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(71) Applicant:
Asahi Kasei Kogyo Kabushiki Kaisha
Osaka-shi, Osaka 530-8205 (JP)

(72) Inventors:
- **KATO, Jinichiro**
Nobeoka-shi, Miyazaki 882 (JP)
- **TANABE, Tadashi**
Nobeoka-shi, Miyazaki 882 (JP)

(74) Representative:
von Kreisler, Alek, Dipl.-Chem. et al
Patentanwälte,
von Kreisler-Selting-Werner,
Bahnhofsvorplatz 1 (Deichmannhaus)
50667 Köln (DE)

(54) POLYESTER FIBERS AND PRODUCT OF DYEING OF FABRIC MADE OF FIBER MIXTURE CONTAINING THE SAME

(57) Disclosed are an improved polyester fiber capable of attaining a heavy shade dyeing with a fast color by dyeing with a disperse dye at a temperature of 95°C or less and a piece dyed fabric of a polyester fiber composite containing therein a fiber selected from a group consisting of a cellulosic fiber, wool fiber, silk, acetate fiber, polyamide fiber, stretch fiber.

The improved polyester fiber having a peak temperature of the loss tangent ranging from 90 to 108°C and composed of a copolymerized polyethylene terephthalate comprising: from 1.5% to 4.5% by weight of polyethylene glycol having a molecular weight ranging from 500 to 4000; and from 9% to 6% by weight of adipic acid in a ratio satisfying 1.3 ≦ % by weight of adipic acid/% by weight of ethylene glycol ≦ 6.

The polyester fiber has a capability of being dyed at a temperature of 95°C or less with a disperse dye, and the dyeing exhibits a superb color fastness to dry-cleaning, color fastness to light and thermal properties. Especially, the polyester fiber exhibits a balanced exhaustion of a dye combination in practical dye combination dyeing, and black dye dyeing can be carried out at 95°C. Accordingly, in the above-mentioned fabric of fiber composite containing non-polyester fiber, a piece dyed fabric, with vivid color, of a polyester fiber composite in which any specific property of a mixed non-polyester fiber can be provided by a very productive means.

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Description

Field of the Invention

This invention relates to a polyester fiber dyeable with a disperse dye at a temperature of 95°C or less, and especially to a polyester fiber capable of effecting an even and ample dye absorptions of various dyes from a dye mixture making up a dye combination whereby the dyeing of the fiber can produce a superb color development with a dye combination (especially, a black dye combination) as good as the color development intended before dyeing.

This invention also relates to a productive polyester fiber able to provide a superb color fastness to dry-cleaning, color fastness to light and superb thermal and mechanical properties with high productivity.

This invention relates also to a piece dyed fabric of a fiber composite having excellent color development, color fastness and hand, in which the fiber composite is composed of the above-mentioned polyester fiber in combination with at least one fiber selected from a group consisting of cellulosic fiber, wool fiber, silk, stretch fiber, polyamide fiber and acetate fiber. The polyester fiber of the invention as well as the fabric of a fiber composite thereof affords useful textile materials for lining cloth, women's outer garments and women's underwears (foundation garments and lingerie).

Background of the Invention

In recent years the serviceability required of textile fibers for clothes use has become diversified. It has become difficult to use a single material to satisfy the emergent requirement. In this state of affairs, use of fiber composite textile materials has become very important. In the case of polyester fiber, a variety of fiber materials including, for example, cellulosic fiber, wool, silk, stretch fiber, polyamide fiber, acetate fiber and the like are combined with a polyester fiber to make up a composite fiber material. However, various problems are encountered in producing a fabric from a composite fiber because every component fiber making up a composite fiber has its own specific physical properties. The problems caused by differences in dyeablity are extremely crucial.

For example, there is a serious problem in dyeing a fabric composed of polyester fiber combined with cellulosic fiber. For dyeing polyester fiber, disperse dyes are generally used, whereas for dying cellulose fiber, direct dyes or reactive dyes are used. In recent years, use of reactive dyes has been dominant to obtain an improved color fastness of dyeings. When a fabric of a fiber composite composed of polyester and cellulosic fibers, a 2 bath-2 step dyeing system in which the uses separate dyeing baths, is generally employed. This is because the dying temperature for dying a conventional polyester fiber with disperse dye is 110°C or greater, generally around 130°C. If dyeing of a fabric of the fiber composite is carried out at a temperature of from 110 to 130°C under a one bath-one step dyeing system, reactive dyes would be deteriorated to fail to develop the color similar to polyester fiber. When dying is carried out at a temperature below 95°C so as to prevent reactive dyes from decomposition, deep dyeing of polyester fiber cannot be obtained because the polyester fiber is impaired in its capability of being dyed. It is for this reason that there is employed a dyeing process in which dyeing is carried out solely for dyeing polyester fiber with a disperse dye before dyeing of cellulosic fiber is carried out at a temperature of 95°C or less. If a single bath dyeing system, in which dyeing is carried out in one dye bath containing 2 kinds of dyes at a temperature of 95°C or less were possible, the dyeing system will be useful in view of reduction of dyeing cost and expediency of dying work.

As discussed above, a fabric composed of a fiber composite in which a known polyester fiber is incorporated is difficult to dye by means of a one bath one step dyeing process because the dyeing temperatures for the respective fibers are far apart.

There are also serious problems in dyeing fabric of a fiber composite in which a polyester fiber is combined with wool fiber or silk. In order to reduce the cost by increasing the weight, dimensional stability and mechanical strength of the fabric made of only wool fiber and silk, it is considered advantageous that a fabric is made of silk or wool fiber in combination with a polyester fiber. Dyeing a conventional polyester fiber, however, needs the use of a dyeing temperature of 110°C or more. At such temperature, wool fiber as well as silk will be embrittled and cannot be dyed into the same shade with that of the polyester fiber and, in addition to this, their inherent tenacity and handling touch will be very much deteriorated. It is for the above reason that the fabric made of the above fiber composite could not be dyed. For dyeing the fabric of the fiber composite referred to above, the only conventionally practiced method is; a knitted or woven union fabric is first prepared by making use of polyester fiber dyed in the form of yarn or fiber, and thereafter wool fiber or silk is dyed with acid dyes. However, this dying system has a serious drawback in productivity and cost of dyeing because the method requires extremely complicated works to obtain diverse color effect. If dyeing is carried out under the normal pressure paying a due consideration to thermal stability of wool fiber or silk, polyester fibers cannot be dyed into a heavy or deep shade because conventional polyester fibers lacks dyeability.

Besides, there is also a serious problem in dyeing of a fabric of a conventional polyester fiber in combination with a stretch fiber. Polyamide fiber has a soft hand and is dyeable under normal pressure. Hence, polyamide fiber is conventionally mixed with a stretch fiber as typified by polyurethane fiber and has been applied to making knitted sport

wears, women's underwear including foundation garments and lingerie, swimwear, socks and the like. However, polyamide fiber has disadvantageous features: the fiber is susceptible to gases and/or light which readily give rise to yellowing of the fiber; when the fiber is used as covering yarn, slippage of the covering yarn is liable to occur due to its poor constraining force; the fiber lacks dimensional stability and a shape retention property or the like. To remove these disadvantages, a fiber mixture of a polyester fiber with a stretch fiber is used.

The problems pertaining to polyamide fiber as explained above can be removed by the use of polyester fiber in place of polyamide. However, this gives rise to a new problem pertaining to dyeability. Namely, the use of high dyeing temperatures of a polyester fiber becomes a problem. In order to carry out heavy shade dyeing of a fabric of a conventional polyester fiber combined with a polyurethane fiber, the dyeing temperature is required to be at least 100°C or more. At this temperature, there are encountered serious problems in that the polyurethane fiber suffers from thermal degradation which gives rise to a sharp deterioration in tensile strength and yellowing of the fiber and the like. If dyeing were carried out under the normal pressure in order to protect polyurethane from thermal degradation of polyurethane, heavy shade dyeing of the polyester fiber cannot be attained because a conventional polyester fiber lacks dyeability.

There is also a serious problem in dyeing of a fabric of a fiber mixture of polyester and polyamide fibers. Although polyamide fiber claims such advantages as the ease of dyeing with acid dyes under normal pressure and a high tensile strength, the fiber poses such problems as tendency toward yellowing and a lack of heat-setting property and dimensional stability. For removal of the problem, the use of polyester fiber mixed in combination has been considered. Problems encountered with the use of a fiber mixture containing a conventional polyester fiber for which dyeing is done at high temperatures, are for example, the polyamide fiber is stained and the resultant dyeings exhibits a stiff touch. It is the matter of course that the normal pressure dyeing is employed in consideration of thermal stability of polyamide fiber, and in consequence, the polyester fiber cannot be dyed in a heavy shade because a conventional polyester fiber lacks dyeability.

In the dyeing of fabric composed of a mixture of polyester and acetate fibers, serious problems are encountered. Acetate fiber is a modified cellulosic fiber in which the hydroxyl groups are acetylated. Although the basis of its molecular structure is similar to cellulose, acetate fiber is not dyeable with direct and reactive dyes. Conventionally, dyeing of acetate fiber is done with disperse dyes under the normal pressure dyeing system. In the case high pressure dyeing at a temperature at above 100°C should be applied, thermaldegradation due to the hydrolysis of acetyl group tends to occur causing the fiber to lose its transparency (become milky white colored). It is known that this phenomenon tends to occur especially with diacetate fiber. To obtain a fabric of acetate fiber composite having an improved mechanical strength, dimensional stability with economical production, the combined uses of polyester fiber have been considered. With a conventional polyester fiber of which dyeing temperature is high, there are, however, problems in that acetate fiber becomes milky white colored (devertified turbid color) and stiff. With dyeing carried out under normal pressure in which acetate fiber is thermally stable, heavy shade dyeing of polyester fiber cannot be attained because a conventional polyester fiber lacks dyeability.

There have been problems in the known polyester fibers having an improved dyeability with disperse dyes. Easily dyeable polyesters making use of copolyamide as raw material are known in the art.

However, any of the known polyester fibers having an improved dyeability with a disperse dye can exhaust only a certain specific dye from a dye combination in which various dyes are compounded, even though the fiber exhibits good dyeing properties with a specific single dye. As a result, the respective dye compounded in a dye combination differs in rate of dye exhaustion. For this reason, there occurs a problem in that the consequent color of a dyeing comes to differ in effect from the color which has been intended on the basis of a mixing ratio of respective dye components before dyeing. This problem is especially remarkable with a black dye. The known polyester fibers improved in dyeing properties have a problem in that black color development of dyeing differs from that intended before dyeing.

There are known easily dyeable polyester fibers in which copolyesters are used as a raw material. Of these known fibers, there are known fibers composed of a polymer produced by copolymerizing with a polyoxyethylene glycol or adipic acid. In Japanese Unexamined Patent Publications (Kokai) No. 3-40880, No. 3-174076, No. 4-41732 and No. 4-41738, there are disclosed polyethylene terephthalate fibers composed of a polymer in which only a polyoxyethylene glycol is copolymerized.

The known fibers are polyester fibers composed of a polymer, in which 6 to 10% by weight of a polyoxyethylene glycol is copolymerized, and the fibers can be dyed at 98°C. However, it has been found, according to the inventor's observations, that the fiber has a low color fastness to light and a low dry-cleaning fastness because of the use of a high copolymerizing ratio of polyoxyethylene. Although the fiber can be dyed at 98°C, it cannot be dyed at 95°C. It may be said that there exists a serious difference between the dyeability at 98°C and the dyeability at 95°C in the sense of a commercial dyeing practice. This is because the dyeing at boiling temperature in a normal pressure dyeing vessel often fluctuates between 95 and 98°C. Accordingly, even though a fiber having a capability of being dyed at 98°C can be dyed into a heavy shade, the fiber has serious shortcomings in that faulty or uneven dyeing tends to take place from batch to batch due to the fluctuations of 3°C in the dyeing temperature. On the other hand, a fiber which can be dyed at 95°C has no such problems. There is another problem in that prominent color dyeing can not be developed in light shade dye-

ing because the gray fiber is deteriorated in whiteness with an increase in a copolymerizing ratio of polyethylene glycol in the polymer. In the polymerization of such copolymerizing composition, bumping tends to occur under an extremely high vacuum; in a serious case, it can cause the vacuum pipe lines to be clogged with a bumping polymer so that the pipe lines may need to be cleaned. Further, it has been found that a polyester fiber composed of a polymer copolymerized with 5% by weight or greater of polyethylene glycol lacks spinnabiliy. That is, it has been observed that an extruded yarn still in the state of melted polymer immediately below the face of the spinneret is liable to bend before being cooled, therefore the yarn tends to break or to become fluffy during melt-spinning. Further, it has been found that production of yarn constituting of fine denier filaments of which a single filament is one denier or less is extremely difficult due to a lack of spinnability. Fine denier yarns are an unavoidably necessary product item with a view to provide the essential descriptions for applications such as linings or women's garments where a soft hand is very much required. With use of such a copolymerizing composition, the field of application will be very much limited due to the difficulty of preparing fine denier yarns. It is, therefore, to be noted that the known easily dyeable fiber yarns referred to above are still problematic in dyeability, color fastness to light, whiteness, polymerizing properties and spinnability.

A polyester fiber from a polymer obtained by copolymerizing adipic acid and polyethylene glycol is also known (for examples, Japanese Unexamined Patent Publications (Kokai) No. 63-85111 and No. 63-235536). In Japanese Unexamined Patent Publication No. 63-85111, there is disclosed a polyester fiber composed of a copolyester in which 0.5 to 10% by weight of polyether unit, and 0.5 to 10% by weight of dicarboxylic acid unit are copolymerized. In an example described in the publication, a polyester fiber composed of a polymer in which 4% by weight of polyethylene glycol and 4% by weight of adipic acid is copolymerized, and there is further described that the fiber achieve a high rate of dye exhaustion with an anthraquine dye. In fact, the dye is easily adsorbed on the fiber since its molecular weight is relatively small (molecular weight: 349) among the conventional disperse dyes. Hence, the attainment of the high rate of dye exhaustion with this dye does not mean that the fiber can exhibit a high rate of dye exhaustion with all dyes. Further, since the fiber contains a smaller percentage by weight of adipic acid/by weight of polyethylene glycol, the fiber is difficult to dye with a dye combination of various dyes and has a tendency to have a poor color yield especially when the fiber is dyed with a black dye. In addition to the above, the fiber exhibits a relatively high peak temperature (herein after called Tmax (°C) of the loss tangent values determined by measuring dynamic viscoelasticity of a fiber. The parameter represents a relative magnitude of density of molecule in the amorphous portion of fiber. Hence, the fiber does not exhibit ease of dyeing with a dye combination of various component dyes, but it exhibits an ease of dyeing only with a limited single dye. For the reason set forth above, the fiber has still serious problems in conventional use.

In Japanese Unexamined Patent Publication (Kokai) No. 63-235536, there is described a polyethylene terephthalate fiber in which 6% by weight of polyethylene glycol and 5.1% by weight of adipic acid are polymerized. The fiber can have as high a rate of dye exhaustion with a single dye as the aforesaid fiber. However, the fiber still has problems in color fastness to dry-cleaning of the dyed fiber, whiteness, polymerizing properties, dyeability with a combination of various dyes, spinnability because of its high polymerizing ratio of polyethylene glycol and a high Tmax (°C) value.

Other known methods making use of copolymerized polyester fibers are: a copolymerized polyester fiber from a copolymer obtained by copolymerizing glycols such as tetra-methylene glycol and 1, 4-cyclohexane diol(Japanese Unexamined Patent Publication No. 58-120815) and a polyester fiber obtained by high speed spinning of a copolymer copolymerized with 2, 2-bis [4-(2-hydroxy) phenyl] propane (Japanese Unexamined Patent Publication (Kokai) No. 59-199814). These polyester fibers obtained by those method mentioned above cannot be dyed at 95°C, though they are improved in dyeability.

There is also known an easily dyeable polyester fiber obtained by using a high speed spinning technology, in which the polyester is obtained by copolymerizing a dicarboxylic acid of aliphatic group containing 8 carbon atom or more in the polymer (Japanese Unexamined Patent Publication No. 5-98512). Although it is true that the fiber exhibits an enhanced dyeablity, the fiber has a problem in that an extreme deterioration of color fastness to dry-cleaning results and that the fabric therefrom deteriorates in the fabric handling because its thermal stress development is lowered and fiber exhibits a low stress development.

There is known a process for modifying polyester fiber by high speed spinning from a polyester in which a metal sulfonate compound is copolymerized (Japanese Examined Publication No. 60-10126). Since the fiber exhibits a low mechanical strength, there is a drawback in that a fabric made from the resulting fiber has a low bursting strength. For this reason, the fiber is not suited to the objective of the invention. Since the polyester copolymerized with metal sulfonate compound tends to contain a large quantity of infusible denatured metal sulfonate compound, the polymer becomes impossible to be melt-spun in a prolonged spinning because spinning head pack is clogged.

There is a conventional process for enhancing the dyeabiltiy of polyester fiber in which 5-sodiumsulfonateisophthalic acid and adipic acid are copolymerized (Japanese Unexamined Patent Publication No. 51-133529, No. 55-158325 and No. 61-239015). The objectives of the invention cannot be attained by making use of a fiber from such polymers because the fiber lacks the dyeability at 95°C.

As attempts to modify dyeing methods or finishing methods, the carrier dyeing technique is known. However, the carrier dyeing technique itself has drawbacks in that it makes the waste dye bath treatment and dyeing work difficult

because high boiling point organic substances such as phenol derivatives, chlorinated aromatic compound or biphenyl must be used.

There are several known method in which polyester fibers obtained by means of high speed spinning are treated under wet-heat in the presence of water to increase the dyeability of the fibers (Japanese Unexamined Patent Publication (Kokai) No. 58-136825 and Japanese Examined Patent Publication (Kokoku) No. 63-73650). The wet-heat treatment, though it improves the dyeability, has a disadvantage in that uneven dyeing may result. Besides, the method has a drawback in that the advantageous productivity inherent in a high speed spinning is impaired because the fibers are additionally heat-treated after spinning.

As discussed above, there is no commercially available polyester fiber which has not only a capability of being dyed at 95°C or less with disperse dyes, but also has an excellent dyeability with a dye combination of various dyes. Even with known polyesters improved in rate of dye exhaustion to a certain degree, they still have problems in color fastness to dry-cleaning, thermal properties, polymerization properties and the spinnability.

Disclosure of the Invention

The object of the invention is to provide a polyester fiber capable of being dyed at 95°C or less with disperse dyes and having an excellent dyeability with a combination of various dyes and; further, a polyester having good properties pertaining to color fastness to dry-cleaning and to light, thermal and mechanical properties and; a polyester fibers capable of being converted by wet-processing including dyeing and finishing at an excellent productivity and; a piece dyed polyester fabric of polyester fiber composite exhibiting an superb color yield (color development), color fastness and hand in which the polyester fiber composite is composed of at least one fiber selected from the group consisting of cellulosic fiber, wool fiber, silk, stretch fiber and polyamide fiber.

It has been found by the present inventors that the objectives of the invention can be achieved by a fiber having a specific range of Tmax determined by measurement of the dynamic viscoelasticity which indicates the molecular density of the amorphous portion in a fiber, and making use of a polyethylene terephthalate copolymerized with a polyethylene glycol and dicarboxylic acid from which adipic acid is specifically selected in a an extremely limited range of copolymerizing ratios. The invention is embodied on the basis of this knowledge.

The first feature of the invention is a polyester fiber characterized in that a copolymerized polyethylene terephthalate comprises 1.5 to 4.5% by weight of a polyethylene glycol having a molecular weight of 500 to 4000 and 9 to 6% by weight of adipic acid in a ratio satisfying 1.3 ≦ % by weight of adipic acid/% by weight of polyethylene glycol ≦ 6, and that the fiber satisfies a peak temperature of the loss tangent of 90 to 100°C.

The second feature of the invention is a piece dyed fabric of a composite fiber containing the above-mentioned polyester fiber combined with cellulosic fiber.

The third feature of the invention is a piece dyed fabric of a composite fiber containing the above-mentioned fiber and wool or silk.

The fourth feature of the invention is a piece dyed fabric of a fiber composite containing the above-mentioned polyester fiber and a stretch fiber.

The fifth feature of the invention is a piece dyed fabric of a fiber composite containing the above-mentioned polyester fiber and polyamide fiber.

The sixth feature of the invention is a piece dyed fabric characterized in that a piece dyed fabric is composed of the above-mentioned polyester fiber and acetate fiber.

A polymer constituting the polyester fiber in accordance with the invention is a copolymerized polyester comprising a polyethylene terephthalate copolymerized with 1.5 to 4.5% by weight of a polyethylene glycol having a molecular weight of 500 to 4000 and 9 to 6% by weight of adipic acid in a ratio satisfying 1.3 ≦ % by weight of adipic acid/% by weight of polyethylene glycol % ≦ 6. In order to obtain an adequate dyeability at 95°C and color fastness, the two copolymerized components, namely a polyethylene glycol and adipic acid are necessary. A polyethylene terephthalate copolymerized with 1.5 to 4.5% by weight of only polyethylene glycol having a molecular weight of 500 to 4000, or with 6 to 9% by weight of only adipic acid cannot exhibit an adequate dyeability at 95°C.

The dyeability herein is defined as dye the property of a fiber capable of developing a heavy shade dyeing in which a dye is absorbed adequately on the fiber; stated most simply, a dyeability can be estimated by a relative magnitude of rate of dye exhaustion from a dye bath containing the dye as exemplified in the Examples hereinafter.

A polyethylene glycol used as a copolymerizing component is an extremely effective component for improving the dyeability. When the molecular weight of the polyethylene glycol is less than 500, a content of the polyethylene glycol unit in the resultant polymer cannot be kept at a prescribed content because a fairly large amount of low molecular weight polyethylene glycol contained in a conventionally available raw polyethylene glycol fractionally removed in the course of polymerization under a high vacuum. In consequence, the resulting fiber as an ultimate product, tends to fluctuate in properties such as tensile properties, dyeability, thermal properties or the like. On the other hand, when the mean molecular weight exceeds 4000, the dyeability and color fastness to light deteriorate due to an increase in high

molecular weight polyethylene glycol which remains unpolymerized in the resultant polymer.

Adipic acid used as the copolymerizing component can do much towards producing adequate disorder in the amorphous structure of the fiber. It is needless to say that an aliphatic dicarboxylic acid component other than adipic acid (number of carbon atoms 6) can be effective as a comonomer which can produce disorder in the amorphous portion for improving the dyeability. However, with an aliphatic dicarboxylic acid having 5 carbon atoms, thermal stability of the polymer deteriorates and the whiteness is lowered. It may be said in this connection that the deterioration in thermal stability becomes greater in proportion to the mole number of the ethylene group adjacent to carboxyl group. On the other hand, when an aliphatic dicaboxylic acid having 7 or more carbon atoms is used, deterioration in color fastness, especially color fastness to dry-cleaning is remarkable because a disorder of the amorphous portion grows too much. It was, however, found that the occurrence of above-mentioned problems diminishes particularly with a single use of adipic acid. As discussed above, adipic acid is an extremely excellent copolymerizing component is effective in a limited range narrow range. It may be postulated that the whole length of 4 methylene groups contained in adipic acid is substantially equal to the length of the benzene nucleus of telephthalic acid. Hence, although the amorphous structure is disordered by the presence of bendable group in the polyester fiber used in the invention, the degree of the disorder is brought to a minimum whereby the polyester fiber used in the invention becomes a modified polyester fiber of which the properties such as the thermal property are most closely alike those of a polyethylene terephthalate fiber.

The amount of polyethylene glycol required to ensure a sufficient dyeability with a disperse dye ranges from 1.5 to 4.5% by weight. When the amount of polyethylene glycol is less than 1.5% by weight, the dyeability become impaired. When the amount of polyethylene glycol exceeds 4.5% by weight, color fastness to light becomes deteriorated, and, in addition, the polymer is difficult to polymeric due to the occurrence of discoloration of the polymer in the course of polymerization and the occurrence of a bumping or bubbling phenomenon in the polymerization vessel under a high vacuum. Still further problems in spinnability with this polymer are the difficulty of spinning fine denier yarn and the occurrences of yarn breakage and the high rate of forming fluffy yarn. The most preferred amount in which the dyeability, color fastness, operability of polymerization and spinnability are all compatible with each other, ranges from 2 to 4% by weight.

On the other hand, the most suitable amount of adipic acid is 9 to 6% by weight, though it varies depending on the amount of polyethylene glycol. Where the amount of adipic acid is less than 6% by weight, the dyeability at 95°C is not satisfactory. However, where the amount exceeds 9% by weight, thermal resistance deteriorates and the resultant fabric has a stiff handling.

A practically advantageous polymer cannot be attained unless selection is made of a polymerization ratio by which the resulting polymer can exhibit a balance between the dyeability and color fastness concurrently with a good polymerizability and spinnability. To attain an increased dyeability, it is preferred to copolymerize a polyethylene glycol as much as possible, however, it is to be noted that use of a large amount of ethylene glycol causes a marked deterioration in color fastness to dry-cleaning and dyeability, polymerizability and spinnability. The deterioration in color fastness to light, color fastness to dry cleaning, dyeability, polymerizability and spinnability and reduced polymerizability can be successfully suppressed by carrying out a copolymerization in which a suitable amount of adipic acid, instead of using a large quantity of a polyethylene glycol is used.

The ratio of adipic acid to a polyethylene glycol is extremely critical condition for attaining an enhanced dyeability not only with a single dye, but also with a dye combination in which various disperse dyes are compounded. Such a copolymerization composition needs to satisfy a ratio of $1.3 \leqq$ % by weight of adipic acid/% by weight of polyethylene glycol $\leqq 6$ (hereinafter called R value). With an R value less than 1.3, color fastness to light as well as color fastness to dry-cleaning deteriorates due to an increased ratio of polyethylene glycol in the case where a polyethylene glycol and adipic acid are copolymerized in amounts sufficient to attain a dyeing property under normal pressure dyeing.

Besides, in case where dyeing is carried out with a dye combination of compounded various dyes therein, color development of a dyeing tends to differ from that intended prior to dyeing; especially poor dyeing may result when dyeing is done with a black color dye combination.

Still further, with an R value exceeding 6, when a polyethylene glycol and adipic acid are copolymerized in amounts enough to attain a dyeing property under normal pressure dyeing, the fiber deteriorates in its thermal resistance and the hand is stiffened during heat-setting stage. Further, even if any ratio of $1.3 \leqq$ R value $\leqq 6$ is satisfied, a fabric article resulting from the fiber composite is liable to stiffen. For obtaining a soft hand touch, selection is made of a value less than 4, especially preferred less than 3.

Further, for attaining an excellent color development on a dyeing when dyeing is done with a dye combination, a preferred value for R is 1.7 to 6, more preferably 1.7 to 4.

In a polyester fiber used in the invention, other copolymerizable components capable of forming a polyester such as another kind of diol and oxycarboxylic acid can be incorporated in an amount of 10% or less, preferably 5% or less by weight. However, it is to be noted that these optionally incorporated components should not deteriorate in color fastness of a dyed fiber.

Further, various additives, for example, a delustering agent, a thermal stabilizer, an antifoaming agent, a flame

retardant, an ultra-violet absorbing agent, an infra-red ray absorbing agent, a nucleating agent and a fluorescent whitening agent can be incorporated by colymerizing or blending if case of needed.

The polymer forming the polyester fiber of the invention can be prepared using a conventional process for polymerization of a polyethylene terephthalate; by copolymerizing by mean of adding, for example, a polyethylene glycol and adipic acid in itself or in the form of its lower alkylester such as monomethyl ester, dimethyl ester, diethyl ester, bis (oxyethyl) ester and the like to the reaction system at any stage before the termination of condensation polymerization. At the time of copolymerization, these copolymerizing components can be added in the component compounds in themselves or in a dissolved or dispersed form in a suitable solvent such as ethylene glycol, after being heat-treated as the case may be.

By way of example, a representative process of polymerization is herein described; an ester exchange reaction between dimethyl terephthalate and dimethyl adipate is carried out in ethylene glycol in the presence of a catalytic amount of manganese acetate, calcium acetate or cobalt acetate at a temperature of 200 to 240°C to ethoxylate the respective terminal groups of telephthalic and adipic acids. In the reaction, the termination is determined by the moment when the theoretical amount of methanol is liberated. This is followed by an addition of a polyethylene glycol and antimony trioxide which is a catalyst for condensation polymerization and the polymerization is carried out at a temperature of 260 to 290°C under a vacuum.

The polyester fiber of the invention must have a peak temperature of the loss tangent of 90 to 108°C which is determined by dynamic viscoelasticity measurement. The reason for this is that a dyeability as intended by the invention can be ensured in this range. Since Tmax corresponds to a density of molecule in the amorphous portion, the smaller the value becomes, the smaller the density of molecule in the amorphous portion widening the gap (interstices) for the easy entry of dye which causes a rate of dye exhaustion to increase.

With Tmax kept within the range, a polyester of the invention not only attains the dyeability at 95°C with a single dye, but also exhibits an excellent dyeability with every diverse disperse dye when dyeing is carried out with a dye combination of various disperse dyes; and a color development as intended prior to dyeing can be attained in consequence. Especially, a black color development becomes extremely superb when dyeing is carried out with a black dye.

Because Tmax value represents the fiber structure, value of Tmax is brought to be different depending not only on spinning conditions such as spinning temperature, spinning speed and draw ratio and but also textile converting processes conditions such as heat-treatment, scouring and alkali reduction processing even when copolymer is composed of the same composition. Especially, since the Tmax value is broadly changed by heat setting temperature, it is important to keep the Tmax range within the above range.

Referring to a general guide line for regulating the heat-setting temperature, for a polymer as specified in the invention, Tmax becomes gradually higher with a heat setting temperature within the range from room temperature to 160°C, however, it lowers remarkably as temperature exceeds about 160°C. Since the proportion of these changes differs from copolymerization composition to composition, Tmax should be determined by considering the relationship between Tmax and the heat-setting temperature. In accordance with the invention, when Tmax become greater than 108°C, improvement in the dyeability is too small to attain an adequate dyeability at 95°C. However, not all low Tmax values are acceptable because of disadvantage to the effect that the ease of entry of dye into the fiber and ease of stripping off of dye occurs concurrently due to the formation of loose amorphous portions. Namely, deterioration in color fastness, especially color fastness to dry-cleaning and color fastness to abrasion in the wet state and color fastness to laundering. In addition, such problems as stiffening the hand and deterioration in dimensional stability occurs when heat-setting is applied. In practice, Tmax ranges from 90 to 110°C, preferably from 95 to 105°C.

The melting point of the polyester fiber of the invention is in a range from 230°C to 245°C. When the melting point is at a temperature of less than 230°C, the polyester fiber suffers from a thermal degradation in conventional processing or daily use as typified by heat-setting or ironing where the fiber is changed in its physical properties and hand. If the melting point exceeds 245°C, the spinnability is deteriorated. Since the polymer forming the polyester of the invention is of a special composition, the spinning temperature for attaining satisfactory spinning needs to be maintained in a range from about 255°C to about 250°C at the spinneret face. When a melting point of a polymer exceeds 245°C, it becomes difficult to maintain this temperature at the spinneret face, because the temperature of extruder for melting the polymer should be raised.

The polyester fiber of the invention can be prepared either by a conventional process in which an undrawn yarn is draw-twisted at a draw ratio ranging from about 2 to 3.5 times at a winding speed of 1,500 m/min., or by a spin-draw process in which spinning stage and draw-twisting are continuously coupled. The fiber may be prepared by making use of a high speed spinning process in which a winding speed of 5,000 m/min or more is used, however this process may not be preferred because the orientation of amorphous portion becomes much lowered (hence, Tmax is much lowered) so that color fastness is much lowered. The spinning conditions are not limitative. Spinning can be carried out using any known condition except that the spinneret face temperature needs to be regulated. That is, it is significant that the spinneret face temperature is in a range from about 255°C to about 270°C. When the temperature is less than 255°C, the spinning at such a low temperature results in the occurrence of slub in the yarn which creates a number of yarn break-

ages. Although spinning is possible at a temperature between from about 270°C to about 300°C, the spinning at such range of temperature, however, encounteres a number of yarn breakages with an increased rate of development of fluffy yarn because of the occurrence of yarn bending.

In the polyester fiber according to the invention, a K/S, which represents a color yield obtained when the fiber is dyed at 95°C is 20 or more. The method for measuring K/S is in accordance with that described in the Examples of the invention. The dye used for estimation of dyeability has a large molecular structure (molecular weight of 518). Accordingly, if an enhanced dyeability is attained with the use of this dye, an enhanced dyeablity can be ensured regardless of the kinds of disperse dye. By an enhanced dyeability is meant a K/S of 20 or more in this case. Accordingly, in a case where dyeing is done at 95°C, it can be considered that a dyeing having a K/S of 20 or more exhibits a color development as good as the polyester fiber that has been dyed at 130°C. Such a color development can generally be attained when a rate of dye exhaustion reaches about 75% or more.

To ensure good color fastness in dyed goods, it is necessary that the rating of color fastness to dry-cleaning for a dyed goods is at grade 3 or higher. In the invention, color fastness to dry-cleaning is evaluated in reference to liquid staining. The method for the evaluation is discussed in Example. The items of color fastness cover a variety of evaluation items including color fastness to water, color fastness to laundry, color fastness to sublimation, color fastness to rubbing and the like. According to a study by the inventors, it has been found that the color fastness of all the various items required in practice including color fastness to water, laundry, sublimation, rubbing and the like excepting color fastness to light for the polyester fiber of the invention can be rated as having ratings satisfying the commercially acceptable level in the case where the color fastness to dry-cleaning is rated at grade 3 or higher. Accordingly, the color fastness to dry-cleaning can be an index representing the entire color fastness properties of the polyester fiber of the invention. Therefore, a dyed article of which color fastness is rated as grade 3 or higher is determined to have a good color fastness properties in practice. For a dyed article enabling use for outer garments, color fastness to light should be rated as being grades 3 to 4, preferably 4 or higher under the dyeing conditions specified in the invention.

The dyed fabric of a fiber composite of the invention is characterized in that a dyed fabric is composed of polyester fiber of the invention and at least one kind of fiber selected from the group consisting of cellulose fiber, wool fiber, silk, stretch fiber, polyamide fiber acetate fiber. The form of the polyester fiber of the invention and mixing method of the fiber of the invention are not specifically limited; namely, any known method can be used. For example, a method for preparation of the fiber composite includes, for example, a woven fabric such as union in which the polyester fiber is used either in warp yarns or in weft yarns, reversible woven fabric, knitted fabric such as tricot and rashel fabric, and further use of doubling of yarn, plaiting and entangling can be employable. Cellulosic fiber used in the invention is not specifically limited, and includes natural fibers such as cotton fibers, linen or ramie, cuprammomium rayon, viscose rayon, polynosic and the like. In a fabric of the fiber composite, the ratio of the polyester fiber component is not limited and a preferred content of the polyester fiber is in a range from 25% to 75% to make the best use of the characteristic hand, hygroscopicity, water absorbency and anti-electricity provided by cellulosic fibers.

Wool fiber and silk used in the invention can be of the fiber as conventionally available. The content of the polyester fiber is not specifically limited in a fabric of the fiber composite, however, a preferred content of the polyester fiber is in a range from 25% to 75% to make the best use of the characteristic nature of wool fibers such as the hand touch, warmness and bulkiness or the characteristics of silk such as the hand and kishimi sound.

The stretch fiber usable in the invention is not specifically limited, and includes dry- or melt-spun polyurethane fiber, polybutylene telephthalate fiber, polyester type elastic fiber which are typified by polytetramethylene glycolether copolymerized polybutylene telephthalate fiber and the like. In a fabric of the fiber composite in which a stretch fiber used, a preferred content of the polyester fiber is in a range from about 60% to about 98%. When a content of the polyester fiber exceeds 70%, it can be used for producing outer garments and casual wear because the stretch properties become suppressed. In contrast, when a content of the polyester is less than 70%, it can be used for producing such end-use articles as women's underwears, foundation garments, swimwear and the like.

The polyamide fiber usable in the invention can be nylon 66 and nylon 6 in which an amide group is substantially contained; and as conventionally available fiber can be used. Content of the polyester fiber is not specifically limited in the fabric of the fiber composite, however, a preferred content of the polyester fiber is in a range from 25% to 75% for making the use of characteristic hand touch peculiar to polyamide.

The acetate fiber usable in the invention can be either diacetate fiber or cellulose triacetate fiber. However, the best use of the advantageous merit of the invention can be made by a fiber composite containing diacetate fiber which lacks thermal stability. Since acetate fiber is dyed with a disperse dye as in the dyeing of polyester fiber, a fiber composite composed of acetate fiber combined with the polyester fiber of the invention can be dyed at a temperature below 95°C to attain a processed article with a good hand touch at a reduced dyeing cost. Although content of the polyester fiber in the composite fiber fabric is not limited, it is preferred that the content of the polyester fiber is preferably in a range from 25% to 70% for making the best use of the hand, brightness of color and luster provided by acetate fiber.

The dyed fabric of the fiber composite of the invention can be incorporated with a fiber other than the fibers not specified in the invention unless the object of the invention is impaired. It is permissible that a small amount of wool

fiber, silk, viscose rayon, cuprammonium rayon, polyamide fiber, polyacryl fiber, acetate fiber or acrylic fiber may be jointly used. In this case, it is possible that an addition of physical properties characteristic of a jointly used fiber can be made.

The dyeing of fabric of the fiber composite can be obtained by carrying out knitting or weaving the fabric followed by a conventional scouring and subsequent dyeing. In case of need, the fabric may be processed by a conventional caustic reduction treatment after scouring and before dyeing. The scouring is carried out at a temperature between 60 and 98°C. When a fiber composite contains stretch fiber as a joint component, it is preferable that scouring is carried out while allowing relaxation to ensure an improvement in elasticity. Dyeing is carried out without use of carrier at a temperature of 95°C or less with a disperse dye for the polyester fiber of the invention; with a direct dye or reactive dye for a cellulosic fiber; with a disperse dye for acetate fiber; with an acid dye for wool fiber, silk or polyamide fiber. Naturally, the most preferred method is the one bath one step dyeing at a temperature of 95°C or less in order to give the advantageous merits full play. Of course, a one-bath two step dyeing system as well as two-bath two step dyeing system may be used. After dyeing is terminated, a conventional soaping or reduction clearing is applied. Especially in a fiber composite containing stretch fiber as the joint component in which the stretch fiber is a polyurethane fiber, it is important for improving color fastness of the fabric that a disperse dye staining the polyurethane fiber should have to be fully removed by application of a reduction cleaning. These methods can be known methods. If there is required a heat-setting treatment before or after dyeing, the heat-setting treatment is done at a temperature between 140 to 190°C, preferably between 160 and 180°C under dry setting conditions.

With making use of the specific polyester fiber, the dyeing of a fabric of fiber composite can be obtained by carrying out dyeing at a temperature of 95°C or less. For this reason, the troubles with deterioration in strength of wool fiber and silk, the yellowing of polyamide fiber, the thermal degradation of polyurethane fiber and the devetrification (delustering) of acetate fiber are eliminated. Besides, dyeing can be carried out at a temperature of 95°C or less where no degradation of the dyes takes place, and, as a result, a bright color dyeing can be attained.

Best Mode for Carrying Out of the Invention

The invention is explained further in detail by the following examples. In the Examples, the measured values were determined according to the following method.

(1) Peak Temperature (Tmax) of the Dynamic Loss Tangent

Using Rheovibron available from Orientec Corporation, the loss tangent (tan δ) and dynamic elasticity were measured at respective temperatures at a measuring frequency of 110 Hz in a dried air while temperature was raised at a rate of 5°C/min. From the results, a loss tangent-temperature curve was drawn to find a Tmax which is the peak temperature Tmax (°C) of a loss tangent on the curve. The measurement was performed at a rate of at a rate of 5°C/min. and at a measuring frequency of 110 Hz.

(2) Melting Point

Using DSC available from Seiko Instruments Inc., the melting point was measured in a nitrogen gas flowing at a flow rate of 100 ml/min., while the temperature was raised at a rate of 20°C/min. The peak value of fusing was determined as the melting point.

(3) Measurement of Rate of Exhaustion, Degree of Color Yield (K/S) (Evaluation of dyeability)

A one feeder single circular knitted fabric was used as a specimen. The knitted fabric was scoured at 70°C in a lukewarm water containing Scourol 400 at 2 g/l for 20 minutes, and dried in a tumbling dryer, and subsequently heat-set at 180°C for 30 seconds on a pin tenter.

The rate of dye exhaustion was estimated by finding the dye exhaustion in a dye bath which had been maintained for 1 hour after the bath temperature was raised from 40°C to 95°C. The dye used was Kayalon Polyester Blue 3RSF (a product available from Nippon Kayaku Co., Ltd.: molecular weight, 518; Color Index Blue 257). The dyeing was carried out in a dye bath containing 6% owf of the dye with a liquor to goods ratio 50:1. The dye bath contained Nicca Sunsalt 7000 (a dispersing agent from Nicca Chemical Co., Ltd.) at 0.5 g/l and acetic acid 0.25 g/l and sodium acetate at 1 g/l were added to adjust the pH to 5.

Dye exhaustion was determined by the following equation in which A and a represent the absorbencies of the initial dye bath and the dye bath at the termination of dyeing obtained by spectrophotometric measurement. The absorbencies were measured at 580 nm where the dye exhibits the maximum light absorbency.

$$\text{Rate of Exhaustion} = (A - a)/A \times 100\ (\%)$$

Degree of Color Depth represents a degree of depth of shade of a dyeing, and was evaluated by using K/S value. The value was determined from the Kubelka-Munk equation below by measuring the spectrophotometric reflectance R of a dyed specimen.

$$K/S = (1 - R)^2/2R$$

The greater the value, the greater the degree of depth of shade becomes; namely, color is well developed. R was measured at 580 nm at which the dye exhibits the maximum absorbency.

(4) Black lightness value L

A one feeder single circular knitted fabric of polyester fiber was used for a specimen. Three pieces of the same knitted specimen were laid one on top of another. A measurement was performed on the three thickness using a color computer (SM-4) available from Suga Test Instruments Co., Ltd.

(5) Color Fastness

Color fastness to dry-cleaning, light and laundering were evaluated respectively according to JIS-L-0860, JIS-L-0842 and JIS-L-0844. For evaluation with polyester fiber itself, use is made of a 500 mg piece of a dyed one feeder single circular knitted fabric prepared according to the method (3).

(6) Elastic Recovery

A test piece having a size of 16 cm length by 2.5 cm width was fixedly secured between chucks at a distance of 10 cm on a tensile tester. An elongation-recovery curve was drawn up to an elongation of 80%, from which a residual elongation (L') was read. The value was determined according to the following formula in which L is defined as 80%.

$$\text{Elastic recovery } (L-L') \times 100/L$$

Example 1

20 parts of dimethyl terephthalate, 714.7 parts of ethylene glycol, 1.52 parts of dimethyl adipate (hereinafter referred to DMA) and 0.01 parts of tetra hydrate of manganese acetate as an ester exchange catalyst were fed and the reaction mixture was gradually heated up by raising the temperature from 150°C to 240°C to carry out an ester exchange reaction while driving off methanol over 3 hours. Then, to the reactant were added 0.44 parts of polyethylene glycol having a molecular weight of 1000 (hereinafter referred to as PEG1000), 0.44 parts of trimethyl phosphate as a stabilizer, 0.1 parts of titanium dioxide as a delustering agent and 0.016 parts of antimony trioxide as a condensation polymerization catalyst, and prepolymerization was then carried out over 50 minutes. The reactant was further placed under a gradually increasing vacuum until the reaction was carried out at 275°C under a pressure of 0.5 Torr for two hours and 40 minutes to obtain a modified polyester having a viscosity of η sp/c = 0.80 in the form of chip. The composition of the obtained polymer contained a PEG 1000 component of 2% by weight and DMA of 7% by weight.

After the prepared chips was dried at 130°C in a nitrogen atmosphere flowing at a rate of 100 l/min. for 20 hours, the dried chips were spun at a spinning temperature of 270°C at a spinning speed of 1500 m/min using a spinneret having 36 holes each having a circular cross section to prepare an undrawn yarn. The resultant undrawn yarn was then draw-twisted at a draw ratio of 2.8 times over a hot roll at 80°C and hot plate at 160°C at a winding speed of 800 m/min to produce a drawn yarn of 75 denier/36 filaments; the yarn had a strength of 5.1 g/d, an elongation of 31% and a Tmax of 103°C.

The dyeability of the polyester of the invention can be evaluated by comparing it with the dyeability exhibited by a polyethylene terephthalate fiber (Tmax: 136°C) spun by a commonly accepted spinning (so called conventional process in which the undrawn yarn which is once wound is drawn) which is dyed at 130°C for 60 minutes with a blue disperse dye. In this case, it is preferable that comparison is made in terms of K/S value because a straight comparison of color yield of the dyeings can be performed. In case where dyeing was carried out in a dye bath with a liquor to goods ratio 50:1 containing 6% owf of Kayalon Polyester Blue 3RSF (Nippon Kayaku Co., Ltd.), a polyethylene terephthalate obtained by a conventional process dyed at 130°C for 60 minutes exhibits a K/S of 21.8. On the other hand, the polyester fiber of the present example dyed at 95°C for 60 minutes exhibited a K/S of 21.8. These results show that the dyeability with dyeing at 95°C for 60 minutes of a polyester fiber of the invention is equal to the dyeability with dyeing at

130°C for 60 minutes of a polyethylene terephthalate fiber obtained by a conventional process.

Regarding color fastness to dry-cleaning of the dyed one feeder single circular knitted fabric, the dyeing does not show color degradation and liquid staining was evaluated as grade 4. Moreover, the dyeing had a good color fastness of the dye in various items of evaluations; color fastness to light (grade 4), color fastness to dry and wet rubbing (grade 5), color fastness to water (grade 5), color fastness to laundering (grade 5) and color fastness to sublimation (grade 4).

Examples 2 - 4

Experiment on polymerization and spinning were performed with varied copolymerizing compositions using the same method according to Example 1. The results are summarized in Table 1. Every case exhibited good dyeability, color fastness and physical properties.

Example 5

Example 1 was repeated with varied molecular weights of polyethylene glycols; 2000, 3000 and 4000. The resultant polyester had the same properties as those in Example 1. The range of K/S is from 21.5 to 21. 7 showing no significant difference from those in Example 1. In every case, both color fastness to dry-cleaning and color fastness to light were rated as grade 4.

Comparative Examples 1 - 8

Polyester fibers composed of various copolymerized compositions as given in Table 1 were prepared and subjected to various items of evaluations. Any copolymerized composition out of the scope of the invention exhibits cannot be acceptable for practical use because these fibers lack dyeability, color fastness or other properties. In Comparative Example 2 and 5, there were often encountered bumping phenomena in the polymerization under an extremely high vacuum, and the content in the vessel moved into the vacuum pipe line. In the event where bumping was violent, the apparatus needed to be disassembled for cleaning. Yellowing also occurred in the obtained polymer.

Further, in comparative Examples 2, 5 and 6, breakages and fluffing of yarn were occasionally encountered owing to the occurrences of bending of yarn beneath the spinneret during spinning. The phenomena could not be rectified even though spinning conditions were varied.

Table 1

Examples	Compositions (% by weight)		R value	tenacity	Elongation	melting point	Tmax	Rate of dye exhaustion	K/S	Color fastness to light	Color fastness to dry-cleaning
	PEG1000	DMA		(g/d)	(%)	(°C)	(°C)	(°C)		(grade)	(grade)
1	2	7	3.5	5.1	31	237	103	75	21.8	4	4
2	2	8	4.0	4.6	34	239	104	76	21.1	4	4
3	3	7	2.3	5.0	35	238	103	78	21.8	4	4
4	4	7	1.8	5.0	40	238	100	87	21.8	3 - 4	3
Comparative Examples											
1	0	10	-	4.6	33	240	108	54	17.5	5	4
2	10	0	0.0	4.3	46	249	94	90	21.7	2	1
3	5	0	-	4.6	33	249	111	61	18.1	3	4
4	4	4	1.0	4.8	35	243	109	65	19.5	3 - 4	4
5	6.5	0	0.0	4.6	35	253	109	63	19.7	3	3
6	6	5	0.8	4.3	33	243	98	86	21.8	2	2 - 3
7	4.5	3.5	0.8	4.5	33	246	111	61	18.2	2 - 3	4
8	3.5	4.5	1.3	4.6	35	243	110	64	19.0	3 - 4	4

Example 6

The one feeder single circular knitted fabric in Examples 3 and 4 were dyed with 5% owf of Sumikalon Black S-BF

(a disperse dye, available from Sumitomo Chemical Co. Ltd.) in a dye bath at a pH of 6 with a liquor to fiber ratio of 50:1 in the presence of Nicca Sunsalt 7000, a dyeing auxiliary (available from Nicca Chemical Co., Ltd.). The dyeing was carried out at 95°C. L values (brightness) of the resultant dyeings were 15.5 and 15.3 respectively presenting good black dyeing in adequate heavy shade.

For comparison, the one feeder single circular knitted fabrics of Comparative Examples 4, 7 and 8 were dyed with the same conditions and L values of the dyeings were 18.8, 18.3 and 19.5 respectively and all of the dyeings looked fade (straw colored). A black dye is generally a dye combination (dye compounded mixture) in which three kinds of dyes have different respective maximum peak absorbencies at 400 nm, 500 nm and 580 nm. In the case of Example 7, the spectroscopic absorbency of the dye bath at the termination of dyeing (exhausted dye bath) was measured and it was found that almost no peak absorbencies remained at the three spectrums. On the contrary, in the cases of the Comparative Examples, as a result of calorimetric measurement on the exhausted dye baths after termination of dyeing, it was found in every Comparative Examples that 90% or more of absorbencies of the exhausted dye baths at 580 nm were extinguished, and that the absorbencies of the exhausted dye baths at 400 nm and 500 nm were, however, left unexhausted about 30% or more of the absorbency of the initial dye bath. Accordingly, the polyester fiber of the present example, when being dyed with a dye combination, exhibits a good rate of dye exhaustion with respective component dyes. However, the fiber out of the scope of the invention does not show a fluctuation of high rate of dye exhaustion with respective component dyes.

Example 7

A plain weave woven fabric was prepared using a polyester fiber of 75d/72f produced in the same manner as in Example 1 for warp yarn and cuprammonium rayon of 75d/44f for filling yarn. The fabric was scoured by a conventional method and then mercerized. The mercerizing treatment was carried out by dipping the fabric in a caustic soda solution containing 75% of sodium hydroxide at room temperature. After neutralization, washing with water and subsequently heat-setting at 180°C for 30 seconds, one bath one step dyeing with a disperse dye and a reactive dye was carried out in the absence of a carrier. As the disperse dye, Kayalon Polyester Blue BRSF (available from Nippon Kayaku Co., Ltd.) was used; as reactive dye, Drimarene Blue X SGN (available from Sandoz K.K.). A dye bath was prepared by adding the dyes in a solution adjusted at pH 11 containing Disper TL, a dispersing agent (available from Meisei Chemical Works, Ltd.) at 1 g/l, sodium sulfate at 50 g/l and of sodium carbonate at 15 g/l. Dyeing was carried out at 95°C with a bath containing 2 owf of the dye in 50:1 liquor ratio for 1 hr. After dyeing, the dyed fabric was soaped at 80°C in a bath containing Granup P (available from Sanyo Chemical Industries Ltd.) at a concentration of 1 g/l with a bath ratio of 50:1 for 10 minutes. The dyed fabric was finally finished in a conventional manner.

The resultant dyed fabric was evenly dyed and had a nice hand; the K/S value was 21.5; and the color fastness to dry-cleaning and color fastness to light were both rated as grade 4.

Example 8

Using the polyester fiber as prepared in Examples 3 and 4, one-bath one step dyeing was carried out in the same manner as in Example 7. The resultant dyed fabric was evenly dyed, and had a nice hand; the K/S values were 21.7 and 22.0. Regarding color fastness, color fastness to dry-cleaning for either of the dyeings was evaluated as grade 3 - 4 and color fastness to light for the both of the dyeings was rated as grade 4.

Comparative Example 9

Using the polyester fiber in Comparative Example 4, Example 7 was repeated. The resultant dyeing was not an even dyeing. This is because the polyester fiber has a low developability of color due to its low dyeability.

Comparative Example 10

Using the polyester fiber in Example 6, Example 7 was repeated. The resultant dyeing was evenly dyed. However, color fastness to dry-cleaning as well as color fastness to light was evaluated as grade 2 - 3 which is not acceptable in commercial practice. Since the fiber is a fine denier fiber of 75d/72f, a lot of fluff occurred in the resultant dyeing.

Example 9

A polyethylene terephthalate fiber copolymerized with 4% by weight of PEG 1000 and 7% by weight of DMA was twisted under heat to produce a torque type bulked textured yarn. The conditions for texturizing were: heating plate temperature, 195°C; number of twists at twisting, 3400 times/m; feeding ratio, -0.2%. The obtained textured yarn was dou-

bled to form a yarn having a denier of 150, and was knitted with a wool yarn having a yarn count of 48 Nm to produce a reversible double-faced union fabric with polyester on top and wool on the back. The mixed ratio of the polyester fiber in the union fabric was 48% by weight. The knitting conditions are: 20 gauge; diameter of needle bed, 20 inches. The knitted fabric was, then, dyed according to a conventional method. Regarding the dyes, Dianix Black BGFS (200% grade; available from Dystar Japan Co. Ltd.) as the disperse dye and Kayalon Black BGL (Nippon Kayaku Co. Ltd.) as the acid dye were used.

One bath one step dyeing was carried out at 95°C applying 7% owf of the respective dyes in a slightly acidic bath in the presence of a dispersing agent. After dyeing, the dyeing was soaped at 70°C in a slightly alkaline bath containing soda ash at 1 g/l and nonionic detergent at 0.5 g/l for 20 minutes. The resulting dyeing had a L value (brightness) of 11.8, a superb value. The lower the brightness, the heavier the shade of black dyeing is obtained. The dyeing was rated as grade 4 in both color fastness to dry-cleaning and color fastness to light.

Examples 10 - 12

Varying the copolymerizing compositions in the same manner as in Example 9, experiments on polymerization and spinning were performed. The results are summarized in Table 2. In all of the cases, remarkable dyeability and color fastness were attained.

Comparative Examples 11 - 16

Polyester fibers having the copolymerizing composition given in Table 2 were subjected to various items of evaluations. The fibers in which copolymerizing compositions were outside the invention had problems with either dyeability or their color fastness.

Table 2

Examples	Compositions (% by weight)		R value	L value	Color fastness to light (grade)	Color fastness to dry-cleaning (grade)
	PEG1000	DMA				
9	4	7	1.8	11.8	3 - 4	4
10	2	8	4.0	11.9	4	4
11	3	7	2.3	11.9	4	4
12	2	7	3.5	11.8	4	4
Comparative Examples						
11	0	10	-	13.0	5	4
12	10	0	0.0	11.7	3	1
13	5	0	0.0	13.1	3	4
14	4	4	1.0	12.3	3	4
15	6.5	0	0.0	12.4	3	3
16	6	5	0.8	11.8	2 - 3	2

Examples 13

A polyethylene terephthalate fiber (75d/72f) composed of a polyethylene terephthalate copolymerized with 4% by weight of PEG 1000 and 7% by weight of DMA was twisted at 300 T/m and then was sized by means of an applicator roll sizer to prepare warp yarn. A plain weave fabric was made using silk yarn (21d/2f) as filling yarns.

For dyeing the fabric, Dianix Black BGFS (200% grade; available from Dystar Japan Co. Ltd.) as the disperse dye and Kayalon Black BGL (Nippon Kayaku Co., Ltd.) as the acid dye were used.

One bath one step dyeing was carried out at 95°C applying 7% owf of the respective dyes in a slightly acidic bath in the presence of a dispersing agent. After dyeing, the dyeing was soaped at 70°C in a slightly alkaline bath containing soda ash at 1 g/l and nonionic detergent at 0.5 g/l for 20 minutes. The resulting dyeing had a L value (brightness) of

11.1, a superb value. The lower the brightness, the heavier the shade of black dyeing is obtained. The dyeing was rated as grade 4 in both color fastness to dry-cleaning and color fastness to light. The dyed goods had the superb hand peculiar to silk.

Comparative Example 17

With the exception that a polyethylene terephthalate fiber was used, Example 13 was repeated. An L value of 15.3 was obtained. The polyester fiber was dyed only to a pale shade. When dyeing was done at a dyeing temperature of 130°C, an L value of 11.5 was reached; the dyed goods lost the hand peculiar to silk and had a stiff hand.

Example 14

A knitted fabric was produced using a polyurethane stretch fiber having a denier of 210, Roica (a product of Asahi Chemical Industries Co., Ltd.) and a polyethylene terephthalate fiber (50d/36f) having a trilobal shaped cross section comprising 4% by weight of PEG 1000 and 7% by weight of DMA as copolymerizing components. The knitting particulars were: a knitting gauge of 28GG and a loop length of 1080 mm/480 courses for the normal pressure dyeable yarn and 112 mm/480 courses for the stretch fiber. The mixed ratio of the polyester fiber was selected at 75.5%.

The grey knitted fabric was scoured in a relaxed state at 90°C for 2 minutes and thereafter dried at 160°C for 1 minute. Dyeing was carried out at 95°C for 60 minutes with 8% owf of Dianix Black FS (available from Dyestars Japan Co., Ltd.) in the presence of Nicca Sunsalt 1200, a dyeing assistant, at a concentration of 0.5 g/l in a dye bath at a pH of 6 adjusted by acetic acid with a bath ratio of 30:1.

The resulting dyeing had a value (black brightness value) of 12.3, and adequate color development. The dyeing had an elastic recovery of 95.3%, and color fastness to laundering was rated as grade 5 and color fastness to light was rated as grade 4. The resulting dyed goods had a soft, pliable and resilient hand.

Example 15 - 17

Example 14 was repeated by varying the copolymerizing compositions. In every case, the resultant dyed goods exhibited a superb color development, good color fastness and a good elastic recovery and had a soft, pliable and resilient hand.

Example 18

Example 14 was repeated using polyethylene glycols differing in their molecular weights; 2000, 3000 and 4000. The properties of the resultant dyed goods were almost the same with those of Example 14. In every case, black lightness value of the resultant dyeing fell within a range from 12.8 to 12.4 showing sufficient development of color. The dyeing had an elastic recovery of 95%, and color fastness to laundering was rated as grade 5 and color fastness to light was rated as grade 4. The resultant dyed goods had a soft, pliable and resilient hand.

Comparative Example 18

For comparison, a warp knitted fabric was made from Roica and a polyethylene terephthalate fiber obtained by a conventional spinning process and dyeing was carried out at 95°C for 60 minutes. The resultant dyeing had an L value of 18.3 showing an inadequate black color development. On the other hand, in another dyeing in which dyeing was carried at 130°C for 60 minutes, an L value of 12.4 was attained. However, it was found that the magnitude of bursting strength of the dyed fabric fell to as low as 2/3 of that of the grey fabric and that the elastic recovery fell to as low as 65% of that of the grey fabric.

Comparative Example 19 - 21

Varying copolymerized composition, various dyed goods were prepared in the same manner as Example 14 (Table 3).

It can be seen that the diabilities and color fastness were insufficient in the cases where the copolymerized compositions were outside the scope of the invention. The resultant dyed goods of Comparative Example 20 had a stiff hand.

Table 3

Examples	Compositions (% by weight)		R value	L value	Elastic recovery (%)	Color fastness to laundry (grade)	Color fastness to light (grade)
	PEG1000	DMA					
14	4	7	1.8	12.3	95.3	5	4
15	2	9	4.5	12.1	96.2	5	4 - 5
16	3	7	2.3	12.4	95.5	5	4 - 5
17	2	6	3.0	12.6	94.2	5	4 - 5
Comparative Examples							
18	6.5	0	0.0	13.5	94.8	3	2 - 3
19	0	5	-	16.3	95.3	4	5
20	0	10	-	12.5	90.3	2 - 3	5
21	10	0	0.0	12.1	95.6	3	2

Example 19

A polyethylene terephthalate fiber (75d/72f) composed of a polyethylene terephthalate copolymerized with 4% by weight of PEG 1000 and 7% by weight of DMA was twisted at 300 T/m, and then was sized by means of an applicator roll sizer to prepare warp yarn. A plain weave fabric was made using the yarn as warp and a folded yarn consisting of 3 ends of nylon 66 yarn having a denier of 50d/96f as filling yarn.

For dyeing the fabric, Kayalon Polyester Blue 3RSF as the disperse dye and Nylosan Blue GFL (available from Sandoz K.K.) as the acid dye were used.

One bath one step dyeing was carried out at 95°C applying 5% owf of the respective dyes in a slightly acidic bath in the presence of a dispersing agent. After dyeing, the dyeing was soaped at 70°C in a slightly alkaline bath containing soda ash at 1 g/l and nonionic detergent at 0.5 g/l for 20 minutes. The resultant dyeing had a K/S value of 21.0, a superb value. The ratings of both color fastness to dry-cleaning and color fastness to light were grade 4. The dyeing had a soft hand touch and a superb match of color with that of nylon 66 was obtained.

Example 20

Except that a fiber (75d/72f) composed of a polyethylene terephthlate copolymerized with 2% by weight of PEG 1000 and 8% by weight of polyethylene terephthalate fiber, Example 19 was repeated. The obtained dyeing has a K/S of 21.0 and the rating of color fastness to dry-cleaning was grade 4 and that of color fastness to light was grade 5. Hand of the dyeing was superb. It exhibited a soft hand touch and superb match of color with nylon 66 was attained.

Example 21

A polyethylene terephthalate fiber (75d/72f) composed of a polyethylene terephthalate copolymerized with 4% by weight of PEG 1000 and 7% by weight of DMA was twisted at 300 T/m and then was sized by means of an applicator roll sizer. Using the yarn as warp yarn and diacetate yarn (100d), a plain weave fabric was produced.

For dyes, use was made of Kayalon Polyester Blue 3RSF as the disperse dye and Kayalon Fast Blue RD200 (available from NIPPON KAYAKU Co., Ltd.) as another disperse dye.

One bath one step dyeing was carried out at 95°C applying 5% owf of the respective dyes in a slightly acidic bath in the presence of a dispersing agent. After dyeing, the dyeing was soaped at 70°C in a slightly alkaline bath containing soda ash at 1 g/l and nonionic detergent at 0.5 g/l for 20 minutes. The resultant dyeing had a K/S value of 21.1, a superb value. The ratings of both color fastness to dry-cleaning and color fastness to light for the dyeing were grade 4. The dyeing had a soft hand touch and was superb in vividness of color.

Example 22

Except that a fiber (75d/72f) composed of a polyethylene terephthalate copolymerized with 2% by weight of PEG 1000 and 8% by weight of DMA, Example 21 was repeated. The obtained dyeing has a K/S of 20.2 and the rating of color fastness to dry-cleaning was grade 4 and that of color fastness to light was grade 5. The hand of the dyed goods was superb. The ratings of color fastness to dry-cleaning and color fastness to light of the dyed goods were grade 4. The dyeing had a soft hand and was superb in vividness of color.

Comparative Example 23

Except that use was made of polyethylene terephthalate fiber and that dyeing was carried out at 130°C, Example 21 was repeated. Although the obtained dyed goods has a K/S of 21.5, the dyeing presented a dull color development and its hand lacked softness.

Industrial Applicability

The polyester fiber of the invention can be dyed with a disperse dye at a temperature of 95°C or less in a depth of shade required in the commercial dyeing practice. The thermal properties and color fastness of the dyed goods can be matched for the level obtainable by a dyeing of the conventional polyester fiber, for example, in color fastness to dry-cleaning and color fastness to light.

The polyester fiber of the invention can exhaust individual dye components composing a dye combination evenly without effecting differential dye exhaustion in dyeing with a disperse dye combination in which various disperse dyes are compounded thereby enabling a fabric to be dyed in a color and depth of shade identical with those normally intended by a dye combination commercially available, for example, a black dye for black dyeing.

The composite fiber fabric composed of the polyester fiber of the invention mixed with cellulosic fiber, wool fiber, silk, polyamide fiber or stretch fiber in combination can be piece-dyed substantially at a normal practical dyeing temperature for any of the mixed fibers whereby all the fiber composing the fabric can be dyed. Accordingly, the piece dyed goods of polyester fiber composite in which properties of fiber having a poor thermal stability remain undamaged can be provided by an exceedingly productive means.

Claims

1. An polyester fiber composed of a copolymerized polyethylene terephthalate characterized in that the polyester fiber satisfies a peak temperature of the loss tangent ranging from 90 to 108°C and the copolymerized polyethylene terephthalate comprises: 1.5 to 4.5% by weight of polyethylene glycol having a mean molecular weight of from 500 to 4000; and 9 to 6% by weight of adipic acid in a ratio 1.3 ≦ % by weight of adipic acid/% by weight of ethylene glycol ≦ 6.

2. A polyester fiber according to claim 1 characterized in that the polyethylene glycol is copolymerized in an amount of from 2 to 4% by weight.

3. A polyester fiber according to claims 1 or 2, characterized in that 1.7 ≦ % by weight of adipic acid/% by weight of polyethylene glycol ≦ 4.

4. A polyester fiber according to any one of claims 1 through 3, characterized in that the peak temperature of the loss tangent is in a range from 90 to 105°C.

5. A polyester fiber according to any one of claims 1 through 4, characterized in that the fiber has a melting point ranging from 230 to 245°C.

6. A piece dyed fabric of a fiber composite composed of a mixture of cellulosic fiber and a polyester fiber in which the polyester fiber is a copolymerized polyethylene terephthalate characterized in that the polyester fiber satisfies a peak temperature of the loss tangent ranging from 90 to 108°C and the copolymerized polyethylene terephthalate comprises: 1.5 to 4.5% by weight of polyethylene glycol having a mean molecular weight of from 500 to 4000; and 9 to 6% by weight of adipic acid in a ratio satisfying 1.3 ≦ % by weight of adipic acid/% by weight of ethylene glycol ≦ 6.

7. A piece dyed fabric according to claim 6 in which the polyester fiber is contained in a range from 25 to 75% by

weight.

8. A piece dyed fabric of a fiber composite composed of a mixture of wool fiber or silk and a polyester fiber in which the polyester fiber is a copolymerized polyethylene terephthalate characterized in that the polyester fiber satisfies a peak temperature of the loss tangent ranging from 90 to 108°C and the copolymerized polyethylene terephthalate comprises: 1.5 to 4.5% by weight of polyethylene glycol having a mean molecular weight of from 500 to 4000; and 9 to 6% by weight of adipic acid in a ratio satisfying 1.3 ≦ % by weight of adipic acid/% by weight of ethylene glycol ≦ 6.

9. A piece dyed fabric according to claim 8 in which the polyester fiber is contained in a range from 25 to 75% by weight.

10. A piece dyed fabric of a fiber composite composed of a mixture of a stretch fiber and a polyester fiber in which the polyester fiber is a copolymerized polyethylene terephthalate characterized in that the fiber satisfies a peak temperature of the loss tangent ranging from 90 to 108°C, and the copolymerized polyethylene terephthalate comprises: 1.5 to 4.5% by weight of polyethylene glycol having a mean molecular weight of from 500 to 4000; and 9 to 6% by weight of adipic acid in a ratio satisfying 1.3 ≦ % by weight of adipic acid/% by weight of ethylene glycol ≦ 6.

11. A piece dyed fabric according to claim 10 in which the polyester fiber is contained in a range from 60 to 98% by weight.

12. A piece dyed fabric of a fiber composite composed of a mixture of a polyamide fiber and a polyester fiber in which the polyester fiber is a copolymerized polyethylene terephthalate characterized in that the polyester fiber satisfies a peak temperature of the loss tangent ranging from 90 to 108°C and the copolymerized polyethylene terephthalate comprises: 1.5 to 4.5% by weight of polyethylene glycol having a mean molecular weight of from 500 to 4000; and 9 to 6% by weight of adipic acid in a ratio satisfying 1.3 ≦ % by weight of adipic acid/% by weight of ethylene glycol ≦ 6.

13. A piece dyed fabric according to claim 12 in which the polyester fiber is contained in a range from 25 to 75% by weight.

14. A piece dyed fabric of a fiber composite composed of a mixture of an acetate fiber and a polyester fiber in which the polyester fiber is a copolymerized polyethylene terephthalate characterized in that the polyester fiber satisfies a peak temperature of the loss tangent ranging from 90 to 108°C and the copolymerized polyethylene terephthalate comprises: 1.5 to 4.5% by weight of polyethylene glycol having a mean molecular weight of from 500 to 4000; and 9 to 6% by weight of adipic acid in a ratio satisfying 1.3 ≦ % by weight of adipic acid/% by weight of ethylene glycol ≦ 6.

15. A piece dyed fabric according to claim 14 in which the polyester fiber is contained in a range from 25 to 75% by weight.

INTERNATIONAL SEARCH REPORT

International application No.
PCT/JP96/02160

A. CLASSIFICATION OF SUBJECT MATTER
Int. Cl^6 D01F6/62

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
Int. Cl^6 D01F6/00-6/96

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Jitsuyo Shinan Koho	1926 - 1996
Kokai Jitsuyo Shinan Koho	1971 - 1996
Toroku Jitsuyo Shinan Koho	1994 - 1996

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	JP, 5-98512, A (Toray Industries, Inc.), April 20, 1993 (20. 04. 93)(Family: none)	1 - 5
A		6 - 15
A	JP, 1-124678, A (Asahi Chemical Industry Co., Ltd.), May 17, 1989 (17. 05. 89)(Family: none)	1 - 15
A	JP, 6-49706, A (Toray Industries, Inc.), February 22, 1994 (22. 02. 94)(Family: none)	1 - 15

☐ Further documents are listed in the continuation of Box C. ☐ See patent family annex.

* Special categories of cited documents:
"A" document defining the general state of the art which is not considered to be of particular relevance
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"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
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"&" document member of the same patent family

Date of the actual completion of the international search October 11, 1996 (11. 10. 96)	Date of mailing of the international search report October 15, 1996 (15. 10. 96)
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