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Striegler et al.

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[54] **BONDED NON-WOVEN FIBRE FLEECE**

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[52] **U.S. Cl.** **161/169,** 161/150, 161/157

[51] **Int. Cl.** **B32b 27/00**

[58] **Field of Search** 161/150, 157, 170, 169

[56] **References Cited**

UNITED STATES PATENTS

3,560,318 2/1971 Miller et al. 161/170

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[57] **ABSTRACT**

A high bulk bonded non-woven fibre fleece containing natural and/or synthetic fibres and binder fibres, which are adhesive at higher temperatures, said binder fibres consisting of a saponified ethylene-vinyl acetate copolymer, the molar ratio of vinyl acetate to ethylene in said copolymer before saponification being in the range of 1:1 to 1:20. In the process to produce the bonded fleece, the fibres containing 5 to 50 percent by weight of binder fibres are formed to a non-woven fleece and the formed fleece is heated to temperatures of above 110° C, to render adhesive the binder fibres.

2 Claims, No Drawings

BONDED NON-WOVEN FIBRE FLEECE

This invention relates to a non-woven fibre fleece which has been bonded with thermoplastic adhesive fibres based on saponified ethylene-vinyl acetate copolymers and to a process for producing these non-woven fibre fleeces.

It is known that staple fibre fleeces which have been produced wet or dry on conventional machines such as carding machines, paper machines, random fleece forming machines and which may subsequently have been mechanically reinforced, for example, by means of stitching machines or by knit stitching techniques involving intimate looping of the fibres may be subjected to a subsequent treatment with synthetic resins. In this treatment, the synthetic resin acts mainly at the points of intersection of the fibres and by bonding the fibres it improves the mechanical properties and especially the tensile strength. This additional strengthening of the fleece, which has become known in technical terminology as "chemical strengthening," improves the resistance of the material to stress in practical use, for example the tearing stresses to which a fleece lining is exposed in the process of chemical cleaning. The synthetic resins mentioned above may be applied by various methods, for example by full bath impregnation or spraying with dispersion binders, or solutions of synthetic resins may equally well be applied by dipping or spraying. Furthermore, thermoplasts may be incorporated in the substrate in the form of powders or fibres and melted by means of heat so that bonds are produced at the points of intersection of the fibres, and when the fleece cools these bonding points effect durable even if not heat resistant bonding of the substrate.

Bonding with thermoplastic fibres affords various advantages, including the greater ease of incorporation by mixing the thermoplastic fibres with the main fleece-forming fibres by carding the two types of fibres together to produce a fibrous web and then leasing it to produce a fleece of high bulk. With this method it is very easy to mix the two types of fibres evenly. It is also very easy to control the quantity of binder by weighing the fibres components. Spraying or impregnating with synthetic resin dispersions, on the other hand, invariably entails difficulties. These difficulties are due to uneven application or distribution of the binder, especially in the vertical direction. Moreover, in the case of spraying with synthetic resins dispersions it is often not possible to cause the dispersion to penetrate from the surface into the interior of the fleece and effect permanent bonding of the fibres there. The consequence of this is that the fleece splits very easily. This disadvantage is to a large extent eliminated by incorporating thermoplastic fibres.

The bonding of fibre fleeces by means of thermoplastic bonding fibres is known per se. Bonding fibres of polyethylene, polypropylene, polyvinyl chloride and copolymers thereof, for example with ethylene and/or vinyl acetate are used for this purpose. Copolyamides and spun cellulose acetates or cellulose acetobutyrate are also customarily used for this purpose. These thermoplasts have, however, various disadvantages which impair the efficiency of the finished articles in use. Thus, for example, fibres of polyolefines or polyvinyl chloride and its copolymers are not sufficiently resistant to chlorinated hydrocarbons used in dry cleaning

with the result that the article partly or completely dissolves or becomes brittle (by extraction of the plasticiser). Fibres of mixed polyamides, for example, are not sufficiently resistant to washing processes because the swelling of the synthetic resin binder with water weakens the bonding of the fibre material and the textile bond, therefore, dissolves in the wash.

Another difficulty is that the softening point of the fibre material used for the bond is, in most cases, very high so that a high temperature is required for working up. This results in reduced production rates in cases where the fleece which is to be bonded is made of fibres which cannot be exposed to a certain temperature without damage. This is particularly disadvantageous in cases where it is necessary to bond, e.g., a fleece of natural fibres such as wool because in the case of wool, for example, the fibres undergo yellowing at elevated temperature, for example at 160° C. and in addition considerable damage to the wool is observed.

The object of this invention is a high bulk non-woven fibre fleece material from natural and/or synthetic fibres and binder fibres, consisting 5 to 50 percent by weight, based on the weight of fibres in the fibre fleece, of binder fibres from a saponified ethylene-vinyl acetate copolymer containing vinyl acetate and ethylene in a molar ratio of 1:1 to 1:20 in copolymerised form and which is saponified to a degree of hydrolysis of 50 to 100 percent.

A further object of the invention is a process for the production of high bulk non-woven fibre fleece from natural and/or synthetic fibres and binder fibre of a thermoplastic polymer, which comprises forming high bulk non-woven fibre fleece containing 5 to 50 percent of binder fibres consisting of a saponified ethylene-vinyl acetate copolymer which prior to saponification contained vinyl acetate and ethylene in a molar ratio of 1:1 to 1:20 incorporated in polymerised form and which has been saponified to a degree of hydrolysis of 50 to 100 percent is heated and heating the formed fleece to a temperature of above 110° C to render adhesive the binder fibre.

Preferably fibres from ethylene-vinyl acetate copolymer were used, having a molar ratio of vinylacetate and ethylene in the range of 1:2 to 1:10 and being saponified to a degree of saponification is 80 to 100 percent. Fibres spun from these substances afford the advantage in a process for the production of fibre fleeces that because of their comparatively low melting points of 100° to 130° C, temperatures starting at 110° to 130° C are quite sufficient for the process of bonding by heat, although temperatures in the region of 130° to 150° C would preferably be employed in order to obtain sufficiently high production rates. No damage to the fibre material of which the finished fleece is formed can be observed at these temperatures whereas other bonding fibres, which have melting points above 130° C in most cases require processing temperature of above 150° C, at which wool, for example, is severely damaged.

Furthermore, fleeces which have been bonded with the above described fibres used according to the invention are resistant to chemical cleaning with perchlorethylene and petroleum hydrocarbons. Polymers which have been produced from polyolefines and polyvinyl chloride and their copolymers with ethylene and/or vinyl acetate are liable to be harmed by these solvents, especially the first mentioned solvent. The binder swells or dissolves and the fibre bond is destroyed.

The material used according to the invention also has excellent resistance to water. Fleeces bonded with this material can be washed substantially unharmed in washing machines at 60° C whereas polymers based on copolyamides undergo severe swelling under these conditions, with the result that the bond is severely damaged and the connection between the fibres dissolves at least partly if the fleeces is in addition subjected to mechanical stresses due to rotation in the drum of the washing machine.

The fibres used are preferably staple fibres cut to a length of 40 to 100 mm if the fleece is produced by mechanical processes. Staple fibres cut to a length of 5 to 20 mm are preferred in hydrodynamic processes, for example. The denier of the fibres may vary within wide limits but is advantageously between 3.3 and 22 dtex. In the fibre material preferably used according to the invention, the basic substance consists of a saponified ethylene vinyl acetate copolymer which contains vinyl acetate and ethylene in a molar ratio of between 1:3 and 1:10 before saponification and which has a degree of hydrolysis of 90 to 100 percent. The melting point of this material is in the range of 105° to 115° C so that working up temperatures of 130° C or more may be employed. At these temperatures, no damage to the fibre material which is to be bonded is liable to occur but on the other hand an intimate bond, which has the advantages already described above, is obtained due to the complete melting of the thermoplast.

Preparation of ethylene vinyl acetate copolymers which may be used as starting materials for the bonded fibres claimed here may be carried out by any desired methods at pressures of 200 to 4,000 excess atmospheres and at temperatures of 30° to 250° C. The average molecular weight of the polymers may vary within wide limits but polymers with average molecular weights of 10,000 to 50,000 are preferred because it has been found that in this range the technological properties are particularly advantageous for the working up processes. In the case of very high molecular weight products the softening rate is too low whereas low molecular weight products have less advantageous mechanical properties, e.g., lower bond strength, lower mechanical strength and higher brittleness. Saponification of the ethylene vinyl acetate copolymers may also be carried out by any known methods.

Preferably, however, saponification is carried out by an ester interchange with aliphatic alcohols in an anhydrous medium in the presence of alkaline or acid catalysts. The alkaline ester interchange catalysts used are advantageously alkali metal alcoholates, e.g., sodium methylate or potassium ethylate, and the acid catalysts used are advantageously hydrogen halides such as hydrogen chloride, sulphuric acid or organic sulphonic acids such as toluene sulphonic acid. The aliphatic alcohols used are preferably methyl alcohol or ethyl alcohol in order to achieve rapid and thorough alcoholysis of the ethylene vinyl acetate copolymer. In the case of methyl alcohol the ester interchange equilibrium can be shifted in the direction of the saponified polymer by removing the methyl acetate by distillation as the most volatile constituent. The suitability of the saponified ethylene vinyl acetate copolymers as bonding fibres is largely determined by their composition. It has been found that a certain vinyl alcohol content is necessary for good resistance to solvents whereas an increase in the vinyl acetate content with unchanging vinyl alcohol content promotes the solubility of the polymer in those organic solvents which are commonly used in dry cleaning, e.g., trichloroethylene, cleaning petrol. If the vinyl alcohol content in the polymer is too high, on the other hand, it renders the polymer too hydrophilic and, therefore, impairs its resistance to aqueous washing liquors. Moreover, saponified ethylene vinyl acetate copolymers which have a high vinyl alcohol content have high softening and melting points which may have an adverse effect on the rate or temperature of bonding. The most suitable saponified ethylene vinyl acetate copolymers for use as bonding fibres are those which prior to saponification contain vinyl acetate and ethylene in a molar ratio of 1:1 to 1:20 preferably 1:3 to 1:10, incorporated in polymerised form, and which have been saponified to a degree of hydrolysis of 50 to 100 percent, preferably 90 to 100 percent. Ethylene vinyl acetate copolymers of this type contain 7 to 60 per cent by weight of vinyl alcohol when the degree of hydrolysis is 100 percent.

Preparation of the bonding fibres may be carried out by known methods, e.g., by melt spinning, dry spinning or precipitation spinning processes. Staple fibres cut to a length of 5 to 100 mm are produced from the resulting threads by known methods.

The fleeces which are to be bonded may consist of various natural or synthetic fibres or mixtures, e.g., cotton regenerated cellulose, wool or fibres of acetyl cellulose, polyacrylonitrile, polyester amides, polyamides and/or polyurethanes.

The following Examples illustrate more particularly the invention:

Example 1

A mixture of polyethylene terephthalate staple fibres (3.3 dtex. 60 mm staple length) and fibres of saponified ethylene/vinyl acetate copolymer (3.3 dtex. 60 mm staple length) which contained ethylene and vinyl acetate in a molar ratio of 5.8:1 incorporated in polymerised form before saponification and in which 98 percent of the acetyl groups are saponified, in a ratio of 4:1, is carded on a carding machine and then leased to produce a fleece which has a thickness of about 40 mm and a weight of 400 g/m². This fleece is passed at the rate of 2 m/min through a 5 m long oven in which the ambient air has been heated to 135° C. A high bulk, springy elastic fleece is obtained which may be used as filling fleece in anoraks, eiderdowns. The fleece is characterised by its high resistance in dry cleaning and in machine washing at 60° C. It has little tendency to splitting.

Example 2

A mixture of polyethylene terephthalate staple fibres (6.7 dtex, staple length 40 mm) and fibres of saponified ethylene/vinyl acetate copolymer (6.7 dtex, staple length 60 mm) which contained ethylene and vinyl acetate in a molar ratio of 1.45:1 in polymerised form before saponification and in which 99.5 percent of the acetyl groups have been saponified is made up into a fleece in the proportion of 3:1 as described in Example 1 and bonded by heating in an oven at 160° C.

The fleece obtained has a very high bulk and springy elasticity and is resistant to dry cleaning in perchloroethylene and mild washing.

Example 3

A mixture of clipped wool (about 5 dtex, length 80 mm) and fibres of saponified ethylene/vinyl acetate copolymer (6.7 dtex, staple length 60 mm) which con-

tained ethylene and vinyl acetate in a molar ratio of 5.8:1 incorporated in polymerised form before saponification and in which 98 percent of the acetyl groups have been saponified is made up into a fleece in the ratio of 7:3 by carding on a carding machine followed by leasing. The fleece has a weight per m^2 of 280 g and is drawn off at the rate of 4 m/min. It is carried through a 5m long oven in which the ambient air temperature has been adjusted to 145° C and it is then rolled up. The fleece obtained has a high springy elasticity and a bulky, soft handle and may be used as filling fleece for eiderdowns or pillows.

Comparison Example A

Polyethylene terephthalate staple fibres (3.3 dtex, staple length 60 mm) are made up into a fleece having a weight per m^2 of about 400 g by processing on a carding machine and then leasing, and it is drawn off at the rate of 3 m/min. The surface of the fleece is then sprayed with a dispersion binder which consists of a copolymer of methyl acrylate with butyl acrylate and cross-linking agent which contains reactive groups and which has a solids content of 50 percent. After its first passage through a drying apparatus it is turned over and the rear surface is sprayed in the same way with the polymer binder described above. The fleece is again passed through the oven and finally rolled up. The fleece has a soft handle but the surface has a hard handle resembling that of a plastics material. The fleece can easily be split in the centre. It can be used for the manufacture of eiderdowns and for filling anoraks and other articles.

Comparison Example B

Polyethylene terephthalate staple fibres (3.3 dtex, staple length 60 mm) and fibres made of a copolymer of vinyl acetate in the ratio of 85:15 (3.3 dtex, staple length 55 mm) are mixed in the ratio of 4:1 and made up into a fleece weighing 400 g/m^2 on a carding machine followed by a leasing machine. The fleece is then passed through a 5 m long oven in which the ambient air has been heated to 160° C. After cooling, a spring elastic, high bulk fleece material is obtained which may be used as filling fleece for the articles described in Example 1. After dry cleaning however, the fibre bond has dissolved and the original high bulk character is, therefore, lost. Articles filled with this fleece has limited durability as regards wear.

Comparison Example C

A mixture of clipped wool (about 5 dtex, length 80 mm) and fibres consisting of a copolyamide (polyamide 6 and polyamide 6,6 in the ratio of 2:1) (6.7 dtex, staple length 50 mm) was made up into a fleece as described in comparison Example B and bonded by heating in an oven at 180° C. It was found that as a result of heating, the fleece had shrunk by about 20 percent in height and width and undergone noticeable yellowing. In contrast to the fleece produced in Example 2, a high bulk, soft and springy elastic handle could not be recorded.

What we claim is:

1. High bulk, non-woven fiber fleece comprising 5 to 50 percent by weight, based on the weight of fibers in the fiber fleece, of: (1) a material selected from the group consisting of natural and synthetic fibers, and (2) binder fibers, differing from said natural and synthetic fibers, said binder fibers consisting essentially of a saponified ehtylene-vinyl acetate copolymer containing vinyl acetate and ethylene in a molar ratio of 1:1 to 1:20 in copolymerized form and which is saponified to a degree of hydrolysis of 50 to 100 percent.

2. High bulk, non-woven fiber fleece comprising 5 to 50 percent by weight, based on the weight of fibers in the fiber fleece, of: (1) a material selected from the group consisting of cotton, regenerated cellulose, wool, acetyl cellulose, polyacrylonitrile, polyester amide, polyamide and polyurethane fibers; and (2) binder fibers, said binder fibers consisting essentially of a saponified ethylene-vinyl acetate copolymer containing vinyl acetate and ethylene in a molar ratio of 1:1 to 1:20 in copolymerized form and which is saponified to a degree of hydrolysis of 50 to 100 percent.

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UNITED STATES PATENT OFFICE

CERTIFICATE OF CORRECTION

Patent No. 3,801,428 (SN 183,687) Dated April 2, 1974

Inventor(s) HELLMUT STRIEGLER, RAOUL RESZ, HERBERT BARTL

It is certified that error appears in the above-identified patent and that said Letters Patent are hereby corrected as shown below:

Claim 1 should read as follows:

--1. High bulk, non-woven fiber fleece comprising (1) a material selected from the group consisting of natural and synthetic fibers; and (2) 5 to 50 per cent by weight, based on the weight of fibers in the fiber fleece, of binder fibers, differing from said natural and synthetic fibers, said binder fibers consisting essentially of a saponified ethylene-vinyl acetate copolymer containing vinyl acetate and ethylene in a molar ratio of 1:1 to 1:20 in copolymerized form and which is saponified to a degree of hydrolysis of 50 to 100 percent.--

Claim 2 should read as follows:

--2. High bulk, bon-woven fiber fleect comprising (1) a material selected from the group consisting of cotton, regenerated cellulose, wool, acetyl cellulose, polyacrylonitrile, , polyester amide, polyamide and polyurethane fibers; and (2) 5 to 50 percent by weight, based on the weight of fibers in the fiber fleece, of binder fibers, said binder fibers consisting essentially of a saponified ethylene-vinyl acetate copolymer containing vinyl acetate and ethylene in a molar ratio of 1:1 to 1:20 in copolymerized form and which is saponified to a degree of hydrolysis of 50 to 100 percent.--

Signed and sealed this 10th day of September 1974.

(SEAL)
Attest:

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