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Steel

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[54] **PROCESS FOR PIGMENTING A PILE SURFACED PRODUCT**

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[52] **U.S. Cl.** **264/164,** 156/72, 156/230, 156/238, 156/277, 161/67, 264/243, 264/284

[51] **Int. Cl.** **B29c 17/02,** B32b 5/00

[58] **Field of Search** 156/72, 238, 240, 241, 156/277; 161/62, 67; 117/38; 8/2.5, 4, 180; 264/164, 280, 284, 241, 243

[56] **References Cited**

UNITED STATES PATENTS

3,027,595	4/1962	Takai et al.	161/62
3,235,438	2/1966	Wisotzky	161/62
3,236,712	2/1966	Mason	156/277
3,390,403	6/1968	Van Tilburg	161/67
3,399,425	9/1968	Lemelson	161/62
3,434,861	3/1969	Luc	156/240
3,600,260	8/1971	Watanabe	161/62
3,632,291	1/1972	Defago et al.	8/2.5
3,666,397	5/1972	Datye et al.	8/2.5
3,696,183	10/1972	Steel et al.	161/62
3,702,752	11/1972	Bent et al.	8/2.5
3,707,346	12/1972	Markert et al.	8/2.5

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[57] **ABSTRACT**

A method of pigmenting a laminated pile surfaced product in which the pile is formed by a tack spinning process. Pigment is applied to a component of the laminate to be subjected to the tack spinning process before the pile is formed.

16 Claims, 5 Drawing Figures

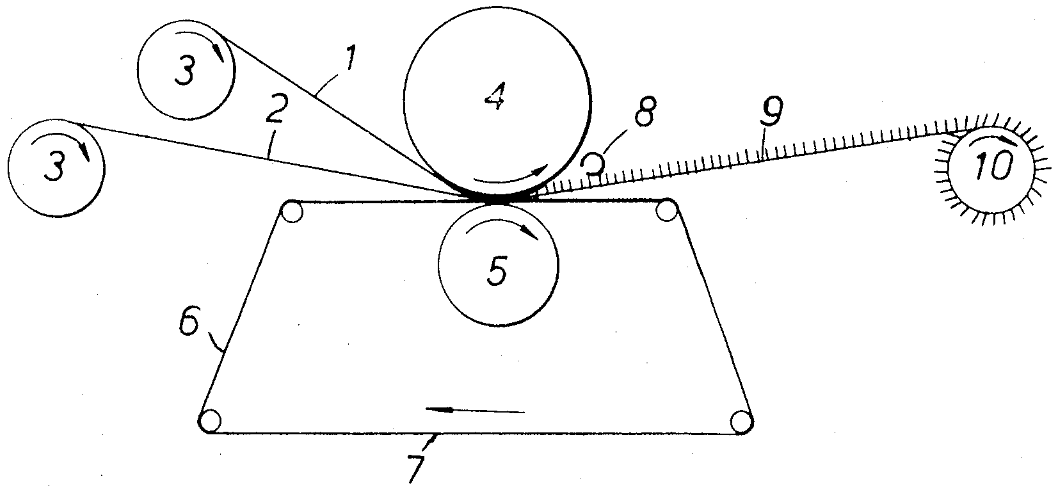


FIG.1.

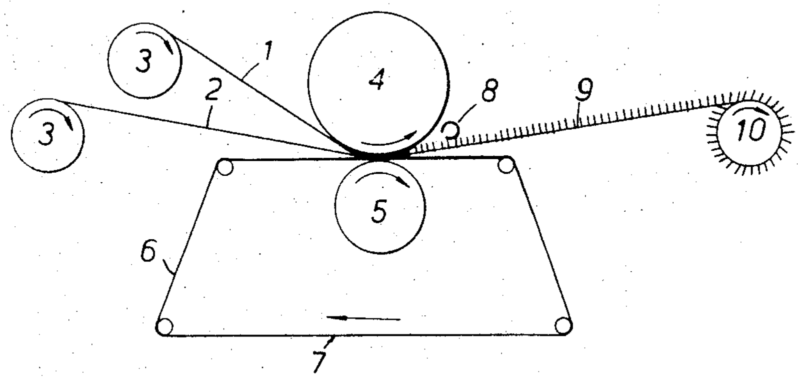


FIG.2.

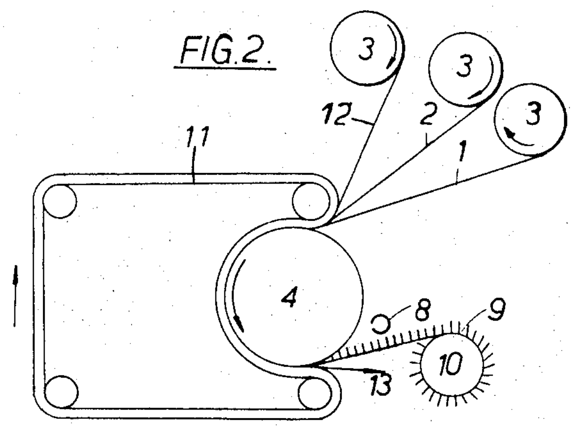


FIG.3.

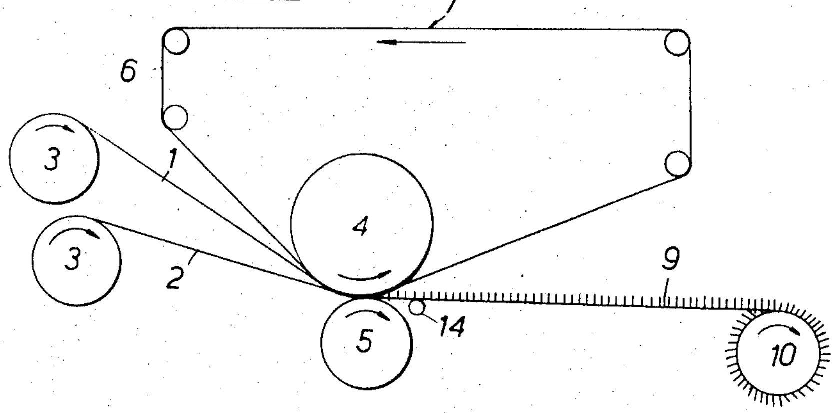


FIG.4.

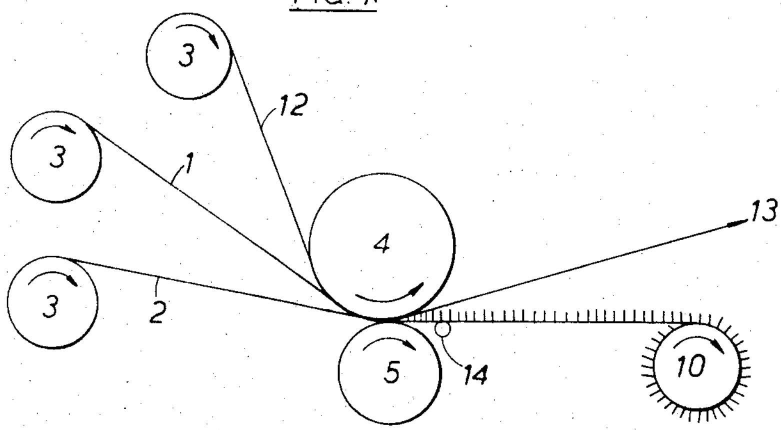
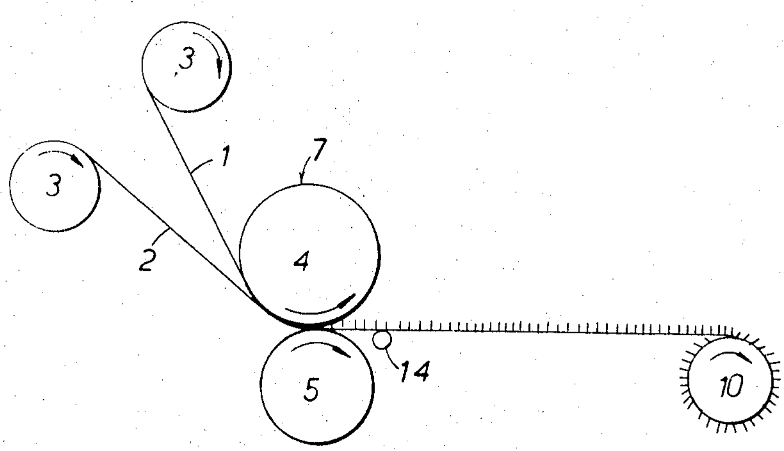


FIG.5.



PROCESS FOR PIGMENTING A PILE SURFACED PRODUCT

This invention relates to composite products.

Our copending U.K. Patent Applications Nos. 53265/69, 55242/70, 61325/70, 61326/70, 12336/71 and 28206/71 relate to the production of pile-surfaced products by drawing a pile comprising a plurality of fibres or tufts of fibres of polymeric material. Such a process in which a pile is formed from polymeric material on a foundation sheet or web may be referred to, for convenience, as "tack spinning."

The processes described in the specifications of the aforementioned applications comprise the steps of interposing a polymeric material between a web (for convenience the polymer and web are referred to as the laminate) and a temporary anchorage surface (the polymeric material being in a state such that it is tacky and capable of adhering to the web and also adhering temporarily to the temporary anchorage surface), separating the web and the temporary anchorage surface so that 'stringing' of the polymeric material occurs with the production of fibres of the polymeric material, hardening the polymeric material by cooling (if it is thermoplastic and had been rendered tacky by heat) or by completion of a cross-linking reaction (where the polymer is a curable polymer and undergoes cure during or after fibre formation), and separation of the fibres from the temporary anchorage surface.

The present invention provides a modification of pile-forming processes involving the processes set out above, in that the product comprises a pigment which is soluble in, adsorbent upon or dispersible in the polymeric material under the pile-drawing conditions employed, or is easily removable from the web for example by mere mechanical forces occuring during pile formation so that on formation of the pile the pigment is carried into the fibres of the pile. By use of the word pigment it will be realised that we do not exclude the use of soluble (whether in water or other solvent) dyestuffs, or volatile colouring materials, whether inorganic or organic in nature.

Accordingly the invention provides a method of pigmenting a pile surfaced product in which the pile is formed by a tack-spinning process, which method comprises the steps of applying pigment to a component of the laminate to be subjected to the tack spinning process before the pile is formed.

The pigment employed may be dispersed within the fabric of the web, as in conventional dip dyeing processes, it may merely be applied to the surface of the web, in which case it may be applied in solution or as a suspension of solid pigment particles in a suspending fluid, and/or it may be applied to the polymeric material. The pigment may be distributed evenly to produce a 'self coloured' product or it may be distributed in a discontinuous fashion, either randomly or to a predetermined pattern, and pigments of different colour may also be employed as in the production of multicoloured patterned products.

When the web is pigmented it will be apparent that if desired it may be pigmented immediately before contacting it with the polymeric material, application of the pigment may be effected on the same apparatus as that in which pile formation occurs.

The pigment applied to the web may be soluble in the in the polymeric material, or any component, including

solvent, of the polymer composition, employed in pile formation.

Where the pigment is insoluble in the polymer component and is carried into the fibres from the web by flow currents in a mechanical fashion the only requirement is that the pigment should be particulate so that it may be so carried, and that it be sufficiently loosely adhered to the web that it may be dislodged from it and carried into the pile under the pile drawing conditions employed. It may be desirable to apply pigment to the web in the presence of a bonding agent which may enhance its adhesion to the web but which, under the pile drawing conditions employed, releases the pigment into the polymeric pile forming material. Thus, where pile formation involves the application of heat to the web a thermoplastic bonding agent may be employed which softens to release the pigment at the temperature employed.

A preferred method of applying pigment to one or more components of the laminate is by a transfer printing process, optinally while the polymeric material is in contact with the temporary anchorage surface used to form the pile in the tack-spinning process. Transfer printing may involve the transfer of pigment by a combination of heat and pressure of a volatilisable disperse dye which is capable of dyeing the polymeric material forming the pile. Dyestuffs particularly suitable for this application are those listed in the Colour Index Second Edition Vol 1, p. 1,655 et seq. and generally are dyes without water-solubilising groups and with appreciable solubility. These volatilisable dyes are well known from the literature, for example U.K. Pat. Specification Nos. 1,189,026 and 1,211,149, and generally are volatilisable, or sublime, at a temperature between 140° and 230°C. The dye may be applied to the surface of a suitable substrate, for example paper or metal foil, for example by known printing methods, and then transferred to the material to be coloured.

The disperse dye may be applied to the polymeric component or it may be applied also or alternatively to the web, and particularly where the web is porous (i.e., has a structure such that the disperse dye may pass through it upon being made volatile e.g., a textile fabric) the disperse dyestuff may be applied to the side of the web distant from the polymeric material, migration of the dyestuff through the web occurring under the appropriate conditions.

In another embodiment, the substrate bearing the dyestuff acts as the temporary anchorage in the pile formation step, the dyestuff being transferred to the newly formed pile from the temporary anchorage prior to and/or during pile formation.

The time during which transfer of the disperse dye occurs will vary with the dyestuff, but generally will be considerably less than 30 seconds, and conditions will be chosen appropriately.

Web materials employed according to the invention include any of those mentioned in the aforementioned patent applications, but particularly preferred is paper, for example in the production of pile-surfaced wall paper, and textile materials of natural or synthetic fibre, which may be employed for the production, for example of patterned pile-surfaced textiles for clothing, furniture covering and the like.

Embossing techniques as described in the aforementioned patent specifications may also be employed in the present invention, to produce figured products in which embossed regions may or may not coincide with patterning obtained by the process of the present invention. Equally, embossing techniques may be employed to impart a surface pattern to self coloured pile-surfaced products. Preferred embossing techniques involve for example the prevention of pile drawing locally by preventing adhesion of the polymeric material to the temporary anchorage surface e.g. by embossing said surface or by interposing between the temporary anchorage surface and the polymeric pile forming material masking material of appropriate pattern. Said masking material is preferably applied to the temporary anchorage surface for example as a coating of suitable non-stick material. Local melting or softening of the pile whereby it is collapsed in predetermined or random areas may produce an embossed effect.

The attached drawings show in diagrammatic sectional elevation different types of apparatus suitable for carrying out the process of the invention in which disperse dyes are employed.

In FIG. **1**, a film of thermoplastic material **1** and a woven fabric web **2** are supplied from feed rolls **3** to the nip between contrarotating rollers **4,5.** The roller **4** is heated and forms the temporary anchorage surface from which the fibrils are drawn, and the roller **5** is a resilient backing roll. A continuous flexible metal strip **6** passes through the nip between the web **2** and the roller **5,** and at a point **7** is printed with a disperse dye by means not shown. In the nip of the rollers, the dye is transfer printed to the back of the web, migrates through the web and into the thermoplastic layer, and is drawn up into the fibrils formed when the web parts from the hot-roll. The fibrils of the pile are cooled by a jet of fluid from a nozzle **8,** and the coloured pile surface material **9** is taken up on a roll **10,** while the flexible strip **6** returns for further printing at **7.**

It may be necessary for the substrate bearing the disperse dye to remain in contact with the web for a longer period than that provided by the passage through the nip of the rollers. This may be achieved as shown in FIG. **2,** in which a resilient belt **11** replaced the backing roller **5** and urges the substrate against the web over approximately half of the circumference of the hot roller **4.** The substrate in this case is transfer paper previously printed with a pattern of disperse dye. The transfer paper **12** is fed between the web **2** and the resilient belt **11,** with the printed side towards the web. During the half revolution of the hot roll the transfer printing and migration of the dye through the web **2** and into the thermoplastic material **1** takes place, and when fibrils are drawn the web leaves the hot roll, the dye is present in the fibrils. The used transfer paper is stripped off at **13.**

FIG. **3** shows apparatus in which the substrate is a flexible metal strip **6,** printed with disperse dye **7,** which passes between the hot-roll **4** and the thermoplastic material **1.** Leaving the nip between the hot roll **4** and the resilient backing roll **5,** fibrils are drawn between the web **2** and the metal strip **6,** which acts as the temporary anchorage and at the same time the dye is transferred to the thermoplastic. A cooled tube **14** contacting the underside of the web **2** provides the necessary cooling of the drawn fibrils. The strip **6** recirculates for further printing.

FIG. **4** shows a further embodiment in which a dye-coated transfer paper **12** is supplied from a feed roll **3** between the hot roll **4** and the thermoplastic material **1,** with the coated face away from the hot roll. Fibrils are drawn from the transfer paper as temporary anchorage, used paper being pulled off at **13.** Finally in FIG. **5,** dye is printed directly upon the hot roll by means not shown, but which could for example involve contacting the hot roll with the coated side of dye-coated transfer paper, and the dye is transferred to the polymeric material **1** from the hot roll and appears in the drawn fibrils.

It may be advantageous to retain some solvent in the printed dye at the point where the substrate heating the dye comes in contact with the web or the polymeric material, as this may ensure greater mobility of the dye into the polymer. Different disperse dyes have different mobilities, and conditions of temperature, pressure and contact time must be selected appropriately for the dye to be used and for the nature of the web and the polymer. Where the substrate bearing the dye acts as the temporary anchorage in the pile-drawing step, it is desirable to avoid high local concentrations of dye which may interfere with the adhesion of the polymeric material to the substrate.

The invention will be further described with reference to the following Examples, in Nos. 1,2,3 and 4 of which the apparatus used was a Kodak 15 TC glazing machine incorporating a hot roll with a resilient belt as illustrated diagrammatically in FIG. **2.** The hot roll was at 180°C and the contact time was approximately 2 minutes.

Example 1

A sheet of polyethylene coated fabric was fed between the roll and the belt of the glazing machine, the polyethylene coating contacting the hot roll. A sheet of paper previously printed by conventional printing techniques with a pattern of disperse dyes (the printing ink employed comprises one or more disperse dyes, a liquid medium and a thickener or binder which is soluble in the liquid. Water or organic solvents, e.g., alcohols, are typical liquid media; in this example the dyes employed were three disperse dyes, each used in 8 percent solution in isopropanol/toluene/ethyl cellulose solution. The dyes employed were:

2-hydroxy-5-methyl-4′-acetylaminoazobenzene, 1:4-diamino-2-methoxyanthraquinone, 1-hydroxy-4-(p-toluidino)anthraquinone), was fed between the coated fabric and the belt, with the printed side in contact with the fabric. On stripping the fabric from the hot roll and cooling the side nearer the hot roll with a blast of air at ambient temperature, a pile-surfaced fabric was obtained in which the pattern originally on the transfer paper appeared both in the fabric and in the fibrils of the pile.

Example 2

The process of Example 1 was repeated except that in place of previously printed transfer paper, a PTFE-coated release paper was coated locally with solutions of the same three disperse dyes as used in Example **1,** dried partially or competely and fed into the glazing machine as before.

The pile-surfaced product showed the colours of the three dyes both in the web and the pile. Better results were obtained if the solvent was not allowed to evaporate fully before the release paper contacted the fabric.

Example 3

Polyethylene coated fabric was fed between the hot roll and the belt as described in Example 1 and previously printed transfer paper was fed between the polyethylene and the hot roll, with the printed surface contacting the polyethylene. On forming a pile surface as described in Example 1 by stripping the fabric from the transfer paper, the pattern originally on the transfer paper appeared in the fibrils of the pile.

Example 4

The three dyes listed in Example 1 were applied directly to the hot roll, and the polyethylene coated fabric fed in as described. On stripping the fabric from the hot roll a pile-surfaced product was obtained in which the pile showed colouring due to the dyes applied to the hot roll.

Example 5

A piece of newspaper printed with printer's carbon black ink, had applied to one printed surface a sheet of colourless polyethylene 0.1 mm and a polyethylene pile was produced on the paper by pressing the polyethylene against a polished stainless steel sheet at 115°C for 10 seconds. On parting the paper from the steel fibres of polyethylene formed between the paper and the steel, and on being cooled by a stream of cold air the fibres separated from the steel surface to form a pile on the paper. It was found that the carbon black had migrated in the polymer so that black fibrils occurred in regions corresponding to printed areas in the original newsprint.

Example 6

Example 5 was repeated using a white porous paper to which had been applied a thin coating of a finely ground coloured mineral-based pigment immediately before contacting the pigmented paper with the polyethylene sheet. The pigment migrated into the polyethylene pile, giving a coloured pile surface to the paper.

Example 7

Example 6 was repeated using a bleached kraft paper which had been printed with a disperse dye of the type described in Example 1. This was contacted with polyethylene sheet. The dyestuff sublimed into the polyethylene pile giving a coloured pile surface to the paper.

Example 8

Bleached kraft paper and disperse dye printed polyethylene film were fed together around a heated roll with the non-printed side adjacent to the hot roll. Tack-spinning was accomplished in the normal way to give a coloured pile product.

What we claim is:

1. A process for the production of a pile surfaced product pigmented with a given pigment comprising passing a synthetic thermoplastic material having at least one surface which is free of the given pigment and a backing material to a heated pressing surface with said one surface of the synthetic material against said pressing surface under conditions such that if the synthetic thermoplastic material is not prebonded to the backing material bonding occurs between the two and the temperature of the pressing surface being such that the synthetic thermoplastic material temporarily adheres to said heated pressing surface, applying the given pigment to at least one of the synthetic material or the backing material prior to separating the synthetic thermoplastic material from said surface, and then separating the synthetic thermoplastic material from said heated pressing surface so that fibrils are drawn out from the synthetic thermoplastic material, the fibrils becoming pigmented with the given pigment under the influence of the action of said heated pressing surface on the synthetic thermosplastic material.

2. A process according to claim **1** in which the pigment is applied to the backing material or the synthetic thermoplastic material in such a way that the pigment is carried into the pile during pile formation.

3. A process according to claim **1** in which the pigment is applied so that its distribution is discontinuous.

4. A process according to claim **1** in which application of the pigment occurs immediately prior to pile formation.

5. A process according to claim **1** in which the pigment is soluble in the synthetic thermoplastic material forming the pile.

6. A process according to claim **1** in which the pigment is finely particulate material insoluble but dispersible in the synthetic thermoplastic material forming the pile.

7. A process according to claim **6** in which the pigment is applied to a web component and bound loosely thereto by means of a temporary bonding agent which in the pile forming process releases the pigment which thereupon is carried into the pile.

8. A process according to claim **1** in which the pigment is transferred from a carrier to either the backing material or the synthetic thermoplastic material.

9. A process according to claim **8** in which the pigment is printed on to the synthetic thermoplastic material by said heated pressing surface.

10. A process according to claim **8** in which the pigment employed is a volatilisable dispersion dye.

11. A tack-spinning process for making a pile surfaced product pigmented with a given pigment comprising passing a laminate of a synthetic thermoplastic material component having at least one surface which is free of the given pigment and a backing material component through a heated nip formed between the surfaces of two moving elements one of which is heated to a temperature at which it temporarily adheres to said one surface of the synthetic thermoplastic material, feeding the given pigment into the nip simultaneously with the laminate so that the action of the nip causes migration of the pigment within the laminate, and separating the laminate from the surfaces of the moving elements under conditions such that the synthetic thermoplastic material is drawn into fibrils thereby forming a pile surfaced product and such that simultaneously the pigment is carried into the fibrils as they are formed.

12. A process as in claim **11** wherein the given pigment is present in or on the surface of said synthetic thermoplastic material component which is opposite said one surface thereof, before said laminate enters the nip.

13. A process as in claim **11** wherein said given pigment is present in or on said one surface of one of the moving elements.

14. A process as in claim **13** wherein said pigmented surface of said one moving element engages said one surface of said synthetic thermoplastic material component in said nip.

15. A process as in claim **13** wherein said pigmented surface of said one moving element engages said backing material component in said nip.

16. A process as in claim **11** wherein said given pigment is present in or on said backing material component, before said laminate enters the nip.

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