United States Patent Office 3,322,554
Patented May 30, 1967

3,322,554

PROCESS FOR PREPARING ELECTRICALLY CONDUCTIVE FLOCK FOR ELECTROSTATIC FLOCKING

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No Drawing. Filed July 30, 1963, Ser. No. 298,568
Claims priority, application Germany, Aug. 11, 1962, S 80,884
19 Claims. (Cl. 117—17)

This invention relates to electrostatic flocking processes using man-made flock fibres.

The manufacture of plush, velvet, suede, and the like by the application of short fibres, called flock, to an adhesive-coated base under the influence of a high tension electrostatic field is a well known process, described for example in United States Patent Nos. 2,173,032 and 2,173,078. Hitherto, the fibres used for this purpose have been mainly rayon fibres. Recently, however, the use of other man-made fibres, such as fibres made from cellulose esters, polyamides, polyesters, and polyacrylonitriles, has become more prominent because of the good properties of these materials. For the manufacture of articles which are exposed to considerable wear and tear, such as carpets or upholstery, nylon flock has, for example, proved to be a very suitable material.

When using man-made fibres in electrostatic flocking processes, difficulties however arise, resulting from the different behaviour of the various materials. While rayon fibres, like natural cellulose fibres, are rather hydrophilic, fibres made from cellulose triacetate and many purely synthetic polymers are more or less hydrophobic and are easily given electrostatic charges by friction. It is known that such charges are very troublesome in ordinary spinning and weaving processes of fibres and yarns; they produce however especially great difficulties in electrostatic flocking processes.

In order to obtain a dense and uniform pile, the single flock fibres must be able to glide smoothly over each other and must flow easily through a metal sieve in the flocking apparatus. They must furthermore spring quickly between the two electrodes of the electric field, until they are finally trapped by the adhesive-coated base. The flow and gliding properties of flock can be improved by the application of softening agents, but a good spring capacity in the electric field depends above all on the electrical conductivity of the fibres.

If this conductivity is low, the flock fibres tend to cling to each other and to the electrodes and spring only slowly or not at all. Frequently, they also form thick, hard clusters of fibres, which cannot pass the sieve and become completely useless.

To overcome these difficulties, it has been suggested to carry out electrostatic flocking at high humidities, as it is known that the conductivity of the fibres depends on their moisture content. A high humidity is however detrimental to the formation of a high-tension electric field. Therefore, the problem is usually dealt with by treating the fibres with antistatic agents, which form on the fibres a coating of substances having a good electrical conductivity, a procedure long adopted in ordinary spinning and weaving processes. The antistatic agents proposed for this purpose belong to many different chemical classes, but most of them are ionized or at least polarized, substances, such as simple electrolytes or surface active agents of cationic, anionic, or non-ionic character. Mineral dispersions, e.g. of calcium carbonate or silica, sometimes mixed with oily lubricants, have also been recommended for this purpose.

Thus United States patent specification No. 2,917,401 describes the preparation of an anti-electrostatic agent made by the reaction of silicon tetrachloride with ethyl acetate and aqueous alcohol. French patent specification No. 1,257,894 proposes the uses of suspensions of montmorillonite, a mineral well known for its swelling properties in water, and French patent specification No. 1,157,657 describes, for the same purpose, mixtures of surface active agents with paraffin oil and silica. The antistatic effect of mixtures containing highly dispersed silica and oily lubricants is mentioned in an article in Melliand's Textilberichte, 33 (1952) 957.

All these anti-electrostatic agents are however only applicable to ordinary textile operations, such as spinning, twisting, and weaving. For the treatment of fibers for electrostatic flocking processes they are either ineffective or even disadvantageous, because of their content of oily or fatty lubricants. To avoid these difficulties, British Patent No. 686,101 proposes to treat rayon or triacetate fibres with aqueous dispersons of calcium carbonate, magnesia, or silica, and German patent specification No. 1,040,497 describes treatment of nylon flock with condensation products of higher fatty acids with aliphatic or aromatic aminosulphonic acids. Finally, German patent application 1,098,913 proposes the treatment of flock with well known anti-electrostatic agents, such as polyglycol esters or phosphoric acid esters with the addition of potassium and sodium salts. The present invention provides a process for electrostatic flocking with man-made fibres in which by a novel method good flow and gliding properties and a good spring capacity in an electrostatic field are imparted to the flock fibres as a new anti-electrostatic combination, while the mechanical properties of the fibres are not impaired.

According to the present invention a process for preparing pile fabrics such as plush, velvet, suede and the like comprise (1) treating man-made flock fibres with an aqueous solution of tannin of pH 4–5, adding potassium antimonyl tartrate to the said solution and continuing the said treatment, and separating and drying the said flock fibres, and (2) securing the flock fibres thus obtained to an adhesive-coated base under the influence of a high tension electrostatic field.

In this specification, the term "tannin" includes gallic acid and digallic acid as well as the various types of tannin.

The use of tannin and potassium antimonyl tartrate as auxiliaries in certain dyeing processes is known, e.g. in dyeing natural and regenerated cellulose fibres with basic dyes and for improving the wet fastness of acid wool dyes. It is also known to treat synthetic fibres and textiles, especially stockings, with solutions containing alum, tannin, and aluminium acetate to improve their durability. Synthetic fibres have also been treated with tannin solutions to make them smooth and waterproof. The use of tannin and potassium antimonyl tartrate as anti-electrostatic agents for man-made fibres has, however, not been described.

The treatment of man-made fibres with tannin and potassium antimonyl tartrate is very effective in producing fibres of good electrical conductivity which possess an excellent spring capacity in an electrostatic field. The treatment is equally applicable to dyed and undyed fibres, and an additional advantage is the durability of the antistatic effect. The fibres treated in accordance with the invention keep their good spring capacity even after prolonged storage; moreover, they are insensitive to changes of temperature and humidity of the air produced by changes of weather and seasons. Another advantage of the process is that the agents used need only be employed in very low concentrations.

A further improvement of the properties of the fibres for electrostatic flocking processes, especially of their flow-

and gliding properties, can be obtained, if, after their treatment with tannin and potassium antimonyl tartrate, the fibres are treated with an aqueous solution containing an anionic finishing agent, and, optionally, also a water-soluble alkali metal or ammonium salt. As the salt, sodium sulphate or ammonium sulphate may be used, and sodium silicate in the form of water-glass is also effective, especially if the solution is made faintly acid. A further modification is possible by treating the fibres before the treatment with the tannin solution with an aqueous solution of an alum, preferably potassium aluminum sulphate.

The known antistatic effects of these finishing agents and alkali metal salts are quite insufficient to impart to the flock fibres a good, durable spring capacity when used alone. Only in combination with the tannin and potassium antimonyl tartrate treatment do they serve to improve the flocking qualities of the fibres. Tannin may be replaced by gallic acid or digallic acid. Other phenolic compounds have however, a much smaller anti-static effect.

The combinations of agents which gives, in any particular case, the best effect depends on the nature, titre, and length of the fibres treated. Generally the treatment with tannin and potassium antimonyl tartrate is followed by a treatment with an anionic finishing agent, mixtures of cetyl alcohol and cetyl alcohol sulphonates being preferred.

To carry out the treatment of the invention, the flock fibres are dispersed in an aqueous solution at, e.g. about 70° C. and brought to a pH of 4–5, e.g. by the addition of acetic acid, and containing preferably between 0.04 and 0.2 percent by weight of tannin. Ordinarily the dispersion is stirred for 10 minutes, 0.01 to 0.05 percent by weight of potassium antimonyl tartrate is then added, and stirring is continued for another 10 minutes. To remove the adhering solution, the fibres are centrifuged for 10 minutes and finally dried. If it is desired to apply a finishing treatment, the drying is omitted, and the fibres are, following centrifuging, transferred to an aqueous solution at, e.g., about 50° C. containing preferably, between 0.2 and 1.0 percent by weight of the finishing agent and stirred for about 25 minutes. The fibres are then centrifuged again and dried.

If, in addition, alkali metal or ammonium salts are to be used, between 0.3 and 1.5 percent by weight of such substances is added to the finishing bath. If sodium silicate is employed as the alkali metal salt, it is recommended to prepare a solution of the required strength, and, prior to addition of the finishing agent, to add acetic acid, so that the pH of the solution is less than 7.5 and preferably between 4 and 5. It is further recommended to use, in combination with sodium silicate, the sodium salt of cetyl alcohol sulphonic acid as finishing agent. Otherwise, the preferred procedure is the same as if a finishing treatment without the addition of an alkali salt is applied.

If the flock fibres are to be pre-treated with an alum, the fibres are dispersed in an aqueous solution at, e.g. 70° C., brought to a pH of 4–4 by addition of acetic acid and containing between 0.02 and 0.1 percent by weight of an alum, usually potassium aluminum sulphate. The dispersion is then stirred for 10 minutes, and tannin and, subsequently, potassium antimonyl tartrate are added, the treatment being carried out, as described above, either with or without the use of anionic finishing agents and alkali metal or ammonium salts.

The weight ratio between fibres and solutions should vary between 1 to 15 and 1 to 25; usually 1 part of fibres requires 20 parts of solution. It is recommended, prior to treatment, to remove any oily or fatty sizes adhering to the fibres. This is simply done by washing the fibres in a warm bath containing soap or a synthetic detergent.

The man-made fibres treated according to the method described, are then secured to an adhesive-coated base under the influence of a high-tension electrostatic field in conformity with well-known processes.

The following examples illustrate the invention:

Example I

50 g. of polyhexamethylene adipamide fibres of 2.5 mm. length and a titre of 2.2 tex[1] are dispersed in 1000 g. of an aqueous solution at 70° C. containing 0.25 g. of 80% acetic acid and 0.5 g. tannin. The dispersion is stirred for 10 minutes, 0.25 g. of potassium antimonyl tartrate are added, and stirring is continued for another 10 minutes. To remove the adhering solution, the fibres are centrifuged for 10 minutes and dried at 25° C. The fibres possess a good spring capacity in an electrostatic field and are very suitable for use in electrostatic flocking processes.

Example II

50 g. of dyed polycaprolactam fibres of 2.5 mm. length and a titre of 2.2 tex are dispersed in 1000 g. of an aqueous solution at 70° C. containing 0.25 g. of 80% acetic acid and 0.5 g. tannin. The dispersion is stirred for 10 minutes, 0.25 g. potassium antimonyl tartrate is added, and stirring continued for another 10 minutes. The fibres are then centrifuged for 10 minutes and dispersed in 1000 g. of an aqueous solution at 50° C. containing 3 g. of a mixture of cetyl alcohol and an ammonium sulphonate of cetyl alcohol, and stirred for 25 minutes. To remove the adhering solution, the fibres are centrifuged for 10 minutes and dried at 25° C. The fibres possess a good flow and good spring capacity in an electrostatic field. They kept these properties after a storage for 6 months and after temporary heating to 70° C., and are very suitable for electrostatic flocking processes. Fibres treated only with the anionic finishing agent solution, did not possess any of these properties.

Example III

50 g. of dyed polyhexamethylene adipamide fibres of 1 mm. length and a titre of 0.3 tex are dispersed in 1000 g. of an aqueous solution at 70° C. containing 0.5 g. of 80% acetic acid and 0.5 g. of potassium aluminum sulphate. The dispersion is stirred for 10 minutes, 1 g. tannin is added, and stirring is continued for 10 minutes, whereupon 0.25 g. of potassium antimonyl tartrate are added and the dispersion is stirred for another 10 minutes. The fibres are centrifuged for 10 minutes and dispersed in an aqueous solution at 50° C. prepared as follows: 8 g. of water-glass of specific gravity 1.4 are mixed with 1000 g. of water, 80% acetic acid is added until a pH of 4.5 is reached, and finally, 3 g. of a mixture of cetyl alcohol and a sodium sulphonate of cetyl alcohol, are added. The fibres are stirred for 25 minutes in this solution, centrifuged for 10 minutes, and dried at 25° C. They possess a good flow and good spring capacity in an electrostatic field and kept these properties after a storage for 6 months and after temporary heating to 70° C. They are very suitable for electrostatic flocking processes.

Cellulose triacetate fibres of 0.5 mm. length and a titre of 0.3 tex, polyethylene terephthalate fibres of 1 mm. length and a titre of 0.3 tex, and polyacrylonitrile fibres of 2 mm. length and a titre of 1.5 tex, treated in the same way, also possess a good flow and good spring capacity in an electrostatical field, and are very suitable for electrostatic flocking processes. However, polyhexamethylene adipamide fibres, cellulose triacetate fibres, polyethylene terephthalate fibres and polyacrylonitrile fibres, which are only treated with an acidified aqueous solution containing water-glass and the foresaid anionic finishing agent, possess an unsatisfactory spring capacity in an electrostatic field and are unsuitable for electrostatic flocking processes.

We claim:

1. Process for preparing pile fabrics which comprises (1) treating flock fibres made of polymers selected from the group consisting of cellulose esters, polyamides, polyesters, and polyacrylonitriles, with an acidified aqueous

[1] To convert tex into denier, multiply by 9, so 2.2 tex equals 19.8 denier, etc.

solution containing tannin, adding potassium antimonyl tartrate to the said solution and continuing the said treatment, the amount of said tannin and the amount of said potassium antimonyl tartrate in said solution being sufficient to confer antistatic properties on said flock fibres so that said flock fibres possess good electrical conductivity and excellent spring capacity in an electrostatic field, and separating and drying the said flock fibres, and (2) securing the flock fibres thus obtained to an adhesive-coated base under the influence of a high tension electrostatic field.

2. Process for preparing pile fabrics which comprises (1) treating flock fibres made of polymers selected from the group consisting of cellulose esters, polyamides, polyesters, and polyacrylonitriles, with an aqueous solution of pH 4–5 containing 0.04 to 0.2 percent by weight of tannin, adding 0.01 to 0.05 percent by weight of potassium antimonyl tartrate to the said solution and continuing the said treatment, and separating and drying the said flock fibres, and (2) securing the flock fibres thus obtained to an adhesive-coated base under the influence of a high tension electrostatic field.

3. Process for preparing pile fabrics which comprises (1) treating flock fibres made of polymers selected from the group consisting of cellulose esters, polyamides, polyesters, and polyacrylonitriles, with an aqueous solution of pH 4–5 containing 0.04 to 0.2 percent by weight of tannin, adding 0.01 to 0.05 percent by weight of potassium antimonyl tartrate to the said solution and continuing the said treatment, separating the flock fibres and treating them with an aqueous solution of an anionic finishing agent, and drying the flock fibres, and (2) securing the flock fibres thus obtained to an adhesive-coated base under the influence of a high tension electrostatic field.

4. Process for preparing pile fabrics which comprises (1) treating flock fibres made of polymers selected from the group consisting of cellulose esters, polyamides, polyesters, and polyacrylonitriles, with an aqueous solution of pH 4–5 containing 0.04 to 0.2 percent by weight of tannin, adding 0.01 to 0.05 percent by weight of potassium antimonyl tartrate to the said solution and continuing the said treatment, separating the flock fibres and treating them with an aqueous solution containing 0.2 to 1 percent by weight of an anionic finishing agent, and drying the flock fibres, and (2) securing the flock fibres thus obtained to an adhesive-coated base under the influence of a high tension electrostatic field.

5. Process for preparing pile fabrics which comprises (1) treating flock fibres made of polymers selected from the group consisting of cellulose esters, polyamides, polyesters, and polyacrylonitriles, with an aqueous solution of pH 4–5 containing 0.04 to 0.2 percent by weight of tannin, adding 0.01 to 0.05 percent by weight of potassium antimonyl tartrate to the said solution and continuing the said treatment, separating the flock fibres and treating them with an aqueous solution containing 0.2 to 1 percent by weight of an anionic finishing agent, and 0.2 to 1.5 percent by weight of a water-soluble salt selected from the group consisting of alkali metal and ammonium salts and drying the flock fibres, and (2) securing the flock fibres thus obtained to an adhesive-coated base under the influence of a high tension electrostatic field.

6. Process for preparing pile fabrics which comprises (1) treating flock fibres made of polymers selected from the group consisting of cellulose esters, polyamides, polyesters, and polyacrylonitriles, with an aqueous solution of pH 4–5 containing 0.04 to 0.2 percent by weight of tannin, adding 0.01 to 0.05 percent by weight of potassium antimonyl tartrate to the said solution and continuing the said treatment, separating the flock fibres and treating them with an aqueous solution containing 0.2 to 1 percent by weight of an anionic finishing agent, and 0.2 to 1.5 percent by weight of a salt selected from the group consisting of sodium sulphate, sodium silicate, and ammonium sulphate and drying the flock fibres, and (2) securing the flock fibres thus obtained to an adhesive-coated base under the influence of a high tension electrostatic field.

7. Process for preparing pile fabrics which comprises (1) treating flock fibres made of polymers selected from the group consisting of cellulose esters, polyamides, polyesters, and polyacrylonitriles, first with an acidified aqueous solution containing an alum and then with an acidified aqueous solution containing tannin, adding potassium antimonyl tartrate to the said solution of tannin and continuing the said treatment, the amount of said tannin and the amount of said potassium antimonyl tartrate in said solution being sufficient to confer antistatic properties on said flock fibres so that said flock fibres possess good electrical conductivity and excellent spring capacity in an electrostatic field, separating the flock fibres and treating them with an aqueous solution of an anionic finishing agent, and drying the flock fibres, and (2) securing the flock fibres thus obtained to an adhesive-coated base under the influence of a high tension electrostatic field.

8. Process for preparing pile fabrics which comprises (1) treating flock fibres made of polymers selected from the group consisting of cellulose esters, polyamides, polyesters, and polyacrylonitriles, first with an aqueous solution of pH 4–5 containing 0.02 to 1.0 percent by weight of an alum and then with an aqueous solution of pH 4–5 containing 0.04 to 0.2 percent by weight of tannin, adding 0.01 to 0.05 percent by weight of potassium antimonyl tartrate to the said solution of tannin and continuing the said treatment, separating the flock fibres and treating them with an aqueous solution containing 0.2 to 1 percent by weight of anionic finishing agent, and drying the flock fibres, and (2) securing the flock fibres thus obtained to an adhesive-coated base under the influence of a high tension electrostatic field.

9. Process for preparing pile fabrics which comprises (1) treating flock fibres made of polymers selected from the group consisting of cellulose esters, polyamides, polyesters, and polyacrylonitriles, first with an aqueous solution of pH 4–5 containing 0.02 to 1.0 percent by weight of an alum and then with an aqueous solution of pH 4–5 containing 0.04 to 0.2 percent by weight of tannin, adding 0.01 to 0.05 percent by weight of potassium antimonyl tartrate to the said solution of tannin and continuing the said treatment, separating the flock fibres and treating them with an aqueous solution containing 0.2 to 1 percent by weight of an anionic finishing agent, and 0.2 to 1.5 percent by weight of a water-soluble salt selected from the group consisting of alkali metal and ammonium salts, and drying the flock fibres, and (2) securing the flock fibres thus obtained to an adhesive-coated base under the influence of a high tension electrostatic field.

10. Process for preparing pile fabrics which comprises (1) treating flock fibres made of polymers selected from the group consisting of cellulose esters, polyamides, polyesters, and polyacrylonitriles, first with an aqueous solution of pH 4–5 containing 0.02 to 1.0 percent by weight of potassium aluminum sulphate and then with an aqueous solution of pH 4–5 containing 0.04 to 0.2 percent by weight of tannin, adding 0.01 to 0.05 percent by weight of potassium antimonyl tartrate to the said solution of tannin and continuing the said treatment, separating the flock fibres and treating them with an aqueous solution containing 0.2 to 1 percent by weight of an anionic finishing agent and 0.2 to 1.5 percent by weight of a salt selected from the group consisting of sodium sulphate, sodium silicate, and ammonium sulphate, and drying the flock fibres, and (2) securing the flock fibres thus obtained to an adhesive-coated base under the influence of a high tension electrostatic field.

11. Process for preparing pile fabrics which comprises (1) treating flock fibres made of a polymer selected from the group consisting of polyhexamethylene adipamide, polycaprolactam, cellulose triacetate, polyethylene terephthalate, and polyacrylonitrile with an acidified aqueous solution of tannin, adding potassium antimonyl tartrate

to the said solution and continuing the said treatment, the amount of said tannin and the amount of said potassium antimonyl tartrate in said solution being sufficient to confer antistatic properties on said flock fibres so that said flock fibres possess good electrical conductivity and excellent spring capacity in an electrostatic field, and separating and drying the said flock fibres, and (2) securing the flock fibres thus obtained to an adhesive-coated base under the influence of a high tension electrostatic field.

12. Process for preparing pile fabrics which comprises (1) treating flock fibres made of a polymer selected from the group consisting of polyhexamethylene adipamide, polycaprolactam, cellulose triacetate, polyethylene terephthalate, and polyacrylonitrile with an aqueous solution of pH 4–5 containing 0.04 to 0.2 percent by weight of tannin, adding 0.01 to 0.05 percent by weight of potassium antimonyl tartrate to the said solution and continuing the said treatment, and separating and drying the said flock fibres, and (2) securing the flock fibres thus obtained to an adhesive-coated base under the influence of a high tension electrostatic field.

13. Process for preparing pile fabrics which comprises (1) treating flock fibres made of a polymer selected from the group consisting of polyhexamethylene adipamide, polycaprolactam, cellulose triacetate, polyethylene terephthalate, and polyacrylonitrile first with an acidified aqueous solution of an alum and then with an acidified aqueous solution containing tannin, adding potassium antimonyl tartrate to the said solution of tannin and continuing the said treatment, the amount of said tannin and the amount of said potassium antimonyl tartrate in said solution being sufficient to confer antistatic properties on said flock fibres so that said flock fibres possess good electrical conductivity and excellent spring capacity in an electrostatic field, separating the flock fibres and treating them with an aqueous solution of an anionic finishing agent, and drying the flock fibres, and (2) securing the flock fibres, thus obtained to an adhesive-coated base under the influence of a high tension electrostatic field.

14. Process for preparing pile fabrics which comprises (1) treating flock fibres made of polymer selected from the group consisting of polyhexamethylene adipamide, polycaprolactam, cellulose triacetate, polyethylene terephthalate, and polyacrylonitrile first with an aqueous solution of pH 4–5 containing 0.02 to 1.0 percent by weight of an alum and then with an aqueous solution of pH 4–5 containing 0.04 to 0.2 percent by weight of tannin, adding 0.01 to 0.05 percent by weight of potassium antimonyl tartrate to the said solution of tannin and continuing the said treatment, separating the flock fibres and treating them with an aqueous solution containing 0.2 to 1 percent by weight of an anionic finishing agent, and drying the flock fibres, and (2) securing the flock fibres thus obtained to an adhesive-coated base under the influence of a high tension electrostatic field.

15. Process for preparing pile fabrics which comprises (1) treating flock fibres made of a polymer selected from the group consisting of polyhexamethylene adipamide, polycaprolactam, cellulose triacetate, polyethylene terephthalate, and polyacrylonitrile first with an aqueous solution of pH 4–5 containing 0.02 to 1.0 percent by weight of potassium aluminum sulphate and then with an aqueous solution of pH 4–5 containing 0.04 to 0.2 percent by weight of tannin, adding 0.01 to 0.05 percent by weight of potassium antimonyl tartrate to the said solution of tannin and continuing the said treatment, separating the flock fibres and treating them with an aqueous solution containing 0.2 to 1 percent by weight of an anionic finishing agent and 0.2 to 1.5 percent by weight of a salt selected from the group consisting of sodium sulphate, sodium silicate and ammonium sulphate, and drying the flock fibres, and (2) securing the flock fibres thus obtained to an adhesive-coated base under the influence of a high tension electrostatic field.

16. Process for preparing flock fibres suitable for use in the preparation of pile fabrics by high tension electrostatic flocking which comprises treating flock fibres made of polymers selected from the group consisting of cellulose esters, polyamides, polyesters, and polyacrylonitriles, with an acidified aqueous solution containing tannin, adding potassium antimonyl tartrate to the said solution and continuing the said treatment, the amount of said tannin and the amount of said potassium antimonyl tartrate present in said solution being sufficient to confer antistatic properties on said flock fibres so that said flock fibres possess good electrical conductivity and excellent spring capacity in an electrostatic field, and separating and drying the said treated flock fibres.

17. Process for preparing flock fibres suitable for use in the preparation of pile fabrics by high tension electrostatic flocking which comprises treating flock fibres made of polymer selected from the group consisting of cellulose esters, polyamides, polyesters, and polyacrylonitriles, with an acidified aqueous solution containing an alum and then with an acidified aqueous solution containing tannin, adding potassium antimonyl tartrate to the said solution of tannin and continuing the said treatment, the amount of said tannin and the amount of said potassium antimonyl tartrate present in said solution being sufficient to confer antistatic properties on said flock fibres so that said flock fibres possess good electrical conductivity and excellent spring capacity in an electrostatic field, separating the flock fibres and treating them with an aqueous solution of an anionic finishing agent, and drying the said treated flock fibres.

18. A pile fabric produced by the process of claim **1**.

19. A pile fabric produced by the process of claim **7**.

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