



Europäisches Patentamt
European Patent Office
Office européen des brevets



(11) Publication number:

0 396 765 B1

(12)

EUROPEAN PATENT SPECIFICATION

- (49) Date of publication of patent specification: **06.07.94** (51) Int. Cl.⁵: **D06M 11/00, D06M 15/00, D06P 5/02**
- (21) Application number: **89910939.1**
- (22) Date of filing: **02.10.89**
- (86) International application number:
PCT/JP89/01006
- (87) International publication number:
WO 90/04671 (03.05.90 90/10)

(54) **COATED POLYESTER FIBER FABRIC AND PROCESS FOR ITS PRODUCTION.**

- (30) Priority: **19.10.88 JP 263412/88**
16.03.89 JP 66851/89
- (43) Date of publication of application:
14.11.90 Bulletin 90/46
- (45) Publication of the grant of the patent:
06.07.94 Bulletin 94/27
- (84) Designated Contracting States:
AT BE CH DE FR GB IT LI LU NL SE
- (56) References cited:
DE-A- 1 469 241 GB-A- 788 151
GB-A- 1 115 376 JP-A- 609 983
JP-A- 6 039 479 JP-A- 6 045 680
JP-A- 6 059 345
- (73) Proprietor: **TORAY INDUSTRIES, INC.**
2-1, Nihonbashi Muromachi 2-chome
Chuo-ku
Tokyo 103(JP)
- (72) Inventor: **TOHYAMA, Syunroku**
1173-36, Tobakou

Yasu-cho
Yasu-gun
Shiga 520-23(JP)
Inventor: **IKEYAMA, Masami**
2030-34, Kamihiraki-cho
Youkaichi-shi
Shiga 527(JP)
Inventor: **NAKABE, Ikuko**
5-10, Nakahozumi 1-chome
Ibaraki-shi
Osaka 567(JP)

- (74) Representative: **Brehm, Hans-Peter, Dr.,**
Dipl.-Chem. et al
Patentanwälte
Kern, Brehm & Partner
Albert-Rosshaupter-Strasse 73
D-81369 München (DE)

Note: Within nine months from the publication of the mention of the grant of the European patent, any person may give notice to the European Patent Office of opposition to the European patent granted. Notice of opposition shall be filed in a written reasoned statement. It shall not be deemed to have been filed until the opposition fee has been paid (Art. 99(1) European patent convention).

EP 0 396 765 B1

Description

The present invention relates to a coated fabric comprising polyester fibers dyed with dye stuff molecules, and at least one resinous coating layer. The coated fabric does not provide staining caused by a migration of the dye stuff molecules. Further, the present invention concerns a method of preparation said coated fabric.

At present time used coated fabrics concern woven and knitted fabrics comprising nylon fibers as a main component and, for example, treated with such treatments to provide repellent and waterproof property, water-vapor permeable and repellent property, breathable property, flameproof and meltproof property and comprising respective coatings.

However, as recently the differences in prices between nylon fibers and polyester fibers have remarkably increased, developments on coating treatments of polyester fibers have been actively carried out. Polyester fibers provide superior properties such as dimensional stability, strength, light resistance and diversity of the raw material with respect to nylon fibers.

However, a coated fabric comprising polyester fibers shows a fatal defect that the dye in/on the polyester fibers migrates to the coating and stains this coated film and probably stains another film being in contact with the coating on the polyester fibers. Namely, in case of dyeing the polyester fibers with a disperse dye, the polyester fibers do not combine chemically with the dye stuff, as, for example, in contrast to the dyeing of nylon with an acid dye stuff. In addition, the typical disperse dye stuffs provide good solubility in and affinity with organic solvents and synthetic resins, so that the dye stuff molecules in/on the polyester fibers could easily migrate to the coated film layer.

Therefore, contacting coated faces of different colors with each other will provide staining. Up to the present time various investigations and research work to solve this problem have been done. But, any perfect solution could not be obtained. Therefore, dyed products of coated fabrics comprising polyester fibers have not gained widely accepted practical use until nowadays.

The document DE-A1-1.469.241 discloses a method for treatment of woven or knitted fabrics comprising synthetic fibers. The fibers may include polyester fibers. The treatment concerns the deposition of metal oxide particles having colloidal particle sizes on the fabric followed by a heating treatment up to the softening point of the fiber material in order to fix the particles. The metal oxide particles include highly dispersed colloidal aluminum oxide or colloidal silica. It is said that said deposition of metal oxide particles reduces essentially the migration of dispersion dye stuffs on the smooth synthetic fibers. This reference does not mention a fabric comprising polyester fibers which has been coated with a resinous coating layer. Therefore, this document does not deal with the technical problem of staining of resinous coating layers deposited on a polyester fiber fabric.

The document JP-A-45.686/1985 discloses a method wherein such fine metal powders made of aluminum, copper and silver and metal oxides such as potassium titanate, titanium dioxide and tin(IV)oxide comprising bad miscibility with disperse dye stuffs were used to prevent a sublimation of the dye stuff molecules. However, with this known method, the migrating dye stuff molecules cannot be caught completely, and staining caused by migration of the dye stuff molecules cannot be prevented perfectly.

The documents JP-A-4.873/1983 and JP-A-53.632/1987 disclose methods wherein fiber structures are treated to obtain a water repellance property. The water repellent agent comprises perfluoroalkyl groups. The fabric is coated with a polyurethane resin coating layer containing porous particles made of SiO_2 as the main component. The purpose is to obtain a waterproof fabric presenting both breathable and water-vapor permeable properties by providing fine pores within porous fine inorganic particles incorporated in a polyurethane resin to provide paths for air and water-vapor diffusion and transpiration. In this document there is not any hint or suggestion that a coating film of said type may prevent staining caused by migrating disperse dye stuff molecules.

The technical problem of the present invention is directed to substantially reduce or to prevent staining caused by migrating dye stuff molecules within a coated fabric comprising polyester fibers dyed with dye stuff molecules, and further comprising at least one resinous coating layer.

In general, the present invention proposes a technical teaching wherein the dye stuff molecules of a disperse dye stuff typically used for dyeing polyester fibers are caught within fine pores having a specific pore diameter and provided by fine porous inorganic particles having defined properties. Said fine pores having a pore diameter of 15 nm or less. Said particles are adhered to the polyester fibers' surface and/or are incorporated into at least one coating layer. Said fine porous inorganic particles adhered to the surface of the polyester fibers comprise an average particle size in the range of 1 to 100 nm (nanometers). Said fine porous inorganic particles being dispersed within at least one coating layer comprise an average particle size of 15 μm (micrometers) or less. In addition, said fine porous inorganic particles comprise a

surface area of 200 m²/g or more. Further, said particles being present in an effective amount to catch dye stuff molecules migrating from said polyester fibers into a coating layer, in order to substantially reduce or to prevent staining.

In accordance with a first aspect of the present invention, there is provided a coated fabric comprising polyester fibers dyed with dye stuff molecules, and further comprising at least one resinous coating layer, characterized in that

- a) additionally providing porous fine inorganic particles being adhered to the surface of the polyester fibers;
- b) said particles comprise an average particle size in the range of 1 to 100 nm;
- 10 c) said particles comprise fine pores having a pore diameter of 15 nm or less; and
- d) said particles comprise a surface area of 200 m²/g or more.

In accordance with a second aspect of the present invention there is provided a coated fabric comprising polyester fibers dyed with dye stuff molecules, and at least one resinous coating layer, characterized in that

- 15 a) additionally providing fine porous inorganic particles being dispersed within at least one coating layer;
- b) said particles comprise an average particle size of 15 μm or less;
- c) said particles comprise fine pores having a pore diameter of 15 nm or less; and
- d) said particles comprise a surface area of 200 m²/g or more.

In accordance with a third aspect of the present invention, there is provided a method of preparation a coated fabric comprising polyester fibers dyed with dye stuff molecules, further comprising at least one resinous coating layer; said method is characterized by the steps:

- a) providing a dyed fabric consisting essentially of polyester fibers;
- b) adhering to at least one main surface of said fabric fine porous inorganic particles; and
- c) depositing at least one resinous coating layer to said treated surface of the fabric; wherein
- 25 d) said particles comprise an average particle size in the range of 1 to 100 nm;
- e) said particles comprise fine pores having a pore diameter of 15 nm or less; and
- f) said particles comprise a surface area of 200 m²/g or more.

In accordance with a fourth aspect of the present invention, there is provided a method of preparation a coated fabric comprising polyester fibers dyed with dye stuff molecules and comprising at least one resinous coating layer; said method is characterized by the steps:

- a) providing a dyed fabric consisting essentially of polyester fibers;
- b) providing a resinous solution containing a homogeneous dispersion of fine porous inorganic particles; and
- c) coating at least one main surface of said fabric with said resinous solution; wherein
- 35 d) said particles comprise an average particle size of 15 μm or less;
- e) said particles comprise fine pores having a pore diameter of 15 nm or less; and
- f) said particles comprise a surface area of 200 m²/g or more.

According to the present invention, disperse dye stuff molecules being used for dyeing a coated fabric comprising polyester fibers and at least one resinous layer are caught by fine porous inorganic particles having defined properties and comprising fine pores of a specified pore diameter in order to prevent or substantially reduce migration of said dye stuff molecules which will cause staining of a fabric surface, of a coating layer of said fabric or of another coating layer of another adjacent coated fabric. In so far, the invention provides a solution to the underlying technical problem.

Within the scope and explanation of the present invention, the term "polyester fiber" or "polyester fiber structure" or "polyester fabric" means not only woven and knitted fabrics and non-woven fabrics consisting to 100 % of polyester fiber but means also mixed spun, combined filament, different yarn-twisted union-woven and union-knitted fabrics wherein the main or essential components are made of polyester fibers without a specific limitation to specific weight % ranges. However, the effects provided with the present invention are more remarkable when the fabric is consisting of polyester fibers by 100 % or wherein the fabric comprises a blend of different fiber materials comprising a high rate of polyester fibers. Further, the effects provided with the present invention are more remarkable when the type of dye stuffs used for dyeing the coated fabric belongs to the group of disperse dye stuffs.

According to the present invention, the fine porous inorganic particles may consist of a material selected from the following group comprising silicon dioxide, titanium oxide, zirconium oxide, aluminum oxide, active carbon and similar inorganic materials. Among them, silicon dioxide is most effective with respect to effect and utility, and preferred fine porous inorganic particles are made of silicon dioxide.

The migrating dye stuff molecules are caught within the pores provided in the fine porous inorganic particles. Thereto, the dimension or diameter of the fine pores in the porous inorganic particles largely

influences the capability of adsorption the migrating dye stuff molecules. Therefore, fine pores comprising a pore diameter of 15 nm (15 nanometers or respectively 150 Å) or smaller provide good results and can be used successfully. Pore diameters ranging of from 1 to 10 nm (10 to 100 Å) provide even better results with respect to the effectiveness of catching migrating dye stuff molecules, and fine porous inorganic particles having pores comprising a pore diameter in the range of 1 to 10 nm are preferred according to the present invention. With pores having a pore diameter larger than 15 nm (150 Å) the adsorption and retention of the migrating dye stuff molecules is not sufficient.

In addition, the surface area of the fine porous inorganic particles has an effect to the retention of migrating dye stuff molecules. Thereto, the fine porous inorganic particles shall comprise a surface area of 200 m²/g or even a larger surface area. According to an even more preferred embodiment of the present invention, the fine porous inorganic particles shall comprise a surface area of 500 m²/g. If the fine porous inorganic particles comprise a surface area smaller than 200 m²/g, then the technical problem underlying the present invention cannot be solved in a sufficient and effective manner.

According to an essential aspect of the present invention, fine porous inorganic particles are provided within a dyed and coated fabric made essentially of polyester fibers in order to substantially reduce or to prevent the migration of the dye stuff molecules which might cause an unpleasant staining. The particle size of said fine porous inorganic particles influences the effectiveness of the present invention. A proper selection of the particle diameter is depending of the manner of incorporating the fine porous inorganic particles into the coated fabric. There are essentially two main and different embodiments of incorporating the fine porous inorganic particles into the coated fabric. According to the first embodiment, the fine porous inorganic particles are adhered on the surface of the polyester fibers before applying a coating. A preferred method of adhering the fine porous inorganic particles directly to the fibers' surface is a padding treatment. According to the second embodiment, the fine porous inorganic particles are incorporated in a film of a coating resin. A preferred method of incorporating the fine porous inorganic particles into the coating comprises the steps of providing a resinous solution containing a homogeneous dispersion of fine porous inorganic particles as defined and coating at least one main surface of said fabric with said resinous solution.

According to the first embodiment wherein the fine porous inorganic particles are adhered directly on the surface of the fibers, the particles may comprise an average particle diameter ranging of from 1 to 100 nm (1 to 100 nanometers which equals 1 to 100 millimicron (mμ)). Preferably, the particles shall comprise an average particle diameter ranging of from 10 to 50 nm (10 to 50 nanometers which equals 10 to 50 millimicron (mμ)). In this case of directly adhering or absorbing the fine porous inorganic particles to the surface of the fibers, the particle diameter has to be selected much smaller than the diameter of the fibers, which is typically in the range of several micrometers or more.

Of course, in this first embodiment of directly adhering the fine porous inorganic particles to the fibers' surface and wherein the particles comprise an average particle diameter ranging of from 1 to 100 nm, preferably of from 10 to 50 nm, the fine pores provided with said particles have a pore diameter of 15 nm or less and smaller than the particle size.

According to this first embodiment wherein the fine porous inorganic particles are adhered directly on the surface of the fibers, it is essential to provide a uniform distribution of the adhered particles on the surface of the fibers in order to obtain a complete effect. According to a preferred method with respect to handling and workability, an aqueous dispersion of the fine porous inorganic particles is provided, and the fine porous inorganic particles are adhered to the surface of the fibers by means of a padding treatment. The padding treatment of the dyed fabric made essentially of polyester fibers is followed by a drying step. The drying temperature shall be maintained in a range of from 80 °C to 160 °C, preferably in a range of from 100 °C to 130 °C; a drying temperature above 160 °C decreases the effect as aimed with the present invention.

According to this first embodiment wherein the fine porous inorganic particles are adhered directly on the surface of the fibers, the amount of adhered fine porous inorganic particles may range of from 0.5 to 15 % by weight based on the weight of the fabric. Even more preferred is an amount of adhered fine porous inorganic particles ranging of from 1.5 to 10 % by weight based on the weight of the fabric.

In order to improve the durability of adherence of fine porous inorganic particles to the surface of the fibers a typical resin as used for finishing treatment may be applied in a parallel manner. This kind of treatment is preferred in order to obtain a good adherence of the fine porous inorganic particles to the polyester fibers.

According to the second embodiment wherein the fine porous inorganic particles are incorporated in a film of coating resin, the particle size of said particles may range of from 15 μm (micrometers) at a maximum to less than 15 μm. The smaller particle sizes are preferred. In a preferred method of obtaining a

product according to this second embodiment, the fine porous inorganic particles are homogeneously dispersed within a resinous coating solution, and this solution is applied on the prepared and dyed fabric. In this case, the particles flocculate, and larger particle sizes can be used than in the first embodiment. In this second method a good dispersion of the fine porous inorganic particles within the resin is important. An agglomeration of the particles causes a decrease in the effect of preventing staining and deteriorates the quality of the coating.

According to this second embodiment wherein the fine porous inorganic particles are homogeneously dispersed in a film of coating resin, the amount of this particles may range of from 10 % by weight to more than 10 % by weight, based on the solid resin content of the resinous coating layer.

According to a specific mode of carrying-out said second embodiment, a coating may be applied which comprises several coating layers 1, 2 and/or 3 as depicted in the drawing. These different coating layers 1, 2 and/or 3 may be prepared from different resinous coating solutions comprising each a different content of fine porous inorganic particles. For example, at least one coating layer may comprise a significant to high content of fine porous inorganic particles, and at least one further coating layer may comprise a small to negligible content of fine porous inorganic particles.

According to this specific mode of carrying-out, a coating structure may be obtained wherein the fine porous inorganic particles are lamellarly distributed within the resin film. For example, as illustrated in the drawing, a lamellar structure may comprise one resin layer selected from the group comprising resin layers 1, 2 and 3, wherein the fine porous inorganic particles are lamellarly dispersed at a high concentration and at least a further resin layer selected from the group comprising layers 1, 2 or 3, wherein the content of fine porous inorganic particles is small or completely missing (negligible).

Practically speaking, a resinous coating solution A containing a high concentration of fine porous inorganic particles in a magnitude of 10 % by weight or more based on the weight of the solid resin content of the resinous coating solution is prepared; further, a second resinous coating solution B is prepared containing no or less than 10 % fine porous inorganic particles. With respect to the order of applying or depositing, the first layer 1 (according to the drawing) may be prepared of the resinous coating solution B and the second layer 2 may be prepared of the resinous coating solution A. Further, a method may be used wherein the procedure is done in a reverse order. Further, a triple layered coating structure may be prepared wherein at least one of the first, second or third layer according to the drawing is prepared of the resinous coating solution A and the other two layers are prepared of the resinous coating solution B. In the case of a double or triple structure wherein the first layer 1 is prepared of a resinous coating A, the adhesiveness of this coating layer 1 with the fiber structure 4 may be reduced in some cases. If a high or significant adhesiveness of the first coating layer 1 with the fiber structure 4 is especially required, it is preferred to prepare the first coating layer 1 of the resinous coating solution B and to prepare the second layer 2 or the third layer 3 of the resinous coating solution A. According to a further preferred aspect, the thickness of a coating layer 1, 2 and/or 3 comprising a high concentration of fine porous inorganic particles shall be selected 3 μm (micrometers) or thicker. No limitation exists with respect to a specific coating method used to apply the several coating layers 1, 2 and/or 3. Especially, in an embodiment wherein the fine porous inorganic particles are lamellarly incorporated and are contained in a high concentration within a resin layer of the coating structure, the high content of fine porous inorganic particles will catch completely any dye stuff molecules migrating from the dyed fiber structure into a resin film. The dye stuff molecules are adsorbed and kept within the fine pores which are provided within the fine porous inorganic particles. Thereto, the present invention provides an effect of permanently preventing the dye stuff molecules from migration.

According to the present invention, a wide range of known and typical coating resins and resinous coating solutions can be used for coating a dyed polyester fabric. For example, the coating resin can be freely selected from a group comprising urethane, acrylic, silicone, vinyl chloride and vinyl acetate coating resins. Further, the usual and well-known coating treatments can be used to apply the coating solution. Further, several different coating layers may be applied prepared from different resinous coating solutions, wherein some resinous coating solutions do contain fine porous inorganic particles, and other resinous coating solutions do not.

In the following the present invention will be explained in more detail by reference to Examples and Comparative Examples. The (inventive) Examples serve for explanation purposes and may not be construed to restrict the scope of the invention. Further, reference is made to a drawing wherein Figure 1 is a cross-sectional representation illustrating an example of a coated fabric made of polyester fibers and obtainable according to Example 4 of the present invention. There

"1" denotes a first resinous coating layer;

"2" denotes a second resinous coating layer;

"3" denotes a third resinous coating layer; and

"4" denotes a fiber structure.

General Remarks:

5

(1) An evaluation of the fastness to dye stuff migration and to the staining in the test pieces obtained according to the following Examples and Comparative Examples was performed according to the following method:

10 A test piece (5 cm x 5 cm) obtained according to an Example or Comparative Example and attached white polyester fabrics (the same raw fabric as used to obtain the test piece and coated with the same resin as used for the test piece; 5 cm x 5 cm) were inserted between two glass plates in such a way that the coated faces of the attached white polyester fabrics were brought into contact with both the coated face and the non-coated face of the test piece. A mechanical force in an amount of 200 g was applied to compress the two glass plates together. The compressed sandwich-like arrangement was placed in an oven hold at a constant temperature of $100^{\circ}\text{C} \pm 2^{\circ}\text{C}$ for 48 hours. After a cooling step the state of dye stuff migration from the test piece to the attached adjacent white fabric was evaluated in terms of a classification by means of a grey scale for evaluating staining. A rating was performed between values ranging from 1 to 5, wherein 1 denotes the lowest and 5 denotes the best fastness against dye stuff migration and staining.

20 The results obtained in Examples and Comparative Examples are summarized in Table 1.

(2) The following resins were used as coating resins in Examples and Comparative Examples:

Polyether polyurethane resin

("Crysbon 8006HV" manufactured by Sanyo Chemical Co., Ltd.);

Acrylic resin

25 ("Cryscat P-1,120" manufactured by Dainippon Ink Chemical Co.Ltd.);

Silicone resin

(Toray silicone "SD 8,001" manufactured by Toray Silicone Co., Ltd.).

Example 1:

30

A plain woven fabric was prepared from polyester filaments each of 50 denier warp yarn and 75 denier weft yarn. The obtained polyester fabric was dyed with a disperse dye "Resoline Blue FBL" of 3 % o.w.f. at 130°C for 60 min. Following the dyeing treatment, the polyester fabric was washed as usual. Following the washing treatment and a usual drying step, a heat-setting treatment at 180°C was performed. Thereafter, a dyed polyester fabric ready for coating was obtained.

35 Thereafter, a padding treatment of this fabric was performed with an aqueous solution comprising 30 g/l trimethylolmelamine and silicon dioxide particles having a particle diameter of 20 nm (20 nanometers, which equals 20 millimicron (μm)), a fine pore diameter of 6 nm (6 nanometers, which equals $60 \text{ \AA}$) and a surface area of $300 \text{ m}^2/\text{g}$. The content of the dispersed silicon dioxide particles within the aqueous padding solution is 15 % by weight of the solid content of the resin. Following the padding treatment, the fabric was dried at 130°C for 1 min. The build-up, that is the weight gain of the polyester fabric by the silicon dioxide particles was 2.4 % by weight based on the weight of the polyester fabric. Thereafter, the fabric was coated with a polyether polyurethane resin solution in dimethylformamide by means of a knife coater. The coating solution was coagulated by means of a wet process to obtain a coated fabric comprising a coating weight of 25 g/m².

45

Comparative Example 1:

50 Essentially, the procedure of Example 1 was repeated, however the padding treatment of the dyed polyester fabric to deposit silicon dioxide particles was omitted. This means, the dyed fabric obtained according to Example 1 was only wet-coated with a polyether polyurethane resin solution in dimethylformamide without prior deposition of silicon dioxide particles.

Example 2:

55

A dyed polyester fabric ready for coating was obtained by the same method as stated in Example 1. Particulate silicon dioxide with a particle diameter of $3 \mu\text{m}$ (3 micrometers), a fine pore diameter of 5 nm (5 nanometers, which equals $50 \text{ \AA}$) and a surface area of $500 \text{ m}^2/\text{g}$ was dispersed in a polyether polyurethane

resin solution in dimethylformamide. The content of the dispersed silicon dioxide particles within the resinous coating solution is 15 % by weight based on the weight of solid resin. The dyed polyester fabric was coated with this silicon dioxide particles containing coating solution by means of a knife coater to obtain a coated fabric.

5

Comparative Example 2:

Essentially, the method of Example 2 was repeated. Deviating, another type of silicon dioxide particles was used comprising a particle diameter of 20 μm , a fine pore diameter of 21 nm (210 Å) and a surface area of 150 m²/g.

10

Example 3:

Essentially, the method of Example 2 was repeated. Deviating, another type of resinous coating solution was used comprising an acrylic and a silicone resin as the coating resin. The obtained coating layer containing said fine porous inorganic particles had a thickness of 10 μm .

15

Comparative Example 3:

Essentially, the method of Example 3 was repeated. Deviating, each an acrylic resin and a silicone resin were separately applied in order to provide a coating which does not contain any silicon dioxide particles.

20

Example 4:

A dyed polyester fabric ready for coating was obtained by the same method as stated in Example 1.

25

At first, this fabric was coated with a polyether polyurethane resin solution in dimethylformamide by means of a knife coater. The resinous coating solution was coagulated by means of a wet process to obtain a film.

Next, a second coating was applied comprising fine porous inorganic particles. Thereto, particulate silicon dioxide comprising a particle diameter of 3 μm , a fine pore diameter of 5 nm (50 Å) and surface area of 500 m²/g was dispersed in the same resinous coating solution. The amount of the dispersed silicon dioxide particles is 30 % by weight based on the weight of the solid resin. The already coated fabric was further coated with this silicon dioxide particles containing coating solution by means of a knife coater. This second coating solution was coagulated by means of a wet process to obtain a top coat of a two-layered coating film.

30

35

In a further embodiment of this Example an additional top coating was applied to the already prepared two-layered coating. This third coating layer was made of the same resinous coating solution containing no fine porous inorganic particles. This third coating solution was applied by means of a knife coater and was coagulated by means of a wet process to obtain a coated fabric comprising a three-layered coating structure, wherein the layer comprising inorganic particles forms an intermediate layer. The thickness of this intermediate layer containing fine porous inorganic particles is 10 μm .

40

Comparative Example 4:

A dyed polyester fabric ready for coating was obtained by the same method as described in Example 1. Particulate silicon dioxide comprising a particle diameter of 3 μm , a pore volume of 0.5 cm³/g, a fine pore diameter of 17 nm (170 Å) and a surface area of 300 m²/g was dispersed in a polyester polyurethane resin solution in dimethylformamide. The content of the dispersed silicon dioxide within the resinous coating solution is 15 % by weight based on the weight of the solid resin. The dyed polyester fabric was coated with this silicon dioxide particles containing coating solution by means of a knife coater to obtain a coated fabric.

45

50

55

Table 1

	Conditions		Rating of Fastness against dye stuff migration and staining
	Silicon dioxide ⁺	Resin	
Example 1	20 nm, 6 nm 300 m ² /g	Urethane	4 - 5
Comparative Example 1	--	Urethane	2
Example 2	3 μm, 5 nm 500 m ² /g	Urethane	4 - 5
Comparative Example 2	20 μm, 21 nm 150 m ² /g	Urethane	2
Example 3	3 μm, 5 nm 500 m ² /g	Acrylic	4 - 5
	3 μm, 5 nm 500 m ² /g	Silicone	4 - 5
Comparative Example 3	--	Acrylic	1
	--	Silicone	1
Example 4	3 μm, 5 nm 500 m ² /g	Urethane	5
Comparative Example 4	3 μm, 17 nm 300 m ² /g	Urethane	2 - 3
		Silicone	1

⁺ Notice: The column of "silicon dioxide" lists the average particle diameter, then the average fine pore diameter and then the surface area of the used silicon dioxide particles in this order.

The coated fabric according to the present invention can be widely used for preparing clothings and for industrial application purposes. Especially, the method according to the present invention is well suited to be performed along with other known procedures to provide various products with repellent and waterproof property, with a water-vapor permeable and repellent property, with breathable, flameproof and meltproof properties, and in order to obtain respective coatings.

Especially, the coated fabric according to the present invention is well suited to overcome some defects of the coated nylon fabrics such as price, dimensional stability, light resistance and versatility of raw materials. The coated polyester fabric according to the present invention may at least substitute a part of the demand for coated nylon fabrics.

Claims

1. A coated fabric comprising polyester fibers dyed with dye stuff molecules, and at least one resinous coating layer, characterized in that
 - a) additionally providing fine porous inorganic particles being adhered to the surface of the polyester fibers;
 - b) said particles comprise an average particle size in the range of 1 to 100 nm;
 - c) said particles comprise fine pores having a pore diameter of 15 nm or less; and
 - d) said particles comprise a surface area of 200 m²/g or more.
2. The coated fabric according to claim 1, wherein said fine porous inorganic particles being present in an amount of 0.5 to 15 % by weight based on the weight of the polyester fibers.
3. The coated fabric according to claims 1 or 2, wherein said fine porous inorganic particles being present in an amount of 1.5 to 10 % by weight based on the weight of the polyester fibers.
4. The coated fabric according to anyone of the claims 1 to 3, wherein said particles comprise an average particle size in the range of 10 to 50 nm.

5. A coated fabric
comprising polyester fibers dyed with dye stuff molecules, and at least one resinous coating layer,
characterized in that
 - a) additionally providing fine porous inorganic particles being dispersed within at least one coating layer;
 - b) said particles comprise an average particle size of 15 μm or less;
 - c) said particles comprise fine pores having a pore diameter of 15 nm or less; and
 - d) said particles comprise a surface area of 200 m^2/g or more.
6. The coated fabric according to claim 5,
wherein said fine porous inorganic particles being present in an amount of 10 % by weight or more,
based on the weight of the solid resin content of the resinous coating layer.
7. The coated fabric according to claim 6,
wherein providing in addition at least one further resinous coating layer,
wherein at least one coating layer comprising a significant to high amount of said content of fine porous
inorganic particles; and wherein at least one further coating layer comprising a small to negligible
amount of said content of fine porous inorganic particles.
8. The coated fabric according to claim 7,
wherein said coating layer comprising a significant to high amount of fine porous inorganic particles has
a layer thickness of 3 μm or more.
9. The coated fabric according to anyone of the claims 1 to 8,
wherein said fine porous inorganic particles being selected from a group comprising silicon dioxide,
titanium oxide, zirconium oxide, aluminum oxide and active carbon.
10. The coated fabric according to anyone of the claims 1 to 9,
wherein said particles comprise fine pores having a pore diameter in the range of 1 to 10 nm.
11. The coated fabric according to anyone of the claims 1 to 10,
wherein at least one resinous coating layer comprises a polyurethane resin.
12. A method for preparing a coated fabric as defined in claim 1,
characterized by the steps:
 - a) providing a dyed fabric consisting essentially of polyester fibers;
 - b) adhering to at least one main surface of said fabric said fine porous inorganic particles as defined;
and
 - c) depositing at least one resinous coating layer to said treated surface of the fabric.
13. The method according to claim 12,
wherein in step (b) said particles are dispersed in an aqueous solution which is used for a padding
treatment of the dyed fabric.
14. A method for preparing a coated fabric as defined in claim 5,
characterized by the steps:
 - a) providing a dyed fabric consisting essentially of polyester fibers;
 - b) providing a resinous solution containing a homogeneous dispersion of fine porous inorganic
particles as defined; and
 - c) coating at least one main surface of said fabric with said resinous solution.

Patentansprüche

1. Beschichtetes Gewebe,
das Polyesterfasern aufweist, die mit Farbstoffmolekülen gefärbt sind, und das ferner wenigstens eine
Beschichtungsschicht aus Kunstharz aufweist,
dadurch gekennzeichnet, daß

- a) zusätzlich feine, poröse anorganische Partikel vorhanden sind, die an der Oberfläche der Polyesterfasern haften;
b) diese Partikel eine mittlere Partikelgröße im Bereich von 1 bis 100 nm aufweisen;
c) diese Partikel feine Poren mit einem Porendurchmesser von 15 nm oder weniger aufweisen; und
5 d) diese Partikel eine Oberfläche von 200 m²/g oder mehr aufweisen.
2. Beschichtetes Gewebe nach Anspruch 1,
wobei diese feinen, porösen anorganischen Partikel in einem Anteil von 0,5 bis 15 Gew.-% vorhanden
sind, bezogen auf das Gewicht der Polyesterfasern.
- 10 3. Beschichtetes Gewebe nach Anspruch 1 oder 2,
wobei diese feinen, porösen anorganischen Partikel in einem Anteil von 1,5 bis 10 Gew.-% vorhanden
sind, bezogen auf das Gewicht der Polyesterfasern.
- 15 4. Beschichtetes Gewebe nach einem der Ansprüche 1 bis 3,
wobei die Partikel eine mittlere Partikelgröße im Bereich von 10 bis 50 nm aufweisen.
5. Beschichtetes Gewebe,
das Polyesterfasern aufweist, die mit Farbstoffmolekülen gefärbt sind, und das ferner wenigstens eine
20 Beschichtungsschicht aus Kunstharz aufweist,
dadurch gekennzeichnet, daß
a) zusätzlich feine, poröse anorganische Partikel vorhanden sind, die in wenigstens einer Beschich-
tungsschicht dispergiert sind;
b) diese Partikel eine mittlere Partikelgröße von 15 µm oder weniger aufweisen;
25 c) diese Partikel feine Poren mit einem Porendurchmesser von 15 nm oder weniger aufweisen; und
d) diese Partikel eine Oberfläche von 200 m²/g oder mehr aufweisen.
6. Beschichtetes Gewebe nach Anspruch 5,
wobei diese feinen, porösen anorganischen Partikel in einem Anteil von 10 Gew.-% oder mehr
30 vorhanden sind, bezogen auf das Gewicht des festen Kunstharzes in der Beschichtungsschicht aus
Kunstharz.
7. Beschichtetes Gewebe nach Anspruch 6,
wobei zusätzlich wenigstens eine weitere Beschichtungsschicht aus Kunstharz vorhanden ist; und
35 wobei wenigstens eine Beschichtungsschicht einen deutlichen bis hohen Anteil des Gehalts an feinen,
porösen anorganischen Partikeln aufweist; und
wobei wenigstens eine weitere Beschichtungsschicht einen kleinen bis vernachlässigbaren Anteil des
Gehalts an feinen, porösen anorganischen Partikeln aufweist.
- 40 8. Beschichtetes Gewebe nach Anspruch 7,
wobei die Beschichtungsschicht mit einem deutlichen bis hohen Anteil an feinen, porösen anorgani-
schen Partikeln eine Schichtdicke von 3 µm oder mehr aufweist.
9. Beschichtetes Gewebe nach einem der Ansprüche 1 bis 8,
45 wobei die feinen, porösen anorganischen Partikel ausgewählt sind aus einer Gruppe, die umfaßt
Siliziumdioxid, Titanoxid, Zirkoniumoxid, Aluminiumoxid und aktiver Kohlenstoff.
10. Beschichtetes Gewebe nach einem der Ansprüche 1 bis 9,
50 wobei die Partikel feine Poren mit einem Porendurchmesser im Bereich von 1 bis 10 nm aufweisen.
11. Beschichtetes Gewebe nach einem der Ansprüche 1 bis 10,
wobei wenigstens eine Beschichtungsschicht aus Kunstharz einen Gehalt an Polyurethan aufweist.
12. Verfahren zur Herstellung eines beschichteten Gewebes nach Anspruch 1,
55 gekennzeichnet durch die Verfahrensschritte:
a) es wird ein gefärbtes Gewebe bereitgestellt, das im wesentlichen aus Polyesterfasern besteht;
b) an wenigstens einer Hauptoberfläche des Gewebes werden feine, poröse anorganische Partikel
mit den bestimmten Eigenschaften angeheftet; und

c) auf der behandelten Gewebeoberfläche wird wenigstens eine Beschichtungsschicht aus Kunstharz aufgebracht.

13. Verfahren nach Anspruch 12,

5 wobei in Verfahrensschritt (b) die Partikel in einer wässrigen Lösung dispergiert werden, mit welcher eine Padding-Behandlung (Imprägnierung, Appretierung) des gefärbten Gewebes durchgeführt wird.

14. Verfahren zur Herstellung eines beschichteten Gewebes nach Anspruch 5, gekennzeichnet durch die Verfahrensschritte:

- 10 a) es wird ein gefärbtes Gewebe bereitgestellt, das im wesentlichen aus Polyesterfasern besteht;
b) es wird eine Kunstharz-Lösung bereitgestellt, die eine homogene Dispersion der feinen, porösen anorganischen Partikel mit den bestimmten Eigenschaften enthält; und
c) wenigstens eine Hauptoberfläche dieses Gewebes wird mit dieser Kunstharz-Lösung beschichtet.

15 Revendications

1. Tissu enduit

comprenant des fibres de polyester teintes par des molécules de matière colorante, et au moins une couche résineuse d'enduction,

20 caractérisé en ce que

- a) il est prévu en outre de fines particules poreuses minérales adhérant à la surface des fibres de polyester ;
b) ces particules ont une granulométrie moyenne comprise entre 1 et 100 nm ;
c) ces particules comportent des pores fins d'un diamètre de 15 nm ou inférieur à 15 nm ;
25 d) ces particules ont une surface spécifique de 200m²/g ou supérieure à 200m²/g.

2. Tissu enduit suivant la revendication 1,

dans lequel les fines particules poreuses minérales représentent de 0 à 15% du poids des fibres de polyester.

30

3. Tissu enduit selon les revendications 1 ou 2,

dans lequel les fines particules poreuses minérales représentent de 1,5 à 10% du poids des fibres de polyester.

35 4. Tissu enduit selon l'une des quelconques revendications 1 à 3,

dans lequel les particules ont une granulométrie moyenne comprise entre 10 et 50 nm.

5. Tissu enduit

comprenant des fibres de polyester teintes par des molécules de matière colorante, et au moins une couche résineuse d'enduction,

40

caractérisé en ce que

- a) il est prévu en outre de fines particules poreuses minérales dispersées au sein d'au moins une couche d'enduction ;
b) ces particules ont une granulométrie moyenne de 15 µm ou inférieure à 15 µm ;
45 c) ces particules ont des pores fins d'un diamètre de 15 nm ou inférieur à 15 nm ;
d) ces particules ont une surface spécifique de 200 m²/g ou supérieure à 200 m²/g.

6. Tissu enduit suivant la revendication 5,

dans lequel les fines particules poreuses minérales représentent 10% ou plus de 10% du poids de la teneur en résine solide de la couche résineuse d'enduction.

50

7. Tissu enduit suivant la revendication 6,

dans lequel il est prévu en outre au moins une autre couche d'enduction résineuse,

l'une au moins des couches d'enduction ayant une teneur significative à grande en fines particules poreuses minérales et au moins une autre couche d'enduction ayant une teneur petite à négligeable en fines particules poreuses minérales.

55

8. Tissu enduit suivant la revendication 7,
dans lequel la couche d'enduction ayant une teneur significative à grande en fines particules poreuses minérales a une épaisseur de 3 μm ou supérieure à 3 μm ;
- 5 9. Tissu enduit suivant l'une des quelconques revendications 1 à 8,
dans lequel les fines particules poreuses minérales sont choisies parmi le dioxyde de silicium, l'oxyde de titane, l'oxyde de zirconium, l'oxyde d'aluminium et le charbon actif.
- 10 10. Tissu enduit suivant l'une des quelconques revendications 1 à 9,
dans lequel les particules ont des pores fins d'un diamètre compris entre 1 à 10 nm.
11. Tissu enduit suivant l'une des quelconques revendications 1 à 10,
dans lequel au moins une couche résineuse d'enduction comprend une résine de polyuréthane.
- 15 12. Procédé de préparation d'un tissu enduit tel que défini à la revendication 1,
caractérisé en ce qu'il consiste :
 - a) à prévoir un tissu teint consistant essentiellement en fibres de polyester ;
 - b) à faire adhérer à au moins une surface principale du tissu les fines particules poreuses minérales telles que définies; et
 - 20 c) à déposer au moins une couche résineuse d'enduction sur la surface traitée du tissu.
13. Procédé suivant la revendication 12,
qui consiste au stade (b) à disperser les particules dans une solution aqueuse qui est utilisée pour un traitement de foulardage du tissu teint.
- 25 14. Procédé de préparation d'un tissu enduit tel que défini à la revendication 5,
caractérisé en ce qu'il consiste :
 - a) à prévoir un tissu teint consistant essentiellement en fibres de polyester ;
 - b) à prévoir une solution résineuse contenant une dispersion homogène de fines particules poreuses minérales telles que définies ;
 - 30 c) à enduire au moins une surface principale du tissu de la solution résineuse.

35

40

45

50

55

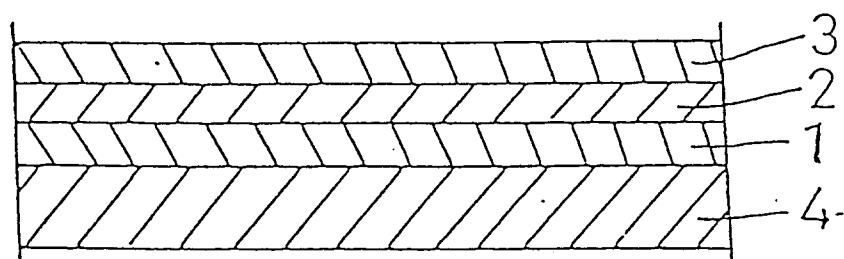


Figure 1