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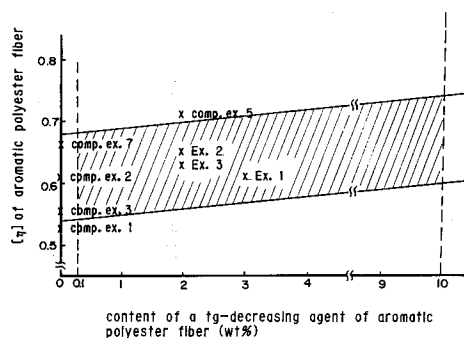
(54) **Cationically dyeable polyester fiber.**

(57) A polyester fiber comprising an aromatic polyester having sulfonic acid salt groups, said fiber containing a specific glass transition point-decreasing agent and a solid fine powder, the intrinsic viscosity $[\eta]$ of said polyester fiber and the content (A% by weight) of said glass transition point-decreasing agent in said polyester fiber satisfying the following condition ①. The fiber has high strength, brightish transparency and good processability and is dyeable with cationic dyes.

$$0.01 \times A + 0.54 \leq [\eta] \leq 0.01 \times A + 0.68 \quad \text{①}$$

wherein $0.1 \leq A \leq 10$.

Fig 1



BACKGROUND OF THE INVENTION

1. Field of the invention

The present invention relates to a polyester fiber being dyeable with cationic dyes and, at the same time, having sufficiently high strength and resistance to flexural fatigue. The fiber has good processability and shows, upon dyeing with cationic dyes, vivid and bright shade and is hence usable for sports wears and like clothing.

2. Description of the prior art

Polyester fibers, in particular polyethylene terephthalate fibers, have excellent mechanical and chemical characteristics and are widely used for clothing and industrial materials.

Polyester fibers have, however, poor dyeability and, in particular, are hardly dyeable with dyes other than disperse dyes. Various proposals have been made to improve the dyeability.

The most representative process for the improvement comprises copolymerizing an isophthalic acid component having a sulfonic acid metal salt, such as sodium 5-sulfoisophthalate (hereinafter referred to as "SIP"), into a raw material polyester. See, for example, Japanese Patent Publication No. 10497/1959.

To obtain dyeability of satisfactory level, however, it is necessary to copolymerise a large amount of SIP. Copolymerization of SIP in a large amount significantly increases the melt viscosity of the resulting copolyester so that it becomes difficult both to increase the degree of polymerization of the copolyester sufficiently and to spin the resulting copolyester stably. One may seek to solve this problem by decreasing the degree of polymerization of the copolyester to such a level that assures ready polymerization and stable melt spinning. Then, there generates another problem of the obtained fiber being only of poor strength with limited end-uses.

For the purpose of obtaining a high-strength fiber of polyethylene terephthalate (hereinafter referred to as "PET") having good spinnability and high degree of polymerization, Japanese Patent Application Laid-open No. 223382/1991 discloses a viscosity-decreasing agent which is added to a PET having high intrinsic viscosity and decreases its melt viscosity. Simple addition of this viscosity-decreasing agent, however, causes, while improving the spinnability though, many fluffs and frequent filament breakage during drawing process of the fiber when it is drawn in a high ratio to obtain high strength.

Japanese Patent Publication No. 22334/1972 (U.S.P. 3,732,183) discloses a process for producing cationically dyeable polyester fiber which comprises using a copolyester containing a copolymerization component of an isophthalic acid having a sulfonic acid phosphonium salt group. With this process, only small thickening effect is exerted during polymerization reaction so that there can be obtained a copolyester having high degree of polymerization and being melt-spinnable by the usual procedure.

With this process, however, the isophthalic acid component having sulfonic acid phosphonium salt group has lower thermal resistance than the above isophthalic acid component having sulfonic acid metal salt group. This causes, under high temperature conditions of polymerization, melt spinning and like processes, decomposition of the isophthalic acid component having sulfonic acid phosphonium salt group itself or the resulting polyester. Then, the degree of polymerization of the polyester decreases or the resulting fiber or shaped articles formed therefrom discolors. The obtained polyester, thus being yellowish brown, cannot give good color shade upon dyeing.

To improve the thermal resistance of polyesters having copolymerized the isophthalic acid component having sulfonic acid phosphonium salt, Japanese Patent Publication No. 61766/1991 proposes addition of an agent that improves the thermal resistance and comprises a non-reactive quaternary onium salt. This method has, however, another drawback that the resulting drawn fiber that exhibits high strength initially, tends, upon false twisting or dyeing treatment, to decrease its strength, i.e. its strength retention is low.

SUMMARY OF THE INVENTION

As a results of an intensive study to obtain high strength of SIP-copolymerized polyester by means of conducting drawing in a higher ratio than that conventionally employed, the present inventors have found the following facts.

If the degree of polymerization of the SIP-copolymerized polyester is increased, within a limit to assure melt spinnability, to obtain a high-strength fiber therefrom, the drawability of the as-spun fiber will decrease so that it becomes difficult to obtain the intended high-strength fiber. On the other hand, if the degree of polymerization is decreased, the drawability will increase and high-ratio drawing will be

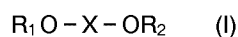
achieved, with a result that the resulting drawn fiber has not sufficient strength due to low degree of polymerization. The key fact found by the present inventors is that incorporation of a specific amount of a glass transition point (hereinafter referred to as "Tg")-decreasing agent and a solid fine powder in a fiber comprising SIP-copolymerized polyester makes it possible, by synergy effect of these two modifications, to draw the fiber in a high drawing ratio and uniformly, thereby yielding a high-strength fiber having good resistance to flexural fatigue, without causing the problems of fluff generation and uneven drawing.

Thus, the present invention provides a cationically dyeable polyester fiber comprising an aromatic polyester having sulfonic acid salt groups, said fiber containing 0.1 to 10% by weight of a Tg-decreasing agent and 0.1 to 1% by weight of a solid fine powder, the intrinsic viscosity $[\eta]$ of said polyester fiber and the content (A% by weight) of said Tg-decreasing agent in said polyester fiber satisfying the following condition ①

$$0.01 \times A + 0.54 \leq [\eta] \leq 0.01 \times A + 0.68 \quad \text{①}$$

wherein $0.1 \leq A \leq 10$.

It is desirable that, in the above cationically dyeable polyester fiber, the Tg-decreasing agent be a compound represented by the following general formula (I), the sulfonic acid salt groups be from sodium 5-sulfoisophthalate and the solid fine powder be a silica having an average particle diameter of 15 to 70 μm and further that the fiber be a microfine fiber having a single filament fineness of not more than 1.5 deniers



wherein X represents an aromatic group and R_1 and R_2 each represents an alkyl or arylalkyl group having 6 to 18 carbon atoms.

BRIEF DESCRIPTION OF THE DRAWING

A more complete appreciation of the invention and many of the attendant advantages thereof will be readily obtained as the same become better understood by reference to the following detailed description when considered in connection with the accompanying drawing, wherein:

FIGURE 1 shows the relationship between the intrinsic viscosity $[\eta]$ and content (A% by weight) of a Tg-decreasing agent observed on fibers comprising an aromatic polyester having sulfonic acid salt group, containing the Tg-decreasing agent and a solid fine powder.

DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENTS OF THE INVENTION

The aromatic polyester having sulfonic acid salt groups used in the invention is an aromatic polyester that is obtained from an acid component principally comprising terephthalic acid and at least one glycol component and comprises a copolymerization component of a dicarboxylic acid having a sulfonic acid salt group. It is preferred to use as the glycol component at least one member selected from ethylene glycol, trimethylene glycol or tetramethylene glycol. It is particularly preferred in view of fiber properties that the aromatic polyester principally comprise ethylene terephthalate units and/or butylene terephthalate units.

The aromatic polyester used in the invention may, as required, comprise other copolymerization components than components of terephthalic acid, dicarboxylic acid having sulfonic acid salt group and the above glycol, in amounts within limits not to impair the effect of the present invention.

Examples of suitable dicarboxylic acid components having a sulfonic acid salt group to be copolymerized into the aromatic polyester are dicarboxylic acids having a sulfonic group, e.g. sodium 5-sulfoisophthalate, potassium 5-sulfoisophthalate and lithium 5-sulfoisophthalate, and 5-tetraalkyl-phosphonium sulfoisophthalates, e.g. 5-tetrabutylphosphonium sulfoisophthalate and 5-ethyltributyl-phosphonium isophthalate, among which sodium 5-sulfoisophthalate is most preferable. These dicarboxylic acid components may be copolymerized into the aromatic polyester singly or in combination.

In the present invention, it is preferable that the dicarboxylic acid component having a sulfonic acid salt group be copolymerized in an amount of 1.2 to 3.0 mol% based on the total moles of the dicarboxylic acid components constituting the aromatic polyester. If the amount is less than 1.2 mol%, the resulting polyester will not give a fiber that produces, when dyed with a cationic dye, a good vivid and bright color. On the other hand, if the amount exceeds 3.0 mol%, the resulting polyester will have too high a melt viscosity to be spun stably and, further, give a fiber having too many dyeable positions that absorbs, when dyed with a

cationic dye, too large amount of the dye to show bright color. The amount of copolymerization of the dicarboxylic acid component having a sulfonic acid salt is preferably in a range of 1.5 to 2.0 mol% in view of spinnability of the polymer and the brightness of color, when dyed, of the fiber obtained therefrom.

The above aromatic polyester can be synthesized by any optional process. For example, to synthesize a polyester principally comprising ethylene terephthalate units, there may be employed the usual process which comprises, while adding a dicarboxylic acid having a sulfonic acid salt group, directly esterifying terephthalic acid and ethylene glycol, conducting transesterification of a lower alkyl ester of terephthalic acid and ethylene glycol or reacting terephthalic acid with ethylene oxide, to obtain glycol ester of terephthalic acid or a low grade polymer thereof, and then heating it under reduced pressure to effect polycondensation to the desired degree of polymerization.

The dicarboxylic acid having a sulfonic acid salt may be added at any time that assures its incorporation as a copolymerization component into the resulting polyester. Thus, it is added, for example, to a starting material of the polyester or after esterification and before polycondensation.

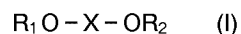
In the present invention, it is necessary that the aromatic polyester having sulfonic acid salt groups constituting the fiber incorporate a Tg-decreasing agent and a solid fine powder. The incorporation realizes that the fiber is dyeable with cationic dyes and, at the same time, has high strength and good flexural resistance.

The mechanism of the fiber of the present invention having high strength and good flexural resistance is not quite clear, but it is considered to be as follows. The Tg-decreasing agent acts to increase the mobility of the polyester molecular chains, thereby markedly improving the drawability of the fiber. As a result, high-ratio drawing becomes possible and the obtained drawn fiber has high strength. Such high-ratio drawing, however, causes the drawn fiber to generate fluffs or uneven drawing, so that incorporation of the Tg-decreasing agent alone hardly gives high-quality fiber. Incorporation of a solid fine powder, together with the Tg-decreasing agent, can, quite unexpectedly, prevent the generation of fluffs or uneven drawing, to give a uniform fiber having high strength.

It is also considered that the incorporation of a solid fine powder together with a Tg-decreasing agent improves the mobility of the polyester molecular chains, not only in the direction of the fiber length but also against bending of the fiber.

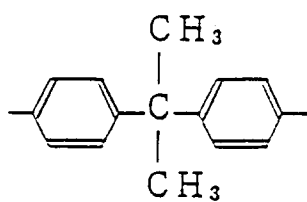
The Tg-decreasing agent used in the invention should have the property of, when added to an aromatic polyester having sulfonic acid salt groups in an amount of 2% by weight, decreasing the Tg of the polyester by at least 2°C as compared with that of the polyester without the addition of the agent. The Tg of the aromatic polyester can readily be determined by DSC or like methods.

In the present invention, there can be used any Tg-decreasing agent insofar as it can decrease, when added in an amount of 2% by weight, the Tg of the aromatic polyester having sulfonic acid salt groups by at least 2°C. It is, however, preferred to use at least one compound represented by the following general formula (I):

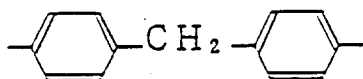


wherein X represents an aromatic group and R₁ and R₂ each represents an alkyl or arylalkyl group having 6 to 18 carbon atoms.

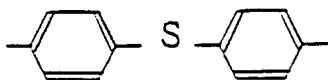
Examples of the aromatic group X in the above compound represented by formula (I) are as follows.



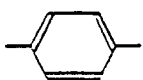
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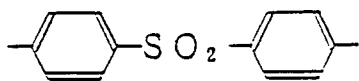
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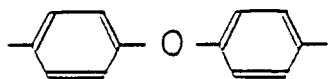
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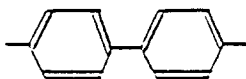
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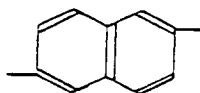
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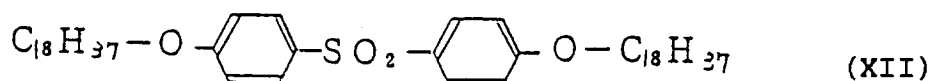
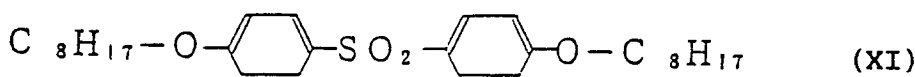
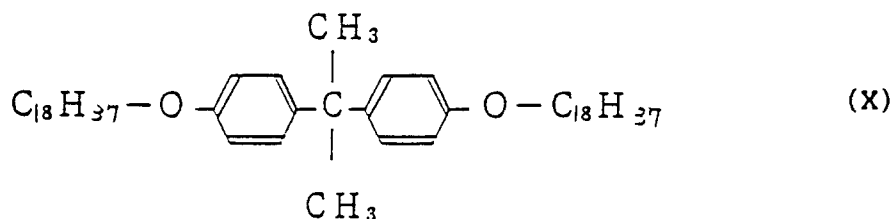


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(IX)

Examples of suitable Tg - decreasing agent used in the present inventions are as follows.



The Tg-decreasing agent is incorporated in an amount of 0.1 to 10% by weight based on the weight of the aromatic polyester constituting the fiber of the present invention. If the amount is less than 0.1% by weight, the Tg-decreasing effect will not be sufficiently produced so that high-ratio drawing cannot be performed. On the other hand, if the amount exceeds 10% by weight, the resulting polyester will discolor or the color fastness of the fiber obtained therefrom will decrease. It is preferable that the content of the Tg-decreasing agent be 1 to 5% by weight.

The solid fine powder used in the present is selected from those having an average particle diameter of 15 to 70 μm . Among such solid powders, inorganic fine powders having an average particle diameter of 15 to 70 μm are particularly preferred. With the average particle diameter being less than 15 μm , there tend to generate drawn fluffs and uneven drawing. With the average diameter exceeding 70 μm , the resultant fiber tends to fibrillate. The solid fine powder may be surface-treated by aluminum-coating, alkyl group-introduction or like methods. The solid fine powder may have any refractive index. Where, however, it is desired to maintain the brightish transparency of the aromatic polyester used, the refractive index is recommendably not more than 1.65, which is smaller than that of aromatic polyesters. For example, it is even possible to maintain the desired brightish transparency of the aromatic polyester by using a solid fine powder having a refractive index of not more than 1.65 in combination with another solid fine powder having one exceeding 1.65. Examples of the solid fine powders usable in the present invention are fine powders of silica, alumina, kaoline and calcium carbonate each having a refractive index of not more than 1.65, among which silica having an average particle diameter of 15 to 70 μm is particularly preferred.

The content of the solid fine powder is 0.1 to 1.0% by weight based on the weight of the polyester fiber of the present invention. If the content is less than 0.1% by weight, good drawability due to synergy effect producible in combination with a Tg-decreasing agent will not develop so that high-ratio drawing cannot be performed. On the other hand, if the content exceeds 1.0% by weight, the resulting drawn fiber will be of low strength. The content is preferably in a range of 0.3 to 0.5% by weight.

The Tg-decreasing agent and the solid fine powder can be added to the aromatic polyester at any time and by any process, with no particular restrictions. For example, the Tg-decreasing agent and the solid fine powder may be added at the same time, successively or at separate occasions. Or, for example, the Tg-decreasing agent and the solid fine powder may be added at appropriate points before, during or after polymerization, added as a master batch to polyester or added during melt spinning.

In the present invention, the polyester fiber may, as required, incorporate additives that are used for conventional polyester fibers and other than the Tg-decreasing agent and the solid fine powder.

In the present invention, the cationically dyeable polyester fiber preferably has a strength of at least 4.3 g/d and an elongation of at least 26%. For this purpose, in the fiber comprising an aromatic polyester having sulfonic acid salt groups and containing a Tg-decreasing agent and a solid fine powder (hereinafter referred to as "the aromatic polyester fiber"), it is necessary that the intrinsic viscosity $[\eta]$ and the content (A% by weight) of the Tg-decreasing agent satisfy the following condition ①

$$0.01 \times A + 0.54 \leq [\eta] \leq 0.01 \times A + 0.68 \quad \text{①}$$

wherein $0.1 \leq A \leq 10$.

The condition is explained by reference to FIGURE 1. Where the $[\eta]$ of the aromatic polyester fiber is positioned above the hatched area, i.e. $[\eta]$ is larger than $(0.01 \times A + 0.68)$, the aromatic polyester has too large thickening effect to be spun stably. On the other hand, where the $[\eta]$ is below the hatched area, i.e. $[\eta]$ is smaller than $(0.01 \times A + 0.54)$, the aromatic polyester has too low polymerization degree to give a fiber having sufficiently high strength.

The aromatic polyester fiber of the present invention can be produced by the usual melt spinning process from a mixture comprising an aromatic polyester having sulfonic acid groups, a Tg-decreasing agent and a solid fine powder. An example of the process is as follows. The aromatic polyester having sulfonic acid groups and containing a Tg-decreasing agent and a solid fine powder is melt spun at a temperature of about 280 to about 310°C to give an as-spun fiber. The as-spun fiber is preheated through a hot roll to a temperature above the Tg of the aromatic polyester (this temperature is generally in a range of 70 to 95°C) and then drawn by taking up onto a take-up roll the speed of which is adjusted so that the designated drawing ratio (in general 3.0 to 5.0) is obtained. The thus heat-drawn fiber is heat treated at a temperature above the crystallization temperature of the aromatic polyester (in general 120 to 180°C), to give a product drawn fiber. The drawn fiber thus obtained has high strength with no fluffs or unevenly drawn portions and is markedly suitable as a cationically dyeable fiber.

The drawn fiber may further be subjected to false-twisting.

As another example, a mixture comprising an aromatic polyester having sulfonic acid salt group, a Tg-decreasing agent and a solid fine powder is melt-spun at a temperature of about 280 to about 310°C and taken up at a high speed of at least 2,000 m/min, in particular at least 3,000 m/min, to give a highly oriented undrawn fiber. The undrawn fiber thus obtained is then subjected to processing of drawing-false twisting.

The aromatic polyester fiber of the present invention can be woven, knit or otherwise processed, to give fabrics or the like which are provided for various uses.

The fineness of the aromatic polyester fiber before drawing or after drawing is not particularly restricted but the drawn fiber or undrawn fiber preferably has a single fiber fineness of not more than 1.5 deniers, more preferably not more than 1.0 deniers, because this fineness range gives the resulting fabrics good flexibility and hand.

Further in the present invention, the aromatic polyester fiber may be a composite fiber or mixed-spun fiber comprising an aromatic polyester having sulfonic acid salt groups and containing a Tg-decreasing agent and a solid fine powder and, also, another polymer.

The aromatic polyester fiber of the present invention, which preferably has a strength of at least 4.3 g/d and an elongation of at least 26%, is excellent in resistance to flexural fatigue and hence fabrics obtained therefrom have high tear strength and are suitably used in the fields of sports wear and the like.

Other features of the invention will become apparent in the course of the following descriptions of exemplary embodiments which are given for illustration of the invention and are not intended to be limiting thereof. In the examples that follow, various measurement data were obtained according to the following methods.

(1) Intrinsic viscosity $[\eta]$ of the aromatic polyester fiber

A fiber specimen is dissolved in a 1/1 by weight mixed solvent of phenol/tetrachloroethane. The solution is filtered and then subjected to viscosity measurement at 30°C.

(2) Strength (g/d) and elongation (%) of drawn fiber and fiber taken out from fabric

JIS L1013 is applied.

(3) Degree of flexural fatigue (H/H_0)

A sample fiber is woven into a 1/1 plain weave fabric having cover factors of $K_1 = 18.5$ and $K_2 = 13$. Specimens having a width of 1.5 cm are cut from the longitudinal length and the transverse length. Each specimen is tested with a flexural fatigue tester (Type MTT, made by Toyo Seiki Co.) and a load of 1.5 g, for the number (H) of strokes until breakage. As a control, a plain weave fabric having the same construction and cover factors is prepared from a fiber comprising the same aromatic polyester but containing neither Tg-decreasing agent nor the solid fine powder, and is tested for the stroke number (H_0) under the same conditions.

The degree of flexural fatigue is expressed by H/H_0 .

(4) Average particle diameter of solid fine powder

A centrifugal particle size tester (Type CAPA-5000, made by Horiba Ltd.) is used and the average value is calculated from the obtained centrifugal sedimentation curve.

Besides, test fabrics were dyed under the following conditions.

Composition of cationic dyeing bath	
Cathilon Brill Red 4GH (made by Hodogaya Chemical Co.)	2% owf
Sodium sulfate	3 g/l
Acetic acid	1% owf
Sodium acetate	0.5% owf
ETA	0.1 g/l
Bath ratio:	1:50
Bath temperature:	120 °C
Dyeing time:	40 min

Example 1

A polyester having an intrinsic viscosity $[\eta]$ of 0.63 dl/g was obtained by transesterification of dimethyl terephthalate containing 2.5 mol% of sodium 5-sulfoisophthalate (hereinafter referred to as "SIP") and ethylene glycol, followed by polycondensation in the usual manner. At a point during the latter period of the polycondensation, there were added 3% by weight of a Tg-decreasing agent represented by formula (X) and 0.3% by weight of a silica powder having an average particle diameter of 30 μm and a refractive index of 1.55.

The polyester chips obtained were melt spun in the usual manner and at a spinneret temperature of 305 °C and a take-up speed of 1,000 m/min, to give an as-spun fiber of 252 deniers/24 filaments. The as-spun fiber thus obtained was, after being preheated on a hot roll at a temperature of 80 °C, drawn in a drawing ratio of 3.4 and then heat set on a hot plate at 150 °C, to give a drawn fiber of 75 deniers/24 filaments. The properties of the polyester chips and the drawn fiber and the decrease in Tg are shown in Table 1.

The drawn fiber was twisted to 250 turns/m and, after being sized, woven into a 1/1 plain weave having cover factors of $K_1 = 18.5$ and $K_2 = 13$. The fabric was scoured and preset in the usual manner. It was dyed under the above-described conditions, subjected to reduction and washing and heat set through a pin tenter. The thus dyed fabric exhibited bright red color and the cover factors of the fabric showed no change before and after the dyeing. The dyed fabric was tested for degree of flexural fatigue and the result is shown in Table 1. Fiber specimens were taken from the dyed fabric by disintegrating it and tested for tensile strength. The result is shown in Table 1.

Comparative Example 1

The spinning was conducted following the same procedure as in Example 1 except that the Tg-decreasing agent and the silica were not added to the polyester, to obtain an as-spun fiber. The obtained as-spun fiber was preheated, drawn in a ratio of 2.5 and heat treated, to give a drawn fiber of 100 deniers/24 filaments. Drawing operation was not stable and many fluffs and uneven drawing occurred, so that the obtained drawn fiber had a large dispersion in its properties. The fiber was woven into a plain weave, which was dyed in the same manner. The cover factors of the fabric before and after dyeing showed no change. The properties are shown in Table 1.

Comparative Example 2

The spinning was conducted following the same procedure as in Example 1 except that the Tg-decreasing agent was not added to the polyester, to obtain an as-spun fiber. The obtained as-spun fiber was preheated, drawn in a ratio of 2.5 and heat treated, to give a drawn fiber of 100 deniers/24 filaments. The obtained drawn fiber was woven into a plain weave, which was dyed in the same manner. The cover factors of the fabric before and after dyeing showed no change. The properties are shown in Table 1.

Comparative Example 3

The spinning and drawing were conducted following the same procedures as in Example 1 except that instead of the polyester that having an intrinsic viscosity $[\eta]$ of 0.55 dl/g was used, to obtain a drawn fiber of 75 deniers/24 filaments. The obtained drawn fiber was woven into a plain weave, which was dyed in the same manner. The cover factors of the fabric before and after dyeing showed no change. The properties are shown in Table 1.

Comparative Example 4

The spinning and drawing were conducted following the same procedures as in Example 1 except that the silica powder was not added to the polyester, to obtain a drawn fiber of 75 deniers/24 filaments. While the spinning was proceeded stably, many fluffs generated during drawing. The obtained drawn fiber was woven into a plain weave, which was dyed in the same manner. The cover factors of the fabric before and after dyeing showed no change. The properties are shown in Table 1.

Example 2 and Comparative Example 5

A polyester having an intrinsic viscosity $[\eta]$ of 0.67 dl/g (Example 2) or 0.73 dl/g (Comparative Example 5) was obtained by transesterification of dimethyl terephthalate containing 1.7 mol% of SIP and ethylene glycol, followed by polycondensation in the usual manner. At a point during the latter period of the polycondensation, there were added 2% by weight of a Tg-decreasing agent represented by formula (X) and 0.3% by weight of a silica powder having an average particle diameter of 30 μ m and a refractive index of 1.55.

The polyester chips obtained were melt spun in the usual manner and at a spinneret temperature of 300 °C and a take-up speed of 1,000 m/min, to give an as-spun fiber of 263 deniers/72 filaments. The as-spun fibers thus obtained were, after being preheated on a hot roll at a temperature of 80 °C, drawn in a drawing ratio of 3.5 and then heat set on a hot plate at 150 °C, to give drawn fibers of 75 deniers/72 filaments. The properties of the polyester chips and the drawn fibers and the decrease in Tg's are shown in Table 1.

While stable spinning and drawing were performed in Example 2, the spinning stability in Comparative Example 5 was not very good due to elevation of pack pressure affected by the thickening effect of the polyester.

The drawn fibers were woven into plain weave fabrics in the same manner as in Example 1 and the fabrics were then dyed in the same manner. The cover factors of the fabrics showed no change before and after the dyeing. The properties are shown in Table 1.

Example 3

A polyester having an intrinsic viscosity $[\eta]$ of 0.65 dl/g was obtained in the same manner as in Example 2 except that a Tg-decreasing agent represented by formula (XI) was used. The polyester chips obtained were melt spun in the usual manner and at a spinneret temperature of 300 °C and a take-up speed of 1,000 m/min, to give an as-spun fiber of 263 deniers/72 filaments. The as-spun fiber thus obtained was, after being preheated on a hot roll at a temperature of 80 °C, drawn in a drawing ratio of 3.5 and then heat set on a hot plate at 150 °C, to give a drawn fiber of 75 deniers/72 filaments. The properties of the polyester chips and the drawn fiber and the decrease in Tg are shown in Table 1.

The drawn fiber was woven into a plain weave fabric in the same manner as in Example 2 and the fabric was then dyed in the same manner. The cover factors of the fabric showed no change before and after the dyeing. The properties are shown in Table 1.

Comparative Example 6

The spinning was conducted following the procedure of Example 3 except that the silica powder was not added to the polyester, obtain an as-spun fiber. The obtained as-spun fiber was drawn in a ratio of 3.5 to give a drawn fiber of 75 deniers/72 filaments. The spinning was stable, but many fluffs occurred during drawing. The drawn fiber was woven into a plain weave, which was dyed in the same manner. The cover factors of the fabric before and after dyeing showed no change. The properties are shown in Table 1.

Comparative Example 7

The spinning was conducted following the procedure of Example 2 except that the silica powder was not added to the polyester, to obtain an as-spun fiber. The obtained as-spun fiber was preheated, drawn in a ratio of 2.8 and heat treated, to give a drawn fiber of 95 deniers/72 filaments. The drawing operation was unstable and many fluffs and uneven drawing occurred, so that the obtained drawn fiber had a large dispersion in properties. The drawn fiber was woven into a plain weave, which was dyed in the same manner. The cover factors of the fabric before and after dyeing showed no change. The properties are shown in Table 1.

Comparative Example 8

Transesterification was conducted in the usual manner with dimethyl terephthalate (DMT) and ethylene glycol. Thereafter, 1.7 mol% based on the mole of DMT of 5-tetrabutylphosphonium sulfoisophthalate, 0.05 mol% on the same basis of tetra-n-butylammonium chloride as a stabilizer and 0.027 mol% on the same basis of antimony trioxide as a polycondensation catalyst were added, to start polymerization. The pressure in the reaction zone was reduced from 760 mmHg to 1 mmHg over 1 hour and, at the same time, the temperature was elevated from 230 to 280 °C over 1 hour, to obtain a polyester having an intrinsic viscosity of $[\eta] = 0.67$ dl/g.

The polyester thus obtained was dried and then melt spun in the usual manner and at a spinneret temperature of 305 °C and a take-up speed of 1,000 m/min, to give an as-spun fiber of 252 deniers/72 filaments. The as-spun fiber thus obtained was, after being preheated on a hot roll at a temperature of 85 °C, drawn in a drawing ratio of 3.3 and then heat set at 145 °C, to give a drawn fiber of 75 deniers/72 filaments. The drawn fiber obtained showed, upon measurement by the above-described method, a strength of 4.5 g/d.

The drawn fiber was woven into a fabric, which was then scoured, preset and dyed. The dyed fabric was disintegrated to give fiber specimens, which were tested for strength. The result showed a decrease down to 3.3 g/d.

From the results shown in Table 1 and obtained in Examples 1 through 3 the following fact will be understood. Where the amounts of a Tg-decreasing agent and the silica are within the range specified in the present invention and the intrinsic viscosity $[\eta]$ satisfies the above condition ①, the resulting drawn fibers have a high strength of at least 4.3 g/d. On the other hand, the results of Comparative Examples 1 through 3 and Comparative Example 7 show that drawn fibers of an aromatic polyester having sulfonic acid salt groups but containing no Tg-decreasing agent have a low strength of less than 4 g/d and fabrics obtained therefrom have low resistance to flexural fatigue.

The results of Comparative Example 5 shows that an aromatic polyester fiber having a high intrinsic viscosity $[\eta]$ of 0.73, which does not satisfy the above condition ①, has, even when it contains a Tg-decreasing agent, a low strength and many fluffs.

Comparison of Example 1 with Comparative Example 4 and that of Example 2 with Comparative Example 6 show that fibers comprising an aromatic polyester having sulfonic acid salt group and containing a Tg-decreasing agent with no silica powder have low strength.

In Comparative Example 8, although the drawn fiber had a high strength of 4.5 g/d, the strength of the fiber after the fabric made therefrom had been dyed decreased to a low value of 3.3 g/d.

Obviously, numerous modifications and variations of the present invention are possible in light of the above teachings. It is therefore to be understood that within the scope of the appended claims, the invention may be practiced otherwise than as specifically described herein.

Table 1

	Polyester chip						Drawn fiber			Woven fabric		
	Content of SIP ¹⁾ (mol%)	Content of Tg-decreasing agent (wt%)	Content of fine powder (wt%)	[η] (dl/g)	De-crease in Tg (°C)	[η] (dl/g)	Strength (g/denier)	Elonga-tion (%)	Single-filament fineness (deniers)	Degree of flex-ural fatigue, H/H ₀		Fiber strength (g/denier)
										Longitu-dinal	Trans-verse	
Example 1	2.5	3	0.3	0.63	10	0.61	4.5	30	3	1.8	1.6	4.1
Comparative Example 1	2.5	-	-	0.65	-	0.61	3.3	30	4	1.0	1.0	3.0
Comparative Example 2	2.5	-	0.3	0.63	-	0.61	3.5	30	4	1.1	1.1	3.2
Comparative Example 3	2.5	-	0.3	0.55	-	0.53	3.9	30	3	1.1	1.1	3.5
Comparative Example 4	2.5	3	-	0.63	10	0.61	3.5	30	3	1.5	1.2	3.2
Example 2	1.7	2	0.3	0.67	6	0.65	5.0	28	1	1.7	1.5	4.0
Comparative Example 5	1.7	2	0.3	0.73	6	0.71	4.0	28	1	1.0	1.0	3.2
Example 3	1.7	2	0.3	0.65	5	0.63	4.8	28	1	3.0	2.6	3.8
Comparative Example 6	1.7	2	-	0.65	5	0.63	4.6	28	1	1.3	1.2	3.6
Comparative Example 7	1.7	-	-	0.67	-	0.65	3.9	29	1	1.0	1.0	3.1
Comparative Example 8	1.7 ²⁾	-	-	0.67	-	0.64	4.5	28	1	1.3	1.2	3.3

1) Sodium 5-dimethylsulfoisophthalate

2) 5-tetrabutylphosphonium sulfoisophthalate

Claims

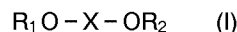
1. A cationically dyeable polyester fiber comprising an aromatic polyester having sulfonic acid salt groups, said polyester fiber containing 0.1 to 10% by weight of a glass transition point-decreasing agent and

0.1 to 1% by weight of a solid fine powder, the intrinsic viscosity $[\eta]$ of said polyester fiber and the content (A% by weight) of said glass transition temperature-decreasing agent in said polyester fiber satisfying the following condition ①

$$0.01 \times A + 0.54 \leq [\eta] \leq 0.01 \times A + 0.68 \quad \text{①}$$

wherein $0.1 \leq A \leq 10$.

2. A cationically dyeable polyester fiber according to Claim 1, wherein said glass transition point-decreasing agent is a compound represented by the following general formula (I)



wherein X represents an aromatic group and R_1 and R_2 each represents an alkyl or arylalkyl group having 6 to 18 carbon atoms.

3. A cationically dyeable polyester fiber according to Claim 1, wherein said aromatic polyester comprises an acid component principally comprising terephthalic acid and a diol component selected from alkylene glycols having 2 to 4 carbon atoms.

4. A cationically dyeable polyester fiber according to Claim 1, wherein said aromatic polyester comprises 1.2 to 30 mol% based on the total moles of the dicarboxylic acid that constitute said aromatic polyester of a copolymerization component of an acid component having a sulfonic acid salt group.

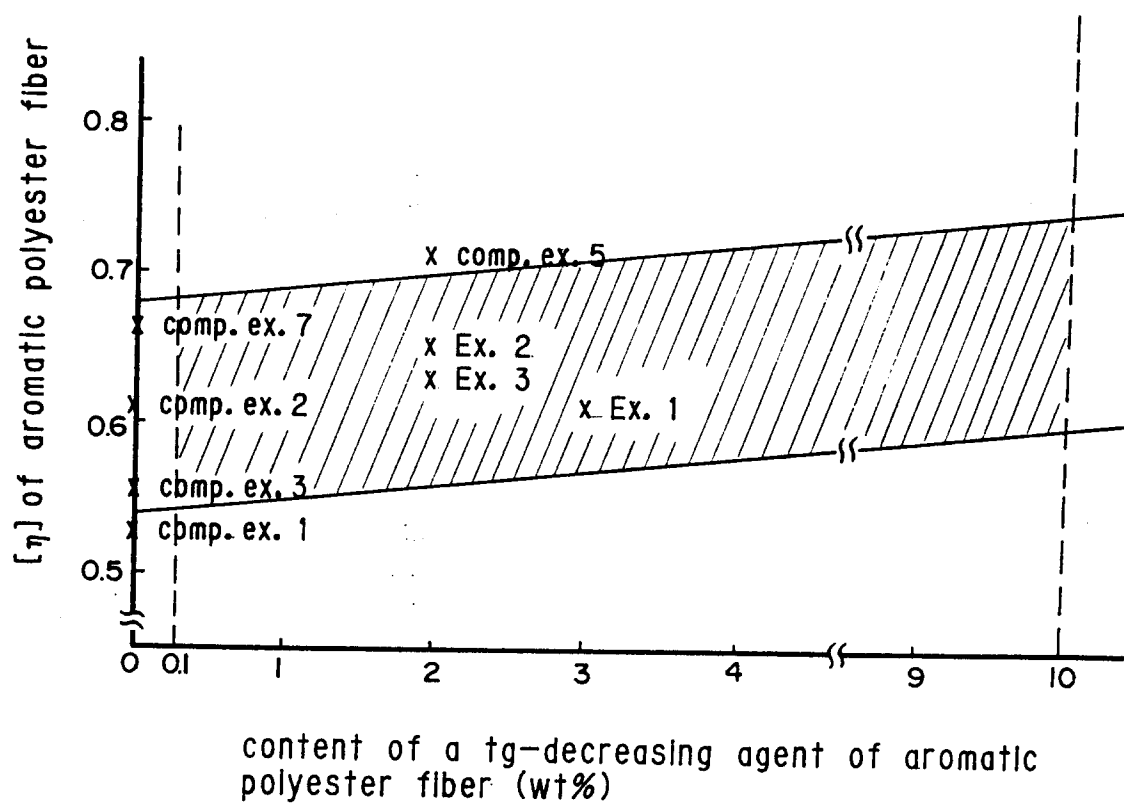
5. A cationically dyeable polyester fiber according to Claim 1, wherein said sulfonic acid salt groups are from sodium 5-sulfoisophthalate.

6. A cationically dyeable polyester fiber according to Claim 1, wherein said solid fine powder has an average particle diameter of 15 to 70 μ .

7. A cationically dyeable polyester fiber according to Claim 1, further having a strength of at least 4.3 g/d and an elongation of at least 26%.

8. A cationically dyeable polyester fiber according to Claim 1, further having a single fiber fineness of not more than 1.5 deniers.

Fig 1





European Patent
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EUROPEAN SEARCH REPORT

Application Number

EP 92 11 9384

DOCUMENTS CONSIDERED TO BE RELEVANT			
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl.5)
P, Y	EP-A-0 491 947 (KAO CORPORATION) * the whole document *	1-8	D01F6/84 D01F6/92 D01F1/10
Y	& WO-A-9 102 111	1-8	
D	& JP-A-3 223 382 (...) ---		
Y	EP-A-0 099 698 (TORAY INDUSTRIES, INC.) * the whole document *	1-8	
P, Y	EP-A-0 503 664 (KAO CORPORATION) * the whole document *	1-8	
Y	US-A-2 819 173 (KARL DITHMAR) * the whole document *	1-8	
A	PATENT ABSTRACTS OF JAPAN vol. 12, no. 297 (C-519)12 August 1988 & JP-A-63 066 322 (KURARAY CO., LTD.) 25 March 1988 * abstract *	1-8	
A	CHEMICAL ABSTRACTS, vol. 104, no. 8, 24 February 1986, Columbus, Ohio, US; abstract no. 52020s, MASUMI GOTO ET AL. 'Mechanically spun polyester yarns' page 61 ;column 2 ; * abstract * & JP-A-60 209 032 (TOYOBO CO., LTD.) -----	1-8	TECHNICAL FIELDS SEARCHED (Int. Cl.5) D01F
The present search report has been drawn up for all claims			
Place of search THE HAGUE		Date of completion of the search 22 FEBRUARY 1993	Examiner TARRIDA TORRELL J.B.
CATEGORY OF CITED DOCUMENTS X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons & : member of the same patent family, corresponding document			