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EP 0 396 765 A1 54 **COATED POLYESTER FIBER FABRIC AND PROCESS FOR ITS PRODUCTION.**

57 A coated polyester fiber fabric free of stain due to migration of dispersed dyes and a process for its production are disclosed. Specifically, inorganic fine particles capable of adsorbing dyes are used to trap dyes migrating through resin and enclose dye molecules in the fine pores thereof, thus preventing stain due to migration of dye molecules to other fabric surface.

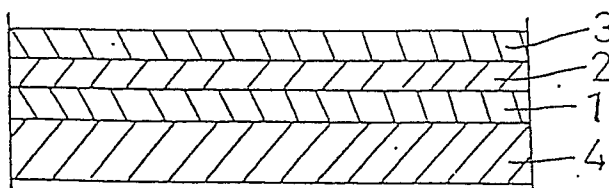
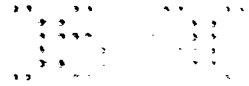


Figure 1



PATENT SPECIFICATION

A coated fabric of a polyester fiber and a method for preparation thereof

Technological Field

The present invention relates to a coated fabric of a polyester fiber exhibiting no staining caused by dye migration and a method for preparation thereof.

Background Technology

As coated fabrics used now, woven and knitted fabrics wherein nylon fibers are main components and for example, treated with such treatments as repellent and waterproof, water-vapor permeable and repellent, breathable, flame-proof and meltproof coatings are being widely used.

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

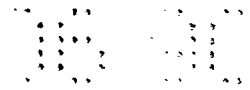
However, the coated fabrics of polyester fiber have such a fatal defect that the dye in the polyester fiber



migrates to the coated film and the coated film itself of a product the base fabric of which is dyed and other coated film which is brought into contact therewith through the film faces thereof are stained. Namely, in case of dyeing of the polyester fiber with a disperse dye, polyester fiber does not combine chemically with the fiber, for example, in contrast with the dyeing of nylon with an acid dye and in addition, the disperse dye has good solubility in and affinity with organic solvents and synthetic resins, so that the dye molecules in the fiber could easily migrate to the coated film layer.

Therefore, when coated faces of different colors are brought into contact with each other, staining is resulted in thereby. Related with this problems, various investigations to solve this problem have been done up to the present time, but any perfect solution could not be obtained and therefore, dyed product of the coated fabric of a polyester fiber has not been widely put to practical use now.

So far in both Japanese Patent Laid-Open No. 4873/1983 and Japanese Patent Publication No. 53632/1987, methods which had quite different purposes from that of the present invention, but wherein fiber structures were treated by providing a water repellent having a perfluoroalkyl group to a fiber structure on



which a polyurethane resin film containing porous particles wherein SiO_2 was the main component was formed, were proposed.

However, the purposes of these inventions were to try to obtain a waterproof fabric exhibiting both breathable and water-vapor permeable characteristics by making fine pores of particles incorporated in a polyurethane resin to paths for air and water-vapor and any suggestion on a coating film which can prevent staining caused by a disperse dye is not practically given.

Disclosure of the Invention

The present invention proposes a coated fabric of a polyester fiber wherein a disperse dye is caught with porous inorganic particles having fine pores of a specified pore diameter and migration and staining of a dye from a fabric face to another fabric face are little and a method for preparation thereof.

The polyester fiber structure in the present invention means not only woven and knitted fabrics and non-woven fabrics of 100% polyester fiber but also mixed spun, combined filament, different yarns-twisted union-woven and union-knitted fabrics wherein polyester fibers are essential components and no limitation exists, but the effect is more remarkable when 100% polyester or

the polyester fiber with a high rate of blend both dyed with a dispersed dye is used.

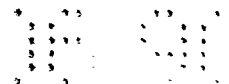
As the porous inorganic particles used in the present invention, silicon dioxide, titanium oxide, zirconium oxide, aluminum oxide, active carbon etc., are used. Among them, silicon dioxide can be most effectively used from the point of effect and utility.

The dimension of the fine pore diameter in the porous inorganic particles largely influences the adsorption effect of the migrated dye. Therefore, the one with a fine pore diameter of 150\AA or smaller can be used. A diameter of $10 - 100\text{\AA}$ is preferable from the point of the effect. If it is larger than 150\AA , adsorption and retention of the migrated dye is not sufficient.

In addition, the surface area of the porous inorganic particle is preferably $200\text{ m}^2/\text{g}$ or larger and more preferably $500\text{ m}^2/\text{g}$. If the surface area is smaller than $200\text{ m}^2/\text{g}$, the effect of the present invention is not sufficient.

As the resin material used in the present invention, various resins used for ordinary coating treatments such as urethane, acrylic, silicone, vinyl chloride and vinyl acetate can be freely selected.

Next, a method for preparation of a coated fabric in the present invention will be described.

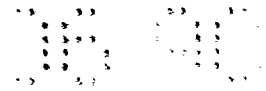


There are two methods. The first one is a method wherein porous inorganic particles are adhered on the fiber surface before coating and the second one is a method wherein the porous inorganic particles are incorporated in a film of a coating resin.

The first method, namely a method wherein porous fine inorganic particles are adhered on the surface of the fiber before coating will be explained. It is essential to make the porous fine inorganic particles adhered on the surface of the fiber uniform to exhibit fully the effect of the present invention. From the view point of handling and workability, it is a preferable method that an ag. dispersion of the porous fine inorganic particles is adhered by means of padding. The drying temperature after padding should be at 80°C - 160°C, preferably in the range of 100°C - 130°C, and above 160°C, the effect decreases. As the amount of adhered porous fine inorganic particles, 0.5 - 15 wt.% is pref. based on the weight of the fabric and 1.5 - 10 wt.% is more preferable.

As the particle size of the porous fine inorganic particles to be used, the particles in the range of 1 - 100 μ can be used and those in the range of 10 - 50 μ are usually preferably used.

To improve durability of the porous fine inorganic particles adhered on the surface of the fiber, a method



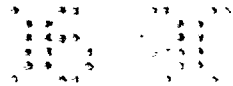
for using an ordinary resin for finishing in parallel is preferably used.

The second method is a method dispersing in advance porous fine inorganic particles in a resin for coating and the particle size to be used is 15 μm at maximum and the smaller one is preferable. In this method, good dispersion of the porous fine inorganic particle in the resin is important. Agglomeration of particles causes decrease in the effect of staining prevention and deterioration of coating quality.

In the second method, there exists a method wherein porous fine inorganic particles are lamellarly distributed in the resin film. For example, as illustrated in Figure 1, a lamellar structure consisting of a resin layer A wherein porous fine inorganic particles are lamellarly dispersed at a high concentration and a resin layer B wherein the porous fine inorganic particles are little or not incorporated is cited.

Practically speaking, a resin solution A containing 10% or more inorganic particles based on the weight of the solid content of the resin as a resin layer containing inorganic particles with a high concentration and a resin solution B containing no or less than 10% inorganic particles as a resin layer are prepared. As the order of coating, either a method wherein the first layer 1 of the

fiber layer 4 is prepared by coating with the resin solution B and the second layer 2 is prepared by coating with the resin solution A or a method where the procedure is done in the reverse order can be used. In addition, to prepare a triple layered structure by coating, either the first, second or third layer in Figure 1 is coated with the resin solution A and other two layers are coated with the resin solution B. In case when the first layer 1 is coated with the resin solution A for coating said double structure or triple structure, adhesiveness with the fiber decreases in some cases. If the adhesiveness with the fiber is especially required, it is preferable that the second layer 2 or the third layer is coated with the resin solution A. In addition, it is preferable that the thickness of the resin layer A wherein porous fine inorganic particles are lamellarly dispersed at a high concentration is 3 μm or thicker. No limitation exists on the coating method. Especially, in the method wherein porous fine inorganic particles with a high concentration are lamellarly incorporated in a resin for coating, as the porous fine inorganic particles with a high concentration catch completely dye molecules migrating from a fiber into a resin film and the dye molecules are adsorbed and kept in the fine pores which the porous fine inorganic particles have, the present invention exhibits thereby an



effect preventing permanently dye from migration.

Brief Explanation of the Drawing

Figure 1 is a cross-sectional figure illustrating an example of a coated fabric of a polyester fiber obtained in Example 4 of the present invention.

- 1: The first layer
- 2: The second layer
- 3: The third layer
- 4: The fiber layer

The Best Embodiment for Practicing the Present Invention

The present invention will be explained in more detail by using Examples hereinbelow, but not restricted by these Examples.

(1) Evaluation of fastness to dye migration and staining in Examples was performed by means of the following method.

A test piece (5 cm x 5 cm) and attached white polyester fabrics (the raw fabric of which was the same as the said test piece and which were coated with the same resin as the one 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 and they were placed in a constant temperature oven (at $100^{\circ}\text{C} \pm 2^{\circ}\text{C}$) for 48 hr while a loading of 200 g was applied thereon. After cooling it, the state of dye migration from the test piece to the attached white fabric was evaluated in terms of a classification by means of a grey scale for evaluating staining.

The results obtained in Examples and Comparative Examples were summarized in Table 1.

(2) In addition, 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

("Cryscot 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

A plain woven fabric prepared by using polyester filaments of each 50 denier as a warp and 75 denier as a weft was dyed with a disperse dye "Resoline Blue FBL" of

3% o.w.f. at 130°C for 60 min and was washed as usual. A dyed fabric for coating was obtained by performing heat-setting treatment at 180°C after drying.

Then, padding of this fabric was performed with an aqueous solution wherein 30 g/l of a trimethylolmelamine and a silicon dioxide with a particle diameter of 20 μm , a fine pore diameter of 60 \AA and a surface area of 300 m^2/g which is 15% of the solid content of the resin were dispersed and the fabric was dried at 130°C for 1 min. The build-up of the silicon dioxide was 2.4%. Then, it was coated with a polyether polyurethane resin solution in dimethylformamide by means of a knife coater and the solution was coagulated by means of a wet process to obtain a coated fabric with a coating weight of 25 g/m^2 .

Comparative Example 1

The dyed fabric obtained in Example 1 was wet-coated only with a polyether polyurethane resin solution in dimethylformamide without treating with silicon dioxide.

Example 2

A dyed fabric for coating was obtained by the same method as that of Example 1. Then, 15% of a silicon dioxide based on the solid content of the resin with a particle diameter of 3 μm , a fine pore diameter of 50 \AA and a surface area of 500 m^2/g were dispersed in a polyether polyurethane resin solution in dimethylformamide and the

fabric was coated with this solution by means of a knife coater to obtain a coated fabric.

Comparative Example 2

A coated fabric using a silicon dioxide with a particle diameter of 20 μm , a fine pore diameter of 210 \AA and a surface area of 150 m^2/g was obtained by the same method as that of Example 2.

Example 3

A coated fabric was obtained by the same method as that of Example 2 except using an acrylic and a silicone resin as the coating resin.

Comparative Example 3

A coated fabric was obtained by the same method as that of Example 3 except using separately each an acrylic resin and a silicone resin without silicon dioxide.

Example 4

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

Then, it was coated with a polyether polyurethane resin solution in dimethylformamide as a coating resin by means of a knife coater and the coated resin solution was coagulated by means of a wet process to obtain a film.

30% of silicon dioxide based on the solid content of the resin with an inorganic particle diameter of 3 μm , a fine pore diameter of 50 \AA and surface area of 500 m^2/g

were dispersed in the same resin solution and the obtained coated fabric was coated with this solution by means of a knife coater and the coated solution was coagulated by means of a wet process to make a top coat of a film. Furthermore, this coated fabric was coated with the same resin containing no porous fine inorganic particle by means of a knife coater and the coated resin solution was coagulated by means of a wet process to obtain a coated fabric of a triple layered structure wherein an intermediate inorganic particle layer existed. The thickness of the porous fine inorganic particle layer was 10 μm .

Example 5

A coated fabric was obtained by the same method as the one in Example 1 except using acrylic and silicon resins as the coating resins. The thickness of the layer of porous fine inorganic particles was 10 μm .

Comparative Example 4

A dyed fabric for coating was obtained by the same method as the one in Example 1. Then, 15% of silicon dioxide with a particle diameter of 3 μ , a pore volume of 0.5 cc/g, a pore diameter of 170 \AA and a surface area of 300 m^2/g based on the solid content of the resin were dispersed in a polyester polyurethane resin solution in dimethylformamide and the fabric was coated with the

obtained solution by means of a knife coater to obtain a coated fabric.

Table 1

	Condition		Fastness to migration and staining
	Silicon dioxide	Resin	
Example 1	20 μm	Urethane	4 - 5
Comparative Example 1	—	Urethane	2
Example 2	3 μm , 50 \AA 500 m^2/g	Urethane	4 - 5
Comparative Example 2	20 μm , 210 \AA 150 m^2/g	Urethane	2
Example 3	3 μm , 50 \AA 500 m^2/g	Acrylic	4 - 5
	3 μm , 50 \AA 500 m^2/g	Silicon	4 - 5
Comparative Example 3	—	Acrylic	1
	—	Silicon	1
Example 4	3 μm , 50 \AA 500 m^2/g	Urethane	5



Table 1 (Continued)

	Condition		Fastness to migration and staining
	Silicon dioxide	Resin	
Example 5	3 μm , 50 \AA 500 m^2/g	Acrylic	4 - 5
	3 μm , 50 \AA 500 m^2/g	Silicon	4 - 5
Comparative Example 4	3 μm , 170 \AA 300 m^2/g	Urethane	2 - 3
		Silicon	1

Notice: The column of silicon dioxide shows the average particle diameter, the average particle pore pore diameter and the surface area from the top.

Possibility of Industrial Application

The coated fabric of the present invention can be widely used for clothings and for industrial uses as various products treated with such treatments as repellent and waterproof, water-vapor permeable and repellent, breathable, flameproof and meltproof coatings.

The coated fabric of the present invention especially supplements the defects of the coated fabrics of nylon fibers such as prices, dimensional stability, light resistance and versatility of raw materials and substitutes for a part of its demand. Developments of new uses can be also expected thereby.

Claims

1. A coated fabric of a polyester fiber characterized by a porous fine inorganic particle having fine pores with a pore diameter of 150\AA or smaller being adhered on the fiber surface or incorporated in a coating resin film.

2. A coated fabric of a polyester fiber as described in Claim 1 wherein a porous fine inorganic particle is at least one compound selected from a group consisting of silicon dioxide, titanium oxide, zirconium oxide, aluminum oxide and active carbon.

3. A coated fabric of a polyester fiber as described in Claim 1 wherein a porous fine inorganic particle is a fine particle with a surface area of $200\text{ m}^2/\text{g}$ or larger.

4. A coated fabric of a polyester fiber as described in Claim 1 characterized by said porous fine inorganic particles being ununiformly distributed in the thickness direction of a coated film.

5. A coated fabric of a polyester fiber as described in Claim 1 characterized by said porous fine inorganic particle with an average pore diameter of the fine pores of $10 - 100\text{\AA}$.

6. A coated fabric of a polyester fiber as described in Claim 1 characterized by said resin being a

polyurethane resin.

7. A coated fabric of a polyester fiber as described in Claim 1 characterized by the amount of adhesion of said porous fine inorganic particle being 1.5 - 10 wt.% based on the weight of the fiber.

8. A method for preparing a coated fabric of a polyester fiber characterized by adhering the surface of a fabric of a polyester fiber with porous fine inorganic particles having fine pores with an average pore diameter of 150Å or smaller and performing a coating treatment on said surface.

9. A method for preparing a coated fabric of a polyester fiber as described in Claim 8 characterized by said porous fine inorganic particle with an average particle diameter of 10 - 50 µm.

10. A method for preparing a coated fabric of a polyester fiber characterized by coating a fabric with a resin solution containing porous fine inorganic particles having fine pores with an average pore diameter of 150Å or smaller.

11. A method for preparing a coated fabric of a polyester fiber as described in Claim 10 characterized by said porous fine inorganic particle with an average particle diameter being 15 µm or smaller.

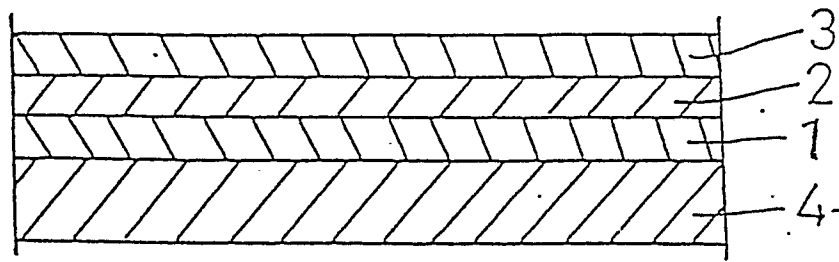


Figure 1

INTERNATIONAL SEARCH REPORT

International Application No PCT/JP89/01006

I. CLASSIFICATION OF SUBJECT MATTER (If several classification symbols apply, indicate all) ⁴ According to International Patent Classification (IPC) or to both National Classification and IPC <div style="display: flex; justify-content: space-between; padding: 5px 0;"> Int. Cl⁴ D06M11/00, D06M11/12, D06M15/00, D06P5/02 </div>																	
II. FIELDS SEARCHED <div style="text-align: center; padding: 5px 0;">Minimum Documentation Searched ⁷</div> <table border="1" style="width: 100%; border-collapse: collapse;"> <tr> <th style="width: 30%; padding: 5px;">Classification System</th> <th style="padding: 5px;">Classification Symbols</th> </tr> <tr> <td style="text-align: center; padding: 10px;">IPC</td> <td style="padding: 10px;">D06M11/00, D06M11/12, D06M15/00, D06P5/02</td> </tr> </table> <div style="text-align: center; padding: 5px 0;">Documentation Searched other than Minimum Documentation to the Extent that such Documents are Included in the Fields Searched ⁸</div> <div style="display: flex; justify-content: space-between; padding: 5px 0;"> <div style="width: 45%;"> <p>Jitsuyo Shinan Koho</p> <p>Kokai Jitsuyo Shinan Koho</p> </div> <div style="width: 50%;"> <p>1926 - 1989</p> <p>1971 - 1989</p> </div> </div>			Classification System	Classification Symbols	IPC	D06M11/00, D06M11/12, D06M15/00, D06P5/02											
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III. DOCUMENTS CONSIDERED TO BE RELEVANT ⁹ <table border="1" style="width: 100%; border-collapse: collapse;"> <tr> <th style="width: 10%; padding: 5px;">Category ⁹</th> <th style="width: 70%; padding: 5px;">Citation of Document, ¹¹ with indication, where appropriate, of the relevant passages ¹²</th> <th style="width: 20%; padding: 5px;">Relevant to Claim No. ¹³</th> </tr> <tr> <td style="text-align: center; vertical-align: top; padding: 5px;">Y</td> <td style="padding: 5px;">JP, A, 60-9983 (Soko Seiren Kabushiki Kaisha) 19 January 1985 (19. 01. 85) Page 1, lower left column, lines 4 to 21, page 2, upper right column, lines 1 to 3, page 2, lower right column, lines 3 to 6 (Family : none)</td> <td style="text-align: center; vertical-align: top; padding: 5px;">1 - 11</td> </tr> <tr> <td style="text-align: center; vertical-align: top; padding: 5px;">A</td> <td style="padding: 5px;">JP, A, 60-45680 (Kitanihon Senko Kabushiki Kaisha) 12 March 1985 (12. 03. 85) (Family : none)</td> <td style="text-align: center; vertical-align: top; padding: 5px;">1 - 11</td> </tr> <tr> <td style="text-align: center; vertical-align: top; padding: 5px;">A</td> <td style="padding: 5px;">JP, A, 60-39479 (Kitanihon Senko Kabushiki Kaisha) 1 March 1985 (01. 03. 85) (Family : none)</td> <td style="text-align: center; vertical-align: top; padding: 5px;">1 - 11</td> </tr> <tr> <td style="text-align: center; vertical-align: top; padding: 5px;">A</td> <td style="padding: 5px;">JP, B2, 60-59345 (Kuraray Co., Ltd.) 24 December 1985 (24. 12. 85) (Family : none)</td> <td style="text-align: center; vertical-align: top; padding: 5px;">1 - 11</td> </tr> </table>			Category ⁹	Citation of Document, ¹¹ with indication, where appropriate, of the relevant passages ¹²	Relevant to Claim No. ¹³	Y	JP, A, 60-9983 (Soko Seiren Kabushiki Kaisha) 19 January 1985 (19. 01. 85) Page 1, lower left column, lines 4 to 21, page 2, upper right column, lines 1 to 3, page 2, lower right column, lines 3 to 6 (Family : none)	1 - 11	A	JP, A, 60-45680 (Kitanihon Senko Kabushiki Kaisha) 12 March 1985 (12. 03. 85) (Family : none)	1 - 11	A	JP, A, 60-39479 (Kitanihon Senko Kabushiki Kaisha) 1 March 1985 (01. 03. 85) (Family : none)	1 - 11	A	JP, B2, 60-59345 (Kuraray Co., Ltd.) 24 December 1985 (24. 12. 85) (Family : none)	1 - 11
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<div style="display: flex; justify-content: space-between;"> <div style="width: 45%;"> <p>¹⁰ Special categories of cited documents:</p> <p>"A" document defining the general state of the art which is not considered to be of particular relevance</p> <p>"E" earlier document but published on or after the international filing date</p> <p>"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)</p> <p>"O" document referring to an oral disclosure, use, exhibition or other means</p> <p>"P" document published prior to the international filing date but later than the priority date claimed</p> </div> <div style="width: 50%;"> <p>"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention</p> <p>"X" document of particular relevance: the claimed invention cannot be considered novel or cannot be considered to involve an inventive step</p> <p>"Y" document of particular relevance: the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art</p> <p>"Z" document member of the same patent family</p> </div> </div>																	
IV. CERTIFICATION <table border="1" style="width: 100%; border-collapse: collapse;"> <tr> <td style="width: 50%; padding: 5px;">Date of the Actual Completion of the International Search</td> <td style="width: 50%; padding: 5px;">Date of Mailing of this International Search Report</td> </tr> <tr> <td style="text-align: center; padding: 5px;">October 19, 1989 (19. 10. 89)</td> <td style="text-align: center; padding: 5px;">October 30, 1989 (30. 10. 89)</td> </tr> <tr> <td style="padding: 5px;">International Searching Authority</td> <td style="padding: 5px;">Signature of Authorized Officer</td> </tr> <tr> <td style="text-align: center; padding: 5px;">Japanese Patent Office</td> <td></td> </tr> </table>			Date of the Actual Completion of the International Search	Date of Mailing of this International Search Report	October 19, 1989 (19. 10. 89)	October 30, 1989 (30. 10. 89)	International Searching Authority	Signature of Authorized Officer	Japanese Patent Office								
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