickness of 0.4 mm is placed on a strip of Teflon that has surface roughness.

This is followed by electrostatic flocking with the flock indicated as **Thread 1**.

Flocking takes place in an environment with a controlled and constant level of humidity at 65%, exposed to an electrostatic field of 40 kV so as to enable the deposition of 210 g/cm² of flock at a line rate of 2.2 m/min.

Subsequently, the product is placed in a convection oven at 140°C for 6 minutes to dry and fix the adhesive.

The flock penetrates into the adhesive to the point of coming into contact with the underlying film.

The intermediate product is called **FK 04.0**.

Example 4.1 PP base - polyurethane glue – 1.0 mm

A layer of aromatic, bicomponent, polyester-based polyurethane glue that can be reticulated by heating, containing a black pigment and having a thickness of 0.4 mm is placed over a film of PP having a thickness of 120 microns that has been pre-treated with plasma to make the surface hydrophilic.

This is followed by electrostatic flocking with the flock indicated as **Thread 1**.

Flocking takes place in an environment with a controlled and constant level of humidity at 65%, exposed to an electrostatic field of 40 kV so as to enable the deposition of 210 g/cm² of flock at a line rate of 2.2 m/min.

Subsequently, the product is placed in a convection oven at 140°C for 3 minutes to dry and fix the adhesive.

The flock penetrates into the adhesive to the point of coming into contact with the underlying film.

The intermediate product is called **FK 04.1**.

Example 5.0 spun-bonded base – silicone glue – PA 6,6 - 0.5 mm

A layer of bicomponent TUBICOAT PROTECT LSR adhesive (100% silicone glue, supplied by CHT) having a thickness of 0.2 mm is placed over a spun-bonded PP fabric having a unit weight of 90 g/m².

This is followed by electrostatic flocking with the flock indicated as **Thread 4**.

Flocking takes place in an environment with a controlled and constant level of humidity at 80%, exposed to an electrostatic field of 22 kV so as to enable the deposition of 191 g/cm² of flock at a line rate of 2.5 m/min.

Subsequently, the product is placed in a convection oven at 150°C for 5 minutes to dry and fix the adhesive.

The flock penetrates into the adhesive by 150 microns.

The intermediate product is called **FK 05.0**.

Example 6.0 fabric base – PUD glue – HWS- 1.0 mm

A layer of bicomponent TUBICOAT PROTECT LSR adhesive (100% silicone glue, supplied by CHT) having a thickness of 0.2 mm is placed over a 100% PET cloth having a unit weight of 82 g/m².

This is followed by electrostatic flocking with the flock indicated as **Thread 5**.

Flocking takes place in an environment with a controlled and constant level of humidity at 75%, exposed to an electrostatic field of 25 kV so as to enable the deposition of 150 g/cm² of flock at a line rate of 3.0 m/min.

Subsequently, the product is placed in a convection oven at 150°C for 4 minutes to dry and fix the adhesive.

The flock penetrates into the adhesive by 150 microns.

The intermediate product is called **FK 06.0**.

Example 7.0 – Dissolution in a bath of NaOH + dyeing (Comparative example)

The intermediate products FK 01.0, FK 01.1, FK 01.2, FK 02.0, FK 03.0, FK 04.0 and FK 04.1, containing TLAS as the sea component in the flock, were washed in a bath containing 8% NaOH (w/w) at a temperature of 80°C for 15 minutes, and then washed in cold water and placed in a convection oven to dry.

In intermediate products FK 01.0 and FK 03.0, it was evident that the flock was detached from the layer of glue, leaving ample bare areas in which only glue and the substrate were present.

In intermediate products FK 01.1, FK 01.2, FK 02.0, FK 04.0 and FK 04.1, the flock remains on the surface of the product and the sea component is completely removed, including the part submersed in the glue; therefore, the adhesion surface is limited to the base of the microfibre only. For this reason, the microfibrinous flocked product tends to be easily removed by abrasion, to the extent that in the subsequent dyeing step in a jet dyeing machine at the temperature of 120°C with disperse dyes, and resulting reduction, the microfibre is completely removed from the glue.

Example 8.0 Dissolution with NaOH paste in air + dyeing

A preparation of thickened NaOH (removal agent) is dispensed using a doctor blade at a rate of 100 g/m² on the flock side of the intermediate products FK 01.0, FK 01.1, FK 01.2, FK 02.0, FK 03.0, FK 04.0 and FK 04.1 containing TLAS as the sea component of the flock.

The corrosive paste, 16% of which is constituted by NaOH and 0.5% of which by DENIMCOL SPEC FTL (Xanthan polysaccharide supplied by CHT), has a pseudoplastic behaviour and a viscosity of about 400 mPa.s under application conditions.

Viscosity was measured using a Brookfield DVIII rotational viscometer at 20°C, with a Small Sample Adapter accessory and an SC4-28 spindle, at a speed of about 5 rpm, corresponding to a shear rate of 5 s⁻¹.

The intermediate products coated with the corrosive paste were thermally treated at 80°C for 10 minutes in an oven and then washed in cold water and placed in a convection oven to dry.

SEM analyses showed that the sea component was affected only superficially (SEM image of sample FK 01.1 in Figure 3).

Example 9.0 - Dissolution with NaOH paste in vapour

A preparation of thickened NaOH (removal agent) was dispensed using a doctor blade at a rate of 100 g/m² on the flock side of the intermediate products FK 01.0, FK 01.1, FK 01.2, FK 01.3, FK 02.0, FK 04.0 and FK 04.1 containing TLAS as the sea component of the flock.

The corrosive paste, 4% of which is constituted by NaOH and 2% of which by DENIMCOL SPEC FTL (Xanthan polysaccharide supplied by CHT), has a pseudoplastic behaviour and a viscosity of about 28.000 mPa.s under application conditions.

The intermediate products coated with the corrosive paste were treated with a saturated vapour current at atmospheric pressure for 3 minutes and then washed in cold water and placed in a convection oven to dry.

In intermediate products FK 01.1, FK 01.2, FK 01.3, FK 02.0, FK 04.0 and FK 04.1, the sea component was completely removed in the non-submersed part of the flock in contact with the corrosive paste revealing the microfibrils, whereas the other part was still had a microfibre nature and adhesion of the bundle to the backing layer was also ensured (SEM image of the FK 01.1 sample, Figures 4 and 5).

Intermediate product FK 01.0 appeared with patches lacking flock on the surface owing to insufficient penetration of the flock into the silicone glue.

Intermediate product FK 03.0 showed a loss of flock and glue on most of the surface.

Dyeing was then carried out in a jet dyeing machine with disperse dyes at 120°C and subsequent reduction of excess dye only on intermediate products FK01.1, FK 01.2, FK 01.3, FK 02.0, FK 04.0 and FK 04.1.

Example 9.1 - Dissolution with NaOH paste in vapour + open-width dyeing

The sea component of intermediate products FK 01.1, FK 01.2, FK 01.3, FK 02.0, FK 04.0 and FK 04.1 was removed as described in Example 9.0. The intermediate products were then dyed by means of a dyeing machine for open-width dyeing, using disperse dyes in water at 120°C and subsequent reduction of excess dye. In this manner, dyed flocked materials were obtained, characterized by a more uniform and even appearance.

Example 10.0 Dual coating and vapour

A preparation of PVA in water with a viscosity of 54.000 mPa.s was applied onto the intermediate products FK 01.1, FK 01.2, FK 02.0 and FK 04.0 by means of an air doctor blade, with the aim of applying a layer of 30 g/m² of solution only at the base of the flocked fibres.

Subsequently, a preparation of thickened NaOH was dispensed by means of a cylinder doctor blade at a rate of 100 g/m² on the flocked side of intermediate product FK 01.1.

The corrosive paste (removal agent), 4% of which is constituted by NaOH and 2% of which by DENIMCOL SPEC FTL (Xanthan polysaccharide supplied by CHT), has a pseudoplastic behaviour and a viscosity of about 28.000 mPa.s under application conditions.

The intermediate products FK 01.1, FK 01.2, FK 02.0 and FK 04.0 coated with the corrosive paste were treated with a saturated vapour current at atmospheric pressure for 3 minutes and then washed in cold water and placed in a convection oven to dry.

The sea component proved to be hydrolyzed only in the apical part of the flocked fibres, that is, for about 1/5 of the emerged length.

Example 11.0 – Dissolution in water and RF - HWS

An aqueous solution of 1.5% DENIMCOL SPEC FTL (Xanthan polysaccharide supplied by CHT) was dispensed by means of a doctor blade at the rate of 120 g/m² on the flock side of intermediate product FK 06.0. The viscosity of the solution under application conditions was about 20.000 mPa.s.

The intermediate product was then subjected to radio frequencies with a parallel field, with a difference in electric potential equal to 1.0 kV, and then washed in cold water.

This was followed by immersion in a 10% calcium chloride solution for 8 minutes at a temperature of 50°C for the purpose of making the residual sea component (HWS) insoluble in the part of the flock that is submersed in the silicone glue, by washing with water without hardening salts and by drying in a convection oven.

The intermediate product remains intact as it passes the next dyeing step for dyeing with disperse dyes at 120°C in a jet dyeing machine and the reduction to eliminate excess dye.

Comparative Example 12.0 – Dissolution in NaOH and MW (microwaves)

A preparation of thickened NaOH was dispensed by means of a doctor blade at the rate of 100 g/m² on the flock side of intermediate product FK 01.1, then heated for 2 minutes in a microwave oven with power equal to 5 KW, washed in cold soft water and dried in a convection oven.

The corrosive paste (removal agent), 1.0% of which is constituted by NaOH and 2.0% of which by DENIMCOL SPEC FTL (Xanthan polysaccharide supplied by CHT), has a pseudoplastic behaviour and a viscosity of about 2.000 mPa.s under application conditions.

Following microwave treatment, the sea component in the part of the fibre emerging from the glue proved to have been entirely removed, revealing the microfibre, while it remained in the part anchored to the backing layer. Dyeing was then carried out in a jet dyeing machine with disperse dyes at 120°C, as was subsequent reduction of excess dye.

CLAIMS

1. A process for the preparation of a flocked material starting from sea-island bicomponent fibres, comprising the steps of:
 - spinning a bicomponent fibre of the “island-in-the-sea” type;
 - cutting the fibre to a length ranging between 0.1 mm and 3 mm, preferably between 0.3 and 1.25 mm;
 - optionally, dyeing the fibres with colouring agents;
 - activating the fibre by application of an aqueous solution comprising inorganic salts;
 - applying an adhesive agent onto a backing layer, preferably made of fabric or non-woven fabric;
 - laying the cut fibre on the backing layer coated with the adhesive agent by means of electrostatic deposition, which correctly orients the fibres;
 - drying and optionally reticulating the adhesive agent;
 - optionally, removing excess fibres;
 - optionally, applying a layer of a removable resin at the base of the fibres to protect the fibre base;
 - selectively and partially removing the sea component of the fibres by applying a removal agent that has a viscosity ranging between 300 mPa.s and 100.000 mPa.s, preferably between 400 and 64.000 mPa.s;
 - optionally, proceeding with dyeing the flocked material.
2. The process according to claim 1, wherein the island component of the bicomponent fibre is chosen from among: modified polyesters, cationic polyesters, nylon or other types of polyamides (PA), polyethylene (PE), polypropylene (PP), polytrimethylene terephthalate (PTT), polybutylene terephthalate (PBT) and polyethylene terephthalate (PET).
3. The process according to claim 1 or 2, wherein the sea component of the bicomponent fibre is chosen from among: nylon 6,6 and co-polyester with a different content of monomer soluble in alkali and modified polyolefins with insertion of polar monomers in the chain, the polar monomers preferably being selected from among vinyl alcohol, vinyl acetate or maleic anhydride .
4. The process according to any one of the preceding claims, wherein following the spinning step, the bicomponent fibre undergoes a drawing step, which is preferably carried out with drawing ratios varying within the range of 4-1, preferably within the range of 3-1, and more preferably in the 2.5-1 range.

5. The process according to any one of the preceding claims, wherein said activation step comprises immersion of the cut fibres in a bath of an aqueous solution comprising inorganic salts.
6. The process according to claim 5, wherein following the activation treatment, the cut fibres are left to dry and after drying they have an inorganic salt content ranging between 0.5% and 2% of the initial weight.
7. The process according to any one of the preceding claims, wherein the backing layer on which the adhesive agent layer is applied consists of an orthogonal fabric or a knit fabric, or a non-woven fabric, preferably a spun-bonded non-woven nylon or polypropylene fabric, or non-woven elastomeric composite fabric, preferably polyester microfibre in a polyurethane matrix, or a film made of polyolefin, preferably polypropylene, or paper.
8. The process according to any one of the preceding claims, wherein the adhesive agent that is placed on the backing layer is chosen from among: a polyurethane adhesive in an aqueous or solvent dispersion, a water-based acrylic adhesive and a silicone glue.
9. The process according to claim 8, wherein a pigment, preferably a conductive pigment capable of facilitating the subsequent flocking process, and/or additives capable of facilitating adhesion with the bicomponent fibre, are added to said adhesive agent.
10. The process according to any one of the preceding claims, wherein said electrostatic flocking of the cut fibres on the adhesive agent layer takes place in an environment with a controlled and constant level of humidity ranging between 60% and 90%, preferably between 70% and 80%, with the application of an electrostatic field ranging between 20 and 50 kV, preferably between 20 and 40 kV.
11. The process according to claim 10, wherein the amount of cut fibres deposited on the substrate ranges between 50 and 250 g/cm², preferably between 140 and 190 g/cm².
12. The process according to any one of the preceding claims, wherein said removal agent comprises a base, preferably NaOH, or an acid, preferably formic acid, preferably mixed with a polysaccharide.
13. The process according to any one of the preceding claims, wherein said removal agent is applied on the flocked fibres in an amount ranging between 80 and 150 g/m².
14. The process according to any one of the preceding claims, wherein following application of the removal agent, the material is thermally treated with a saturated vapor current, with radio frequencies, with microwaves or thermally with hot air.

15. The process according to any one of the preceding claims, wherein the viscosity of said layer of removable resin is greater than that of said removal agent, preferably within the range of 1,000 mPa.s and 150,000 mPa.s.
16. A flocked material comprising:
 - a backing layer, preferably made of fabric or non-woven fabric;
 - an adhesive layer applied onto the surface of the backing layer or a portion thereof;
 - a fibrous layer comprising a plurality of sea-island fibres that are preferably oriented in a direction perpendicular to the backing layer, partially immersed in the adhesive layer and having the sea component still present in the portion of fibres immersed within the adhesive layer and totally or partially absent in the section of fibres that emerges from the adhesive layer.
17. The flocked material according to claim 16, wherein said adhesive layer is of a thickness ranging between 0.05 mm and 0.50 mm, preferably between 0.10 mm and 0.35 mm.
18. The flocked material according to claim 16 or 17, wherein said plurality of sea-island fibres are included within the adhesive layer for a depth that ranges from 40 microns to the entire thickness of the adhesive layer.
19. The flocked material according to any one of claims 16 to 18, wherein the island component of the sea-island fibres has a titre in the range of 0.04 to 0.30 dtex.
20. A use of the flocked material according to any one of claims 16 to 19 in the field of automobiles, furnishings and consumer electronics, replacing all parts currently coated with fabrics, non-wovens or leathers.
21. The use of the flocked material according to any one of claims 16 to 19, to coat surfaces and structures of the interiors of motor vehicles, objects for interior decorating (walls, sofas, armchairs, etc.), handbags, suitcases or other accessories, covers or cases for weapons, musical instruments or electronic devices, or to make carpets.

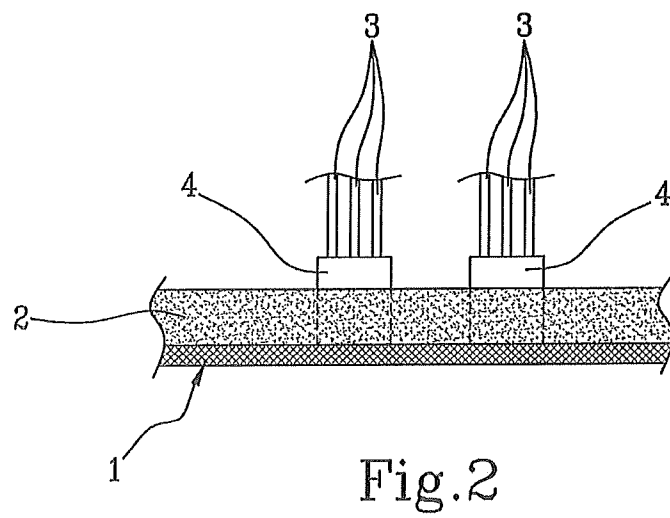
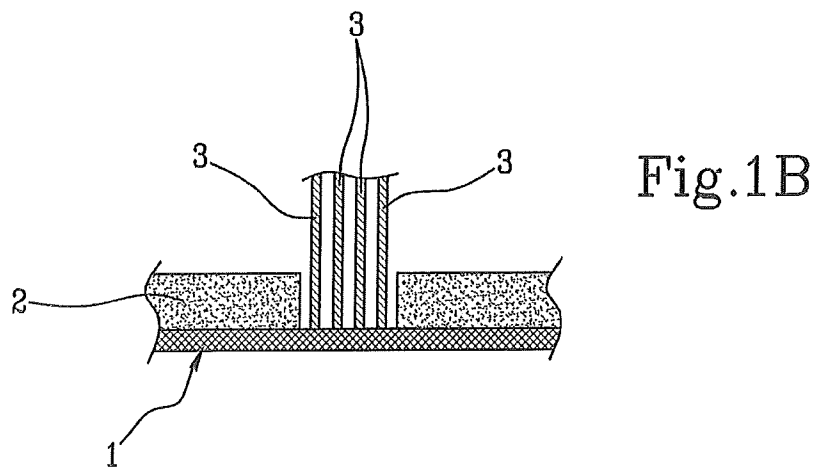
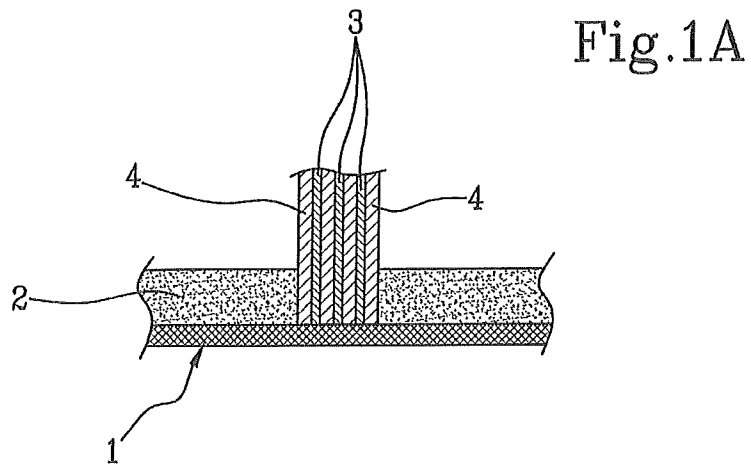




Fig. 3

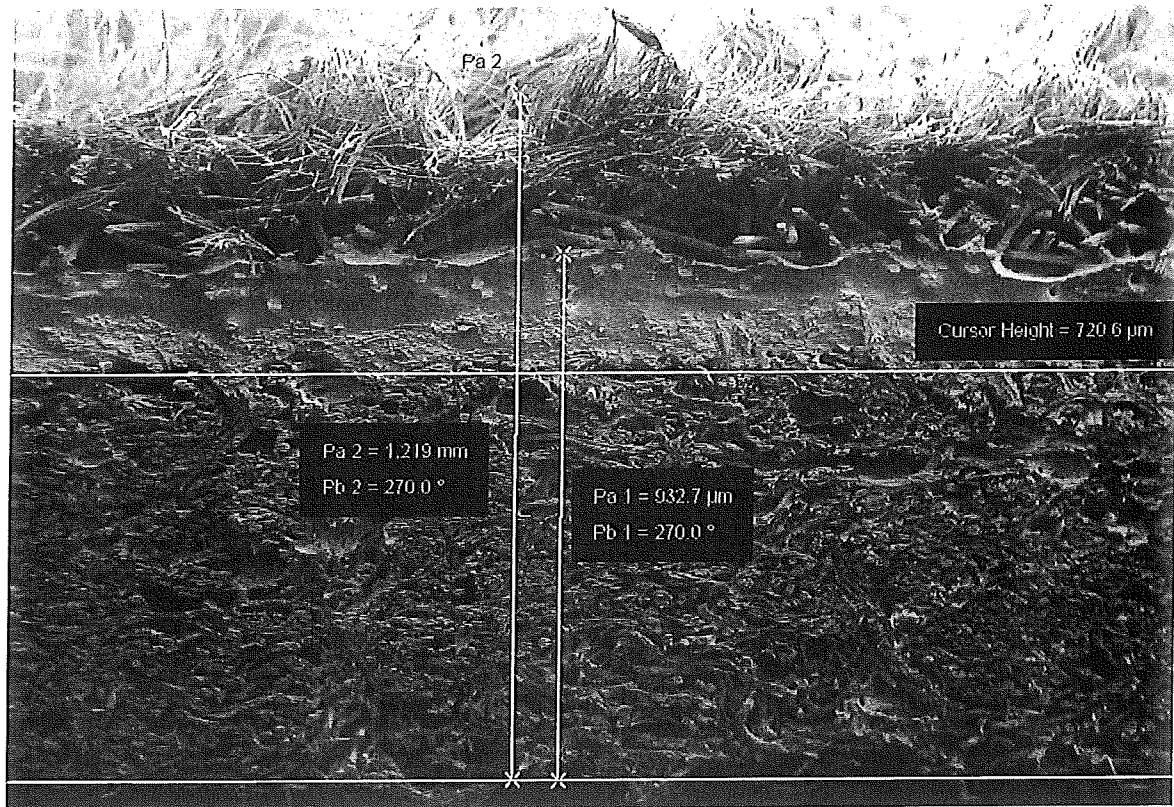


Fig.4

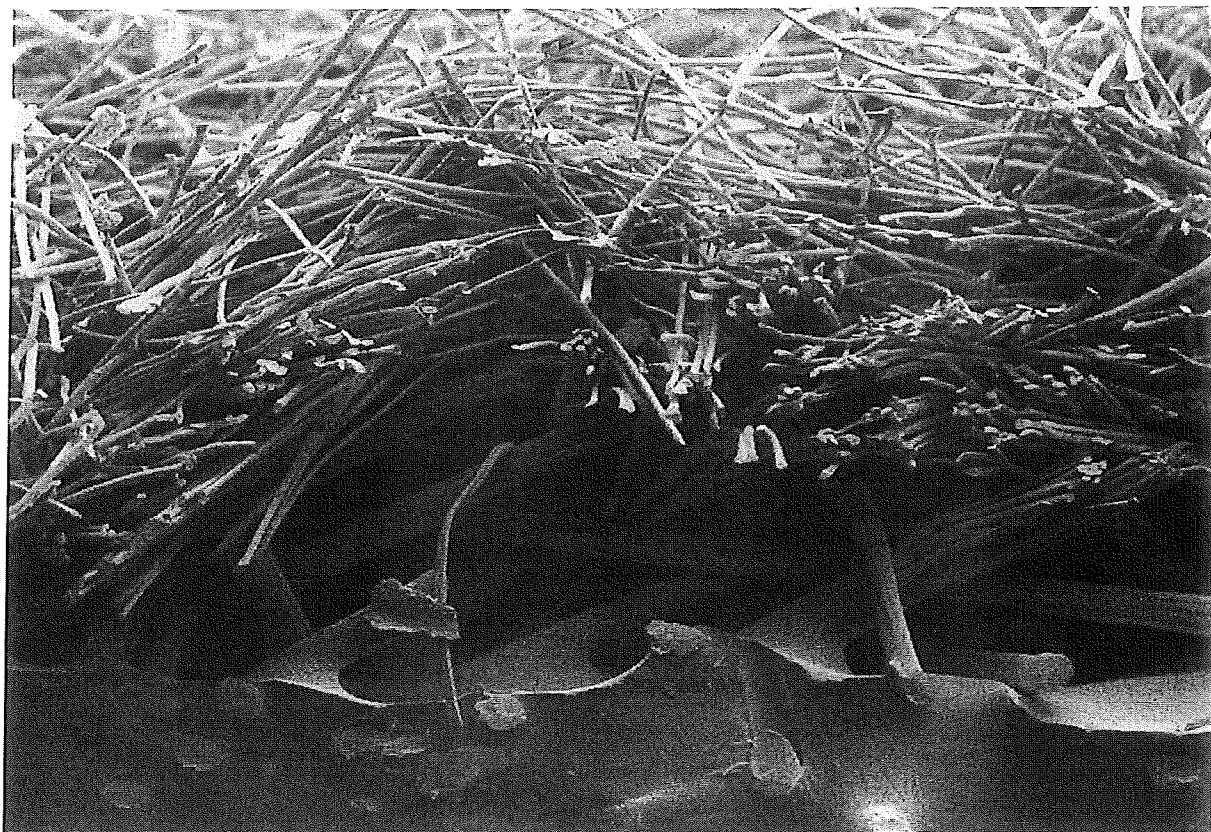


Fig. 5

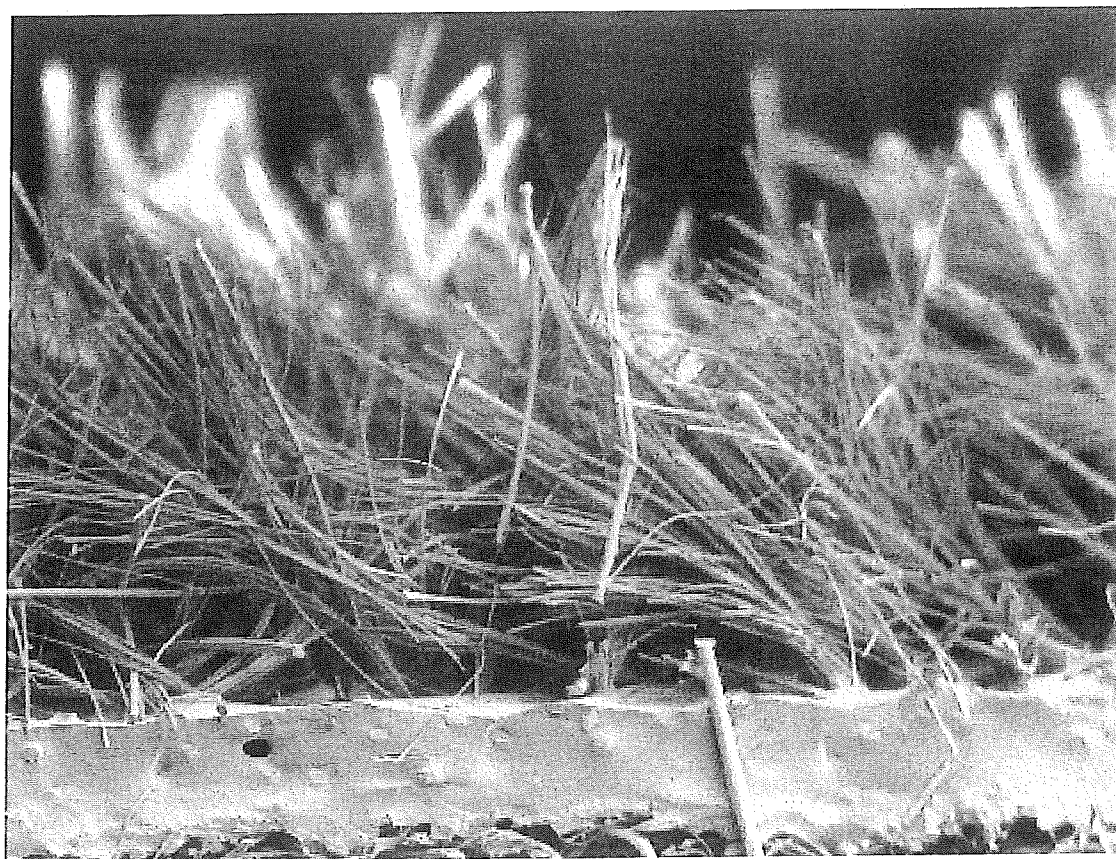


Fig. 6

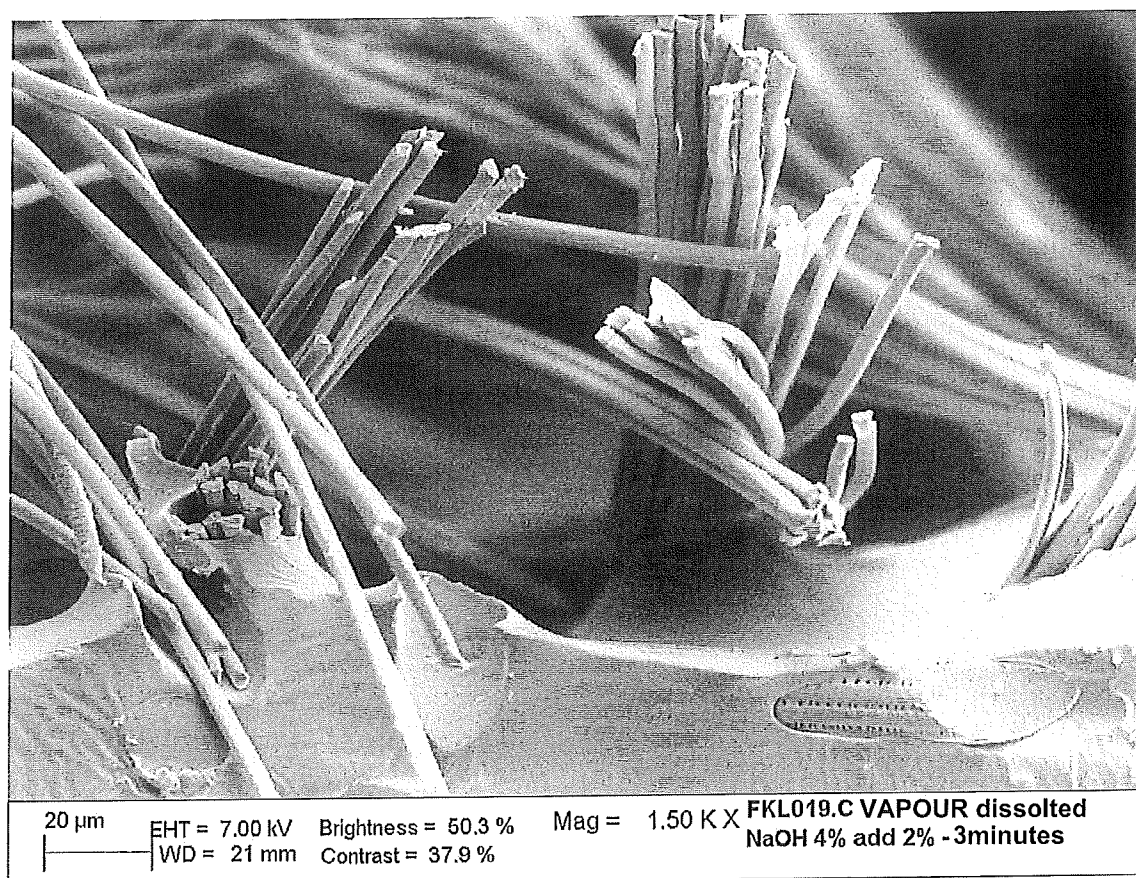


Fig. 7

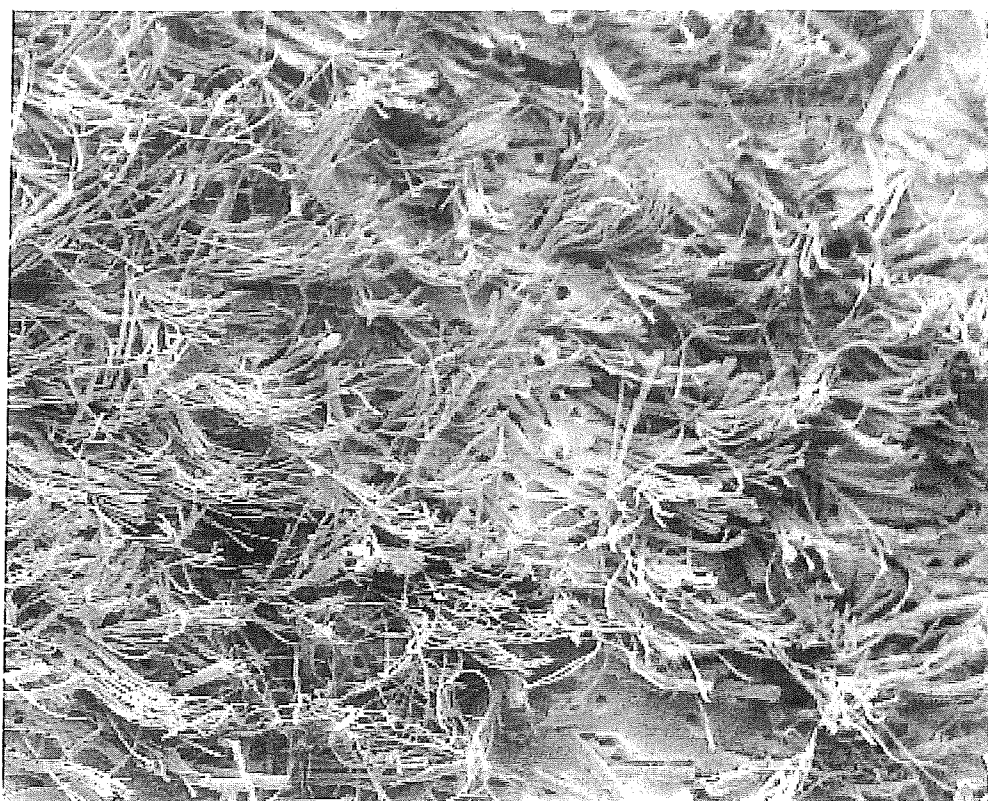


Fig. 8

INTERNATIONAL SEARCH REPORT

International application No

PCT/IB2015/053345

A. CLASSIFICATION OF SUBJECT MATTER

INV. D04H11/00 D04B21/02 D06N3/14 D04H1/541
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)

D04H D04B D06N

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

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☒ Further documents are listed in the continuation of Box C.

☒ See patent family annex.

* Special categories of cited documents :

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Date of the actual completion of the international search

7 July 2015

Date of mailing of the international search report

20/07/2015

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INTERNATIONAL SEARCH REPORT

International application No

PCT/IB2015/053345

C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
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