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FABRICS AND LIKE MATERIALS

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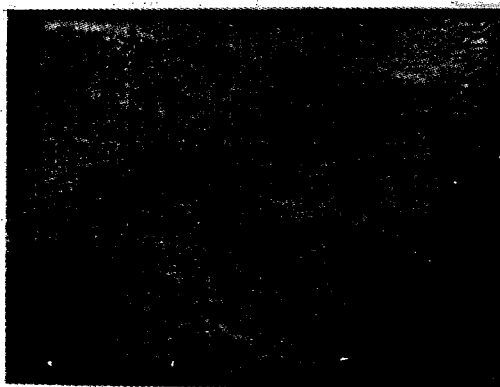


*Fig. 1.*



*Fig. 2.*

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## UNITED STATES PATENT OFFICE

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## FABRICS AND LIKE MATERIALS

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28 Claims. (Cl. 8—131)

This invention relates to the manufacture of fabrics and like materials, and to the manufacture of pile fabrics and analogous materials which are wind-proof or water-proof materials.

The present application is a continuation in part of our co-pending application S. No. 440,038, filed April 22, 1942.

We have found that a new effect may be obtained by subjecting pile fabrics, of which the pile comprises flat filaments of a cellulose derivative, to the crinkling action of a boiling aqueous medium. When treated in this way, the pile of such fabric is compacted and, if the treatment is applied while the fabrics are substantially free from tension, may assume a granulated appearance, which may resemble the appearance of animal pelts, e. g., lamb's wool.

The filaments from which the pile of fabrics to be treated according to the invention is made may be produced by any method which yields filaments of a bone-shaped or other relatively flat cross-section. For example, suitable filaments having a basis of cellulose acetate may be produced from spinning solutions in which the proportion of cellulose acetate is relatively low; thus a solution of about 21–22% of cellulose acetate in acetone may be spun under ordinary evaporative spinning conditions. Alternatively, more concentrated acetone solutions of cellulose acetate containing a proportion of paradichlorobenzene or other plasticiser may be spun. Again, filaments of flat cross-section may be produced by extruding acetone solutions of cellulose acetate into an evaporative medium, of which, in the neighbourhood of the extrusion orifices, the acetone concentration is maintained abnormally high.

Cellulose acetate has been mentioned above as the base material of the filaments, but it is to be understood that the invention may be applied to fabrics having a pile of fibres, the base of which is any other cellulose derivative or like material which is susceptible to the crinkling action of hot aqueous media, for example to fabrics having a pile of cellulose aceto-propionate or aceto-butyrate fibres.

The length of the pile may be varied over a wide range, depending upon the purpose for which the fabric is being produced. For example, in producing velvet fabric for use in light-weight textiles, the pile length may be of the order of  $\frac{1}{8}$ – $\frac{1}{4}$  inch, while for rugs the pile length may be as much as 1 inch, or up to  $1\frac{1}{2}$  or  $1\frac{3}{4}$  inch, or even more; it is found that the treatment according to the invention may reduce the depth of the pile by approximately one half.

The backing of the fabric may comprise cotton or other natural textile material, or a synthetic textile material, e. g., a material having a basis of regenerated cellulose, formed, for example, by the viscose or cuprammonium process or by saponification of a cellulose ester material. Alternatively, the backing may comprise some other sheet material, e. g., a flexible cellulosic film. The backing should be such that it is not adversely affected by the action of boiling water.

Long pile fabrics for treatment according to the invention may be produced by attaching bundles of staple fibres to the backing fabric by sewing, whereby the bulky effect which would result when bundles of staple fibre are knotted on to a backing may be avoided. When shorter pile products are required, the fabric may be produced by one of the more usual methods of producing pile fabrics, e. g., by weaving and subsequently cutting. The pile components of such fabrics may be made from continuous filament yarns, staplised yarns, formed, for example, by subjecting continuous filament yarns to abrasion, or yarns composed of a staple fibre of suitable length. Whatever the type of yarn which is employed for making the pile components, the twist should be kept as low as possible consistent with satisfactory processing.

The treatment of the pile fabrics may be carried out by immersing them in the bath of boiling aqueous medium for a period sufficient to produce the desired degree of crinkle, preferably for a period of at least 2 hours, and advantageously for a period of 4 hours or more, especially when no preliminary treatment designed to accelerate the crinkling action has been applied. An increased degree of crinkle may be secured in a corresponding period of treatment by passing the boiling liquid through the fabric from the back to the front, and this method is, therefore, to be preferred in cases in which the backing permits it. Other things being equal, when a fabric of which the pile has a basis of a cellulose ester is treated according to the invention, a similar degree of crinkle may frequently be obtained in a shorter period of boiling if, before the fabric is treated with the boiling bath, it is padded with a caustic soda solution having a concentration of approximately 0.25%, the liquor being expressed to about 75% increase in weight on the fabric. This caustic soda treatment appears to result in a slight superficial saponification of the pile fibres, and is particularly effective when the boiling bath contains Glauber's salts or other electrolyte. Such a caustic soda pretreatment leads

to a somewhat delustred product, but it has been found that treatment of the fabric with dilute hydrochloric acid, e. g., 1.5-2.0 gms. per litre, before boiling substantially restores the original lustre without impairing the crinkling effect.

When the pile fabrics are treated with boiling water alone for a period of 2 hours or more, besides the simple crinkling of the staple fibre, the fibre becomes somewhat delustred. A good matt lustre is obtainable by boiling in a liquor having a pH value of about 11. Should it be desired to delustrate the materials fully, a proportion of soap, e. g., a proportion of 2½ to 5 grams per litre, or other wetting agent may be incorporated in the treatment medium, while, if it is desired to counteract the delustring tendency, salts or sugars, and particularly Glauber's salts, may be added in small proportions to the water, e. g., a solution containing 20 grams of Glauber's salts per litre may be employed. By suitably modifying the composition of the aqueous treatment medium as indicated, it is possible to obtain a range of lustre or delustre suitable for a variety of purposes.

As indicated above, if the treatment with the boiling aqueous medium is carried out in the absence of tension, the pile face of the fabric acquires a granulated or patterned appearance, particularly if the treatment is carried on for about 4 hours or more. This granular effect depends for its degree upon the length of the pile, being generally less noticeable with the shorter piles. The pile length is advantageously at least about ¾ inch. Pile lengths of about ¾-1 inch, before treatment, yield products having the appearance of lambs' wool on the skin. The granulation is resistant to crushing or other distortion, and remains in the material after washing or other treatments to which it may be subjected in normal use. If desired, the fabric may be folded or creased, or the pile face thereof may be embossed, before the boiling water treatment with a view to pre-disposing the pile to crinkle in a particular pattern.

Fabrics may be treated locally according to the invention for the production of fancy effects. For example, by suitable stencils the boiling water may be prevented from acting on selected areas of the fabric, and the products will then show areas of crinkled pile with or without a modified lustre, alternating with areas of uncrinkled pile having the lustre of the original material.

After treatment with the aqueous medium according to the invention, the fabrics may be subjected to further treatments adapted to modify their appearance or other characteristics as may be desired. Thus, they may be treated with a reagent adapted to modify the chemical composition of the pile and/or of the backing materials. For example, a fabric comprising yarns having a basis of cellulose or a cellulose derivative containing free hydroxyl groups, e. g., acetone-soluble cellulose acetate, may be subjected to the action of an adipylating agent. Again, such a fabric may be impregnated with a dilute aqueous solution of an etherifying agent, such as for example the pyridinium compound of octadecyloxy-methyl chloride, and subsequently dried and baked at about 120° C. By such a treatment as that last-mentioned long pile fabrics of which the pile comprises acetone-soluble cellulose acetate fibres and which have been treated according to the invention may have their somewhat "scroopy" handle reduced, and may be given a yellowish appearance, a reduced lustre and a somewhat

greasy handle, such as is usually associated with undressed sheepskin.

The production of wind- or water-proof materials according to the invention may be achieved by impregnating the backing of the pile fabric, preferably after the pile has been crinkled by the treatment with a boiling aqueous medium, with a suitable hydrophobe material. Rubber, applied for example as rubber latex, is an example of such a material, but we prefer to employ synthetic hydrophobe materials. Thus, according to the present invention the backing may be impregnated with synthetic resins, among which may be mentioned the alkyd resins and derivatives of polyvinyl alcohol or of polyacrylic or polymethacrylic acids. Again, the backing may be impregnated with synthetic rubber-like materials, such as, for example, an inter-polymer of two or more dienes, of a diene and acrylo-nitrile, of a diene and styrene, or of a diene and isobutylene; suitable dienes are, for example, 1,3-butadiene, isoprene and chloroprene. Another class of materials which may be employed according to the invention is that of cellulose derivatives, and it has been found that when back-filling with the same cellulose derivative as that present in the pile of the treated fabric the operation may frequently be carried out without special precautions to protect the roots of the cellulose derivative pile during such treatment. The cellulose derivative employed may be, for example, cellulose acetate, cellulose butyrate, cellulose aceto-butyrate, cellulose aceto-stearate, or other simple or mixed cellulose ester of an organic acid, or a cellulose ether, e. g., ethyl cellulose, hexyl cellulose or benzyl cellulose; cellulose ether-esters may also be employed.

The method of application of the filling agent will largely depend upon the nature of the agent, but broadly comprises spreading a paste, emulsion or a thickened solution of the agent over the back of the fabric, e. g., by means of a roller or spreading knife, removing the dispersion medium or solvent, and, if necessary, heating or otherwise treating the filling agent to increase its water-resistance and to reduce any residual tackiness. For example, a thermo-setting synthetic resin may be applied from solution or dispersion in an organic solvent while in the partially condensed condition, and the treated fabric may be subjected to heat in order to convert the resin into the thermoset insoluble state. Again, the backing of a fabric may be impregnated with a solution of a monomeric or partially polymerised derivative of vinyl alcohol, acrylic acid or methacrylic acid, and the fabric then subjected to treatment which will convert the monomer or partial polymer to a highly polymerised condition in which it is resistant to water.

Plasticisers, pigments and other substances may be incorporated in the pastes or solutions with a view to modifying the flexibility, colour or other characteristics of the products. The back-filling composition may have the same colour as that of the pile, or a different one.

While the filling agent is in general applied after the fabric has been treated with a boiling aqueous medium according to the process described above, it may be applied before such treatment, provided that the agent or another component of the composition is not adversely affected by the treatment.

Fabrics produced according to the invention are suitable for a number of purposes, such for example as linings, fancy fabrics, rugs, carpets and

in substitution for lambs' wool, e. g., as shuttle linings and roller clearers. Their suitability for the various purposes will of course be affected by the filament denier of the pile as well as by its length. For example, pile of filament denier greater than 10 has a somewhat rough handle, but is very resistant to crushing, and proves suitable for linings, e. g., for linings for heavy coats to be worn in cold climates; with pile filament deniers varying from 3 to 7 the fabrics are suitable for dress fabrics and outer-wear fabrics generally; while the finest deniers, e. g., 1 or 2, give very soft surfaces, and fabrics having pile of such deniers may be employed for lining oxygen and other masks which are required to fit the face tightly. Pile fabrics treated according to the present invention may be employed for heat insulating purposes, particularly when two or more fabrics are superimposed.

The accompanying figures illustrate the invention. Figure 1 is a photographic representation of a fabric produced according to Example 1, and Figure 2 is a photographic representation of a glove made up from fabric produced according to Example 2.

As examples of particular methods of applying the present invention, the following examples are given:

#### Example 1

A pile fabric is made up having a ground warp of 2/120's cotton yarn (136 ends per inch), a pile warp of 170 denier yarns of 26 cellulose acetate filaments of flat cross-section having a twist of 0.5 turn per inch (68 ends per inch) and a ground weft of 70's cotton (90 picks per inch), the pile length being  $\frac{3}{8}$  inch. The fabric is boiled for 4 hours in an aqueous bath containing  $2\frac{1}{2}$  grams of soap per litre, and is then dried and subjected to fan beating. The fabric possesses a soft pile finish with a slightly granular appearance, the pile strongly resisting crushing.

#### Example 2

A pile fabric is produced having a ground warp of 2/70's cotton yarn (136 ends per inch), a pile warp of 172 denier yarns of 26 cellulose acetate filaments of flat cross-section having a twist of 0.5 turn per inch (68 ends per inch), and a ground weft of 32's cotton (78 picks per inch), the pile length being  $\frac{3}{8}$  inch. This fabric is padded through an aqueous solution of 2.5 grams caustic soda per litre, expression being adjusted to 75% increase in weight, and is dried and then boiled for 2 hours in an aqueous bath containing 20 grams per litre of Glauber's salts. It is then dried, padded with a liquor containing 40 grams of octadecyl-oxymethyl pyridinium chloride per litre and 8 grams of sodium acetate per litre, expression being adjusted to 80% increase in weight, stenter-dried at 80° C. and stoved for 2 minutes at 140° C. After washing and re-drying the fabric is finally subjected to fan beating. It is then found to possess the appearance of lambs' wool on the skin, and may, for example, be employed in the production of gloves and in substitution for lambs' wool in shuttle linings. In the production of gloves from such a fabric, two sheets of fabric may be placed back to back so that both the outside and the inside of the glove present a pile surface.

#### Example 3

A mixture of 10 parts by weight of toluene, 10 parts by weight of alcohol and 66 parts by weight

of tricresyl phosphate is mixed with 200 parts by weight of the synthetic resin sold under the trade name "Bedafin 2001"; the resulting mixture is gradually added to 400 parts by weight of kaolin, while grinding to produce a smooth paste. 400 parts of the plasticiser sold under the trade name "Bedafin 285X" is then stirred into this paste, followed by 70 parts by weight of water. A paste is thus produced which has an excellent consistency for back-filling and which has a white opaque appearance which is retained in the finished fabric. The "Bedafin" compounds referred to are marketed by the Imperial Chemical Industries Limited.

A pile fabric produced according to Example 1 or 2 is given two back-filling treatments with the paste on a mangle under conditions such that about 10-15% of the paste, based on the weight of the fabric, is applied thereto. The fabric is dried by passing over steam-heated rolls, and after stoving for about 2 minutes at 140° C. is found to be substantially wind- and water-proof. Any residual tackiness in the product may be reduced by the application of talc.

#### Example 4

A solution of 45 parts by weight of cellulose acetate in 135 parts by weight of acetone is thoroughly stirred into a paste of 150 parts by weight of kaolin and 175 parts by weight of di-(methyl glycol) phthalate. 60 parts of water is stirred into the resulting paste, and the composition so produced is applied to a pile fabric as described in Example 3, in quantity about 13-20% based on the weight of the fabric. The fabric is dried by passing over steam-heated rolls. The product is highly wind- and water-proof.

#### Example 5

A solution of 45 parts by weight of ethyl cellulose, having an ethoxy content of 46.5-48.5% and a viscosity of about 250 centipoises, in 60 parts by weight of methylated spirits and 60 parts by weight of solvent naphtha containing 45 parts by weight of castor oil is thoroughly mixed with a suspension of 75 parts of French chalk in 60 parts by weight of solvent naphtha. Three coatings of the resulting composition are applied to the backing of a pile fabric, as described in Example 3, the fabric being dried by passing over steam-heated rolls.

Having described our invention what we desire to secure by Letters Patent is:

1. Process for the production of effect fabrics resembling animal pelts in appearance, which comprises subjecting a pile fabric, of which the pile comprises flat filaments of a cellulose ester of an organic acid, to the crinkling action of a boiling aqueous medium while the fabric is substantially free from tension.

2. Process for the production of effect fabrics resembling animal pelts in appearance, which comprises passing a pile fabric, of which the pile comprises flat filaments of a cellulose ester of an organic acid, through a caustic soda bath to produce at most a slight superficial saponification of the pile, and thereafter subjecting the fabric to the crinkling action of a boiling aqueous medium.

3. Process for the production of effect fabrics having a granulated appearance, which comprises subjecting a pile fabric, of which the pile comprises flat filaments of a cellulose ester of an organic acid and of which the pile length before treatment is at least about  $\frac{3}{8}$  inch, to the crinkling action of a boiling aqueous medium.

4. Process for the production of wind- or water-proof effect fabrics, which comprises impregnating the backing of a pile fabric, of which the pile comprises flat filaments of a cellulose ester of an organic acid, with a hydrophobe coating material, and subjecting the fabric to the crinkling action of a boiling aqueous medium.

5. Process for the production of wind- or water-proof effect fabrics, which comprises subjecting a pile fabric, of which the pile comprises flat filaments of a cellulose ester of an organic acid, to the crinkling action of a boiling aqueous medium, and thereafter impregnating the backing of the said pile fabric with a synthetic hydrophobe coating material.

6. Process for the production of wind- or water-proof effect fabrics, which comprises impregnating the backing of a pile fabric, of which the pile comprises flat filaments of a cellulose ester of an organic acid, with a synthetic hydrophobe coating material, and thereafter subjecting the fabric to the crinkling action of a boiling aqueous medium.

7. Process for the production of effect fabrics resembling animal pelts in appearance, which comprises subjecting a pile fabric, of which the pile comprises flat filaments of a cellulose acetate, to the crinkling action of a boiling aqueous medium for at least 2 hours while the fabric is substantially free from tension.

8. Process for the production of effect fabrics resembling animal pelts in appearance, which comprises subjecting a pile fabric, of which the pile comprises flat filaments of a cellulose acetate and of pile length at least about  $\frac{3}{4}$  inch, to the crinkling action of a boiling aqueous medium for at least 2 hours while the fabric is substantially free from tension.

9. Process for the production of effect fabrics resembling animal pelts in appearance, which comprises passing a pile fabric, of which the pile comprises flat filaments of a cellulose acetate, through a caustic soda bath, and then subjecting it to the crinkling action of a boiling aqueous medium, for at least 2 hours, while the fabric is substantially free from tension, any saponification of the pile of the fabric due to said caustic soda treatment being at most slight and superficial.

10. Process for the production of effect fabrics resembling animal pelts in appearance, which comprises passing a pile fabric, of which the pile comprises flat filaments of a cellulose acetate, through a caustic soda bath, next treating the fabric with dilute hydrochloric acid, and then subjecting it to the crinkling action of a boiling aqueous medium for at least 2 hours, while the fabric is substantially free from tension, any saponification of the pile of the fabric resulting from said caustic soda treatment being at most slight and superficial.

11. Process for the production of effect fabrics resembling animal pelts in appearance, which comprises passing a pile fabric, of which the pile comprises flat filaments of a cellulose acetate, to the crinkling action of a boiling aqueous medium containing a wetting agent, for at least 2 hours, while the fabric is substantially free from tension.

12. Process for the production of effect fabrics resembling animal pelts in appearance, which comprises passing a pile fabric, of which the pile comprises flat filaments of a cellulose acetate, through a caustic soda bath, and then subjecting it to the crinkling action of a boiling aqueous

medium containing a wetting agent, for at least 2 hours, while the fabric is substantially free from tension, any saponification of the pile of the fabric due to said caustic soda treatment being at most slight and superficial.

13. Process for the production of effect fabrics resembling animal pelts in appearance, which comprises passing a pile fabric, of which the pile comprises flat filaments of a cellulose acetate, to the crinkling action of a boiling aqueous medium containing an agent adapted to counteract the delustering tendency of the medium for at least 2 hours, while the fabric is substantially free from tension.

14. Process for the production of effect fabrics resembling animal pelts in appearance, which comprises passing a pile fabric, of which the pile comprises flat filaments of a cellulose acetate, through a caustic soda bath, and then subjecting it to the crinkling action of a boiling aqueous medium containing an agent adapted to counteract the delustering tendency of the medium, for at least 2 hours, while the fabric is substantially free from tension, any saponification of the pile of the fabric due to said caustic soda treatment being at most slight and superficial.

15. Process for the production of effect fabrics resembling animal pelts in appearance, which comprises passing a pile fabric, of which the pile comprises flat filaments of a cellulose acetate, to the crinkling action of a boiling aqueous medium containing a small proportion of Glauber's salts, for at least 2 hours, while the fabric is substantially free from tension.

16. Process according to claim 7, wherein the treatment with the boiling aqueous medium is applied only to selected areas of the fabric.

17. Process according to claim 9, wherein the treatment with the boiling aqueous medium is applied only to selected areas of the fabric.

18. Process for the production of effect fabrics resembling animal pelts in appearance, which comprises subjecting a pile fabric, of which the pile comprises flat filaments of a cellulose acetate, to the crinkling action of a boiling aqueous medium for at least 2 hours while the fabric is substantially free from tension and thereafter subjecting the fabric to a further treatment with an adipylating agent.

19. Process for the production of effect fabrics resembling animal pelts in appearance, which comprises subjecting a pile fabric, of which the pile comprises flat filaments of a cellulose acetate, to the crinkling action of a boiling aqueous medium for at least 2 hours while the fabric is substantially free from tension and thereafter subjecting the fabric to a further treatment with a dilute aqueous solution of an etherifying agent.

20. Process for the production of effect fabrics resembling animal pelts in appearance, which comprises subjecting a pile fabric, of which the pile comprises flat filaments of a cellulose acetate, to the crinkling action of a boiling aqueous medium for at least 2 hours while the fabric is substantially free from tension and thereafter treating the fabric with an aqueous solution of the pyridinium compound of octadecyl-oxy-methyl chloride, drying the fabric, and subsequently baking it at about 120° C.

21. Process for the production of wind- or water-proof effect fabrics, which comprises impregnating the backing of a pile fabric, of which the pile comprises flat filaments of a cellulose derivative, with a synthetic resin, and subjecting

the fabric to the crinkling action of a boiling aqueous medium.

22. Process according to claim 21, wherein the synthetic resin is produced in the backing by polymerization of a substance selected from the group consisting of polymerizable derivatives of vinyl alcohol, acrylic acid, methacrylic acid and partial polymers thereof.

23. Process for the production of wind- or water-proof effect fabrics, which comprises impregnating the backing of a pile fabric, of which the pile comprises flat filaments of a cellulose derivative, with an inter-polymer of a diene and another polymerizable unsaturated compound, and subjecting the fabric to the crinkling action of a boiling aqueous medium.

24. Process for the production of wind- or water-proof effect fabrics, which comprises impregnating the backing of a pile fabric, of which the pile comprises flat filaments of a cellulose derivative, with a cellulose derivative, and subjecting the fabric to the crinkling action of a boiling aqueous medium.

25. Process for the production of wind- or

water-proof effect fabrics, which comprises impregnating the backing of a pile fabric, of which the pile comprises flat filaments of a cellulose derivative, with an ethyl cellulose, and subjecting the fabric to the crinkling action of a boiling aqueous medium.

26. Effect fabrics which resemble animal pelts in appearance and which comprise a pile fabric, of which the pile comprises crinkled flat filaments of a cellulose ester of an organic acid.

27. Effect fabrics having a granulated appearance and which comprise a pile fabric of which the pile comprises crinkled flat filaments of a cellulose ester of an organic acid, the uncrinkled length of the pile being at least about  $\frac{3}{8}$  inch.

28. Effect fabrics which resemble animal pelts in appearance and which comprise a pile fabric, of which the pile comprises crinkled flat filaments of a cellulose ester of an organic acid, having a backing impregnated with a hydrophobe coating material.

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