

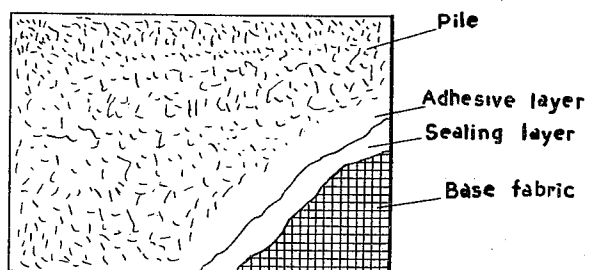
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PROCESS OF MAKING PILE FABRIC

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## PROCESS OF MAKING PILE FABRIC

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This invention relates to process of making pile fabric.

The invention provides a method of bonding a flock of textile fibres to a base fabric to give a pile fabric. According to the process of the invention, the base fabric is first provided with a sticky coating of a thermoplastic polymer and a mixture of two plasticizers therefor of substantially different volatilities, the staple fibre is then applied to this coating so as to adhere thereto, and by heating the coated fabric to cause preferential evaporation of the more volatile plasticiser, the sticky coating is converted into a non-sticky flexible coating. The term "plasticizer" in this specification means a substance capable of incorporation with the thermoplastic polymer to give a composition of greater flexibility, softness and extensibility than the polymer alone, and having a vapour pressure at 100° C. not greater than 50 mm. of mercury.

In making the pile fabrics of the invention, the base fabric may be provided with a sticky coating of the plasticised thermoplastic polymer by application of a solution thereof in a volatile solvent, this solvent being evaporated before application of the staple-fibre. The staple-fibre which is to form the pile may be applied to the surface of the adhesive coating by any suitable method, for example by brushing, sifting or beating or blowing, and the adhesive may then be converted to the flexible, non-sticky form by heating the coated fabric under such conditions that a major proportion, e. g. 60 to 70 or 80% or more of the more volatile plasticiser is lost by evaporation without substantially reducing the quantity of less volatile plasticiser.

The accompanying drawing shows a pile fabric made according to the invention with portions of the pile, the adhesive layer, and the sealing layer removed to show the construction.

The following examples illustrate the invention:

*Example 1*

A madapolam fabric is coated with an acetone solution of an adhesive of the composition: 50 parts polyvinyl acetate, 10 parts tricresyl phosphate, and 40 parts dimethyl phthalate, the parts being by weight. The coating is effected by extruding the composition, on to the travelling fabric, excess of the composition being removed by a doctor blade positioned between two guide bars over which the fabric passes. This arrangement enables the thickness of the coating to be controlled between narrow limits and minimises

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the effect of any slubs or joins in the cloth to be coated.

The acetone is evaporated from the coating at a temperature of 50° C. by passing the coated fabric through a festoon drier, means being provided for recovery of the acetone vapours.

To the sticky coating is applied a flock of regenerated cellulose fibres of fibre-length about 1 mm. and denier 8 dyed in a bright colour. The flock is sifted on to the travelling fabric and distributed by means of a beater, care being taken that the beater blade does not come into actual contact with the surface of the adhesive. The pile may at this stage, if desired, be set (i. e. the fibres may be caused to lie all in one direction) by brushing, care being taken to avoid damaging the sticky coating by having the brush too close.

The fabric is now stoved at about 150° C. for 15–20 minutes. This removes a major proportion (over 60%) of the dimethyl phthalate without substantially reducing the proportion of tricresyl phosphate present, so converting the adhesive to the non-sticky, flexible form and firmly anchoring the pile.

*Example 2*

The process is carried out as in Example 1, but before applying the polyvinyl acetate composition the fabric is spread with a thin coating of 60 parts of polyvinyl chloride and 40 parts of tricresyl phosphate: this serves to prevent the subsequently applied polyvinyl acetate composition from penetrating the fabric.

*Example 3*

The process is carried out as in Example 1, but using a cellulose acetate staple-fibre of the same fibre length and denier as the regenerated cellulose fibre of that example.

*Example 4*

The process is carried out as in Example 3, except for the following differences:

- (1) Glycol diacetate is used instead of dimethyl phthalate;
- (2) The staple fibre is blown in a stream of air on to the sticky coating of the fabric;
- (3) Preferential evaporation of the glycol diacetate is carried out by air drying at room temperature for 3 days, in the course of which over 80% of the glycol diacetate is lost.

Instead of the above specific adhesive compositions, other adhesive compositions may be employed, as for example acetone solutions of poly-

vinyl acetate, dimethyl phthalate or glycol diacetate, and tricresyl phosphate in the proportions of 60 to 90 parts by weight of dimethyl phthalate or glycol diacetate and 20 to 30 parts by weight of tricresyl phosphate per 100 parts by weight of polyvinyl acetate.

To facilitate the breaking up of the flock into individual fibres during application, the flock should be substantially free from oil. It may, as indicated above, be dyed, and the adhesive may also be of the same or a different colour, suitable pigments or dyestuffs being incorporated for this purpose.

Instead of polyvinyl acetate other water-insoluble thermoplastic polymers, especially oxygen-containing vinyl polymers and  $\alpha$ -substituted acrylic ester polymers, can be used. Examples of such polymers are: polyvinyl acetals (including partial acetals, i. e. acetals which retain a proportion of the ester groups present in the polyvinyl ester from which they are derived by hydrolysis and acetalisation and/or a proportion of the hydroxyl groups resulting from the hydrolysis) in which the aldehyde component may be, for example, formaldehyde, acetaldehyde, propionaldehyde or butyraldehyde; polyacrylic esters, for instance polyethyl acrylate, and poly *n*-butyl acrylate; and polymethacrylic esters, e. g. polymethyl methacrylate. It is, of course, necessary that the polymer should be capable of being rendered sticky by the plasticiser mixture. To meet this condition the presence of side-chains or side-rings in the polymer, as in the compounds specified above, and the absence of strong inter-molecular forces, is of advantage. Thus, polymers having very strong inter-molecular forces such as polyacrylonitrile, polyvinylidene chloride and poly-tetra-fluor-ethylene are unsuitable, and vinyl chloride polymers, if used at all, should be of relatively low molecular weight and/or contain substantial proportions of a co-polymerised monomer giving a less polar side-chain, e. g. vinyl acetate or other vinyl ester, or an ester of maleic acid or of acrylic acid, or a vinyl alkyl ketone. Co-polymers of more than two kinds of unsaturated monomer can be used as can mixtures of two or more compatible polymers. In addition to the capability of being rendered sticky by the plasticiser, a further requirement is that the polymer, after evaporation of more volatile plasticiser, shall give a flexible non-sticky coating, and to meet this requirement the factors referred to above as making for stickiness in the initial polymer-plasticiser mixture, viz. relatively low molecular weight, low inter-molecular forces and the presence of side-groups adapted to space apart the polymer chains, must not be unduly predominant. The presence in the polymer molecule of a proportion of groups that increase inter-molecular attraction, in addition to a proportion of groups tending to reduce that attraction by spacing the chains apart, may be of advantage not only in achieving the desired degree of hardness in the polymer, but also in facilitating adhesion to the base fabric and to the pile. Polymers of this kind can be made, for example, by co-polymerising vinyl acetate with butyl acrylate and acrylic acid. Further examples are acetals containing free hydroxyl and free ester groups. Although it is possible to anchor the pile by the use of a polymer which is without specific adhesion for the material either of the pile or of the backing fabric, it is of great advantage for there to be some specific adhesion between the polymer and these materials, i. e. some

attraction between elements or groups in the polymer and elements or groups in both of the textile materials. From this point of view polymers which are without any active group, e. g. polyisobutylene, are defective. The various oxygen-containing polymers referred to above, however, show specific adhesion to cellulose, including regenerated cellulose, probably on account of hydrogen bonding between hydroxyl hydrogen of the cellulose and the ether or carbonyl oxygen of the polymer. This is of great advantage in bonding a cellulose pile to a cellulose backing. To a less extent this advantage is obtained also when either the pile or the base fabric is of a partially hydrolysed cellulose ester such as the cellulose acetate of commerce. When however one of the components (basic fabric or pile) is a highly water-resistant material containing carbonyl groups, e. g. ester or amide groups, and the other component contains hydroxyl groups, it is preferable to use a polymer containing some hydroxyl groups in addition to ester or ether groups such as the co-polymer of vinyl acetate, butyl acrylate and acrylic acid referred to above as the basis of the adhesive. Ternary co-polymers of the kind last mentioned are capable of wide application to a variety of materials, e. g. they are very suitable for bonding natural silk pile to a cotton fabric. One further property which is desirable in the polymer is that it should be soluble in a volatile solvent for the plasticisers used. This enables the base fabric to be coated from a solution of polymer and plasticisers in the volatile solvent which can then be evaporated off.

Instead of dimethyl phthalate, or glycol diacetate, there may be used as the more volatile plasticiser triethyl phosphate, tributyl phosphate, triacetin, triethyl citrate and other plasticisers of comparable volatility at temperatures between 100 and 150° C. Instead of tricresyl phosphate, triphenyl phosphate or other plasticiser of comparable volatility within the specified temperature range may be used. A convenient criterion of the volatility of the plasticisers is given by their vapour pressures at the temperature at which evaporation of the more volatile plasticiser is effected. At this temperature the vapour pressure of the more volatile plasticiser is preferably at least fifty times that of the less volatile and may be considerably higher, e. g. one hundred times or more. It is generally preferable, with a view to speed, to carry out the evaporation at about 120 to 150° C. and when so doing the vapour pressure of the less volatile plasticiser at 150° C. should not be greater than 0.1 mm. of mercury, while that of the more volatile plasticiser should be at least 1 mm. at that temperature. As illustrated, however, in Example 4 above, by choice of a sufficiently volatile plasticiser such as glycol diacetate, it is possible to evaporate the more volatile plasticiser over a longer period at a lower temperature, when this is more convenient. Naturally the more volatile plasticiser must not be so volatile as to be lost to a substantial extent in evaporation of the volatile solvent in which the adhesive composition is applied, and on the other hand must be sufficiently volatile to enable a major proportion of this plasticiser to be evaporated during a heat treatment that can be effected without damaging the materials. Both the less volatile and the more volatile plasticisers must, of course, be compatible with the thermoplastic polymer. Preferably the more volatile plasticiser has a greater softening power than the less volatile one. It is

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of advantage for the plasticiser of lower volatility to be of a relatively high viscosity, for example to have a viscosity not less than about 50 centistokes at 20° C. The viscosity of the more volatile plasticiser is preferably somewhat lower, for example less than 20 and preferably less than 15 centistokes at the same temperature. The weight-ratio of the more volatile to the less volatile plasticiser should be not less than 3:1 and is preferably somewhat higher, e. g. 4:1.

The base fabric is preferably of cotton or regenerated cellulose. Fabrics of high tenacity regenerated cellulose fibre such as is obtainable by saponification of cellulose acetate fibre which has been highly stretched in moist steam, hot water or an organic swelling agent, for example to give a tenacity of 3-6 or more grams per denier, may be employed and are of particular advantage where lightness of weight is important. Naturally, the volatile solvent from solution in which the adhesive is applied, must not be a solvent or strong swelling agent for the material of the base fabric. Thus, for example, when a base fabric of cellulose acetate is used, acetone cannot be used to dissolve the adhesive. A suitable volatile solvent when the base fabric is cellulose acetate is a mixture of 60 parts by volume of methyl ethyl ketone with 40 parts of methyl isobutyl ketone.

The flock used to provide the pile may, as indicated above, be of regenerated cellulose or cellulose acetate, or may be of other fibres, for example cotton, silk, or a water-insoluble fibre-forming derivative of cellulose other than cellulose acetate, e. g. cellulose propionate, cellulose butyrate, cellulose acetate-propionate, cellulose acetate-butyrate, cellulose acetate-stearate, ethyl cellulose and benzyl cellulose. Or the flock may be of a synthetic fibre-forming polymer such as nylon, polyethylene terephthalate, polyvinyl chloride-acetate, Saran, or a co-polymer of acrylonitrile with vinyl chloride or methacrylonitrile.

The invention has been described with particular reference to the production of pile fabrics. The adhesives described above, however, are believed to be broadly novel. The invention includes these adhesives and solutions and the use of the adhesives and compositions in the manufacture of products other than pile fabrics. The adhesives and solutions may, for example, be used to form water-resistant coatings on textile materials. By the application of the adhesive in sticky form to the fabric followed by stiffening by preferential evaporation of the more volatile plasticiser, a coating can be obtained which is more strongly adherent than could be obtained by the use of the polymer plasticised with the less volatile plasticiser alone, and which is harder and more permanent in respect of its physical properties than if it contained substantial proportions of the more volatile plasticiser. The adhesives and compositions of the invention may be used to bond together textile materials of various kinds, especially materials having a basis of cellulose, regenerated cellulose or a water-insoluble cellulose derivative under conditions in which preferential evaporation of the more volatile plasticiser can be effected.

As indicated above, the preferred adhesive is a composition which comprises a solution in a volatile solvent of an oxygen-containing polyvinyl compound or an alpha-substituted polyacrylic ester, together with two plasticisers, one having a vapour pressure at least fifty times that of the other, the weight-ratio of the more vola-

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tile to the less volatile plasticiser being at least 3:1. The best results have been obtained when the polymer is polyvinyl acetate, the less volatile plasticiser is tricresyl phosphate, and the more volatile plasticiser is dimethyl phthalate (for high temperature evaporation) or glycol diacetate (for lower temperature evaporation).

Having described my invention, what I desire to secure by Letters Patent is:

10 1. Process for the production of a pile fabric by bonding staple fibres to a base fabric, which comprises applying to said base fabric a sticky coating substantially free from volatile solvent and comprising a water-insoluble thermoplastic polyvinyl resinous material and two plasticizers for the polyvinyl resinous material, one plasticizer having a vapor pressure not greater than 0.1 mm. of mercury at 150° C., and the second plasticizer having a vapor pressure of about 5 mm. of mercury to about 10 mm. of mercury at said temperature, the weight ratio of the second plasticizer to the first plasticizer being at least 3:1, feeding the staple fibre onto said sticky coating, and converting said coating into a non-sticky flexible coating by heating the coated fabric to a temperature between 100 and 150° C. for a period of time sufficient to drive off at least 60% of the second plasticizer without substantially reducing the quantity of the first plasticizer.

30 2. Process for the production of a pile fabric by bonding staple fibres to a base fabric, which comprises applying to said base fabric a sticky coating substantially free from volatile solvent and comprising polyvinyl acetate and two plasticizers for the polyvinyl acetate, one plasticizer having a vapor pressure not greater than 0.1 mm. of mercury at 150° C., and the second plasticizer having a vapor pressure of about 5 mm. of mercury to about 10 mm. of mercury at said temperature, the weight ratio of the second plasticizer to the first plasticizer being at least 3:1, feeding the staple fibre onto said sticky coating, and converting said coating into a non-sticky flexible coating by heating the coated fabric to a temperature between 100 and 150° C. for a period of time sufficient to drive off at least 60% of the second plasticizer without substantially reducing the quantity of the first plasticizer.

50 3. Process for the production of a pile fabric by bonding staple fibres to a base fabric, which comprises applying to said base fabric a sticky coating substantially free from volatile solvent and comprising a polyvinyl acetal and two plasticizers for the polyvinyl acetal, one plasticizer having a vapor pressure not greater than 0.1 mm. of mercury at 150° C., and the second plasticizer having a vapor pressure of about 5 mm. of mercury to about 10 mm. of mercury at said temperature, the weight ratio of the second plasticizer to the first plasticizer being at least 3:1, feeding the staple fibre onto said sticky coating, and converting said coating into a non-sticky flexible coating by heating the coated fabric to a temperature between 100 and 150° C. for a period of time sufficient to drive off at least 60% of the second plasticizer without substantially reducing the quantity of the first plasticizer.

70 4. Process for the production of a pile fabric by bonding staple fibres to a base fabric, which comprises applying to said base fabric a sticky coating substantially free from volatile solvent and comprising a water-insoluble thermoplastic polyvinyl resinous material, dimethyl phthalate and tricresyl phosphate in the weight ratio of at

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least 3:1, feeding the staple fibre onto said sticky coating, and converting said coating into a non-sticky flexible coating by heating the coated fabric to a temperature between 100 and 150° C. for a period of time sufficient to drive off at least 60% of the dimethyl phthalate without substantially reducing the quantity of tricresyl phosphate.

5. Process for the production of a pile fabric by bonding staple fibres to a base fabric, which comprises applying to said base fabric a sticky coating substantially free from volatile solvent and comprising a water-insoluble thermoplastic polyvinyl resinous material, glycol diacetate and tricresyl phosphate in the weight ratio of at least 3:1, feeding the staple fibre onto said sticky coating, and converting said coating into a non-sticky flexible coating by heating the coated fabric to a temperature between 100 and 150° C. for a period of time sufficient to drive off at least 60% of the glycol diacetate without substantially reducing the quantity of tricresyl phosphate.

6. Process for the production of a pile fabric by bonding staple fibres to a base fabric, which comprises applying to said base fabric a sticky coating substantially free from volatile solvent and comprising polyvinyl acetate, dimethyl phthalate and tricresyl phosphate in the weight ratio of at least 3:1, feeding the staple fibre onto said sticky coating, and converting said coating into a non-sticky flexible coating by heating the coated fabric to a temperature between 100 and 150° C. for a period of time sufficient to drive off at least 60% of the dimethyl phthalate without substantially reducing the quantity of tricresyl phosphate.

7. Process for the production of a pile fabric by bonding staple fibres to a base fabric, which comprises applying to said base fabric a sticky coating substantially free from volatile solvent and comprising polyvinyl acetate, glycol diacetate and tricresyl phosphate in the weight ratio of at least 3:1, feeding the staple fibre onto said sticky coating, and converting said coating into a non-sticky flexible coating by heating the coated fabric to a temperature between 100 and 150° C. for a period of time sufficient to drive off at least 60% of the glycol diacetate without substantially reducing the quantity of tricresyl phosphate.

8. Process for the production of a pile fabric by bonding textile staple fibres to a base fabric, which comprises first applying on said base fabric a layer of plasticized polyvinyl chloride, then superimposing on said layer a sticky coating substantially free from volatile solvent and comprising a water-insoluble thermoplastic polyvinyl resinous material and two plasticizers for said polyvinyl resinous material, one plasticizer having a vapor pressure not greater than 0.1 mm. of mercury at 150° C., and the second plasticizer having a vapor pressure of about 5 mm. of mercury to about 10 mm. of mercury at said temperature, the weight ratio of the second plasticizer to the first plasticizer being at least 3:1, feeding the staple fibre onto said sticky coating, and convert-

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ing said coating into a non-sticky flexible coating by heating the coated fabric to a temperature between 100 and 150° C. for a period of time sufficient to drive off at least 60% of the second plasticizer without substantially reducing the quantity of the first plasticizer.

9. Process for the production of a pile fabric by bonding textile staple fibers to a base fabric which comprises coating a cotton fabric with an acetone solution of polyvinyl acetate, dimethyl phthalate and tricresyl phosphate in the proportions of 60 to 90 parts by weight of dimethyl phthalate and 20 to 30 parts by weight of tricresyl phosphate per 100 parts by weight of polyvinyl acetate, evaporating off the acetone at a temperature not greater than about 50° C., applying a flock of staple fibres to the sticky coated fabric to produce the desired pile, and heating the fabric at a temperature of 120° C. to 150° C. for a period of time sufficient to drive off at least 60% of the dimethyl phthalate and to convert the sticky coating into a flexible non-sticky layer without substantially reducing the quantity of tricresyl phosphate.

10. Process for the production of a pile fabric by bonding textile staple fibres to a base fabric which comprises coating a cotton fabric with an acetone solution of polyvinyl acetate, glycol diacetate and tricresyl phosphate in the proportions of 60 to 90 parts by weight of glycol diacetate and 20 to 30 parts by weight of tricresyl phosphate per 100 parts by weight of polyvinyl acetate, evaporating off the acetone at a temperature not greater than about 50° C., applying a flock of staple fibres to the sticky coated fabric to produce the desired pile, and heating the fabric at a temperature of 120 to 150° C. for a period of time sufficient to drive off at least 60% of the glycol diacetate and to convert the sticky coating into a flexible non-sticky layer without substantially reducing the quantity of tricresyl phosphate.

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## REFERENCES CITED

The following references are of record in the file of this patent:

## UNITED STATES PATENTS

Number	Name	Date
286,039	Mayall	Oct. 2, 1883
574,332	Johnston	Dec. 28, 1896
1,895,711	Foley	Jan. 31, 1933
2,031,629	Atkins	Feb. 25, 1936
2,046,386	Strain	July 7, 1936
2,057,671	Dreyfus	Oct. 20, 1936
2,098,788	Morgan	Nov. 9, 1937
2,112,030	Klinkenstein	Mar. 22, 1938
2,188,396	Semon	Jan. 30, 1940
2,245,708	Patton	June 17, 1941
2,290,193	Kirkpatrick	July 21, 1942
2,327,123	Renfrew et al.	Aug. 17, 1943
2,358,227	Hiers	Sept. 12, 1944
2,428,716	McGill et al.	Oct. 7, 1947