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(54) PROCESSING AGENT FOR POLYESTER FIBER STRUCTURE AND PRODUCTION METHOD FOR POLYESTER FIBER STRUCTURE USING SAME

(57) The present invention provides a terminal blocking processing agent of a polyester-based fiber structure allowing a treated product to efficiently take up a terminal blocking agent, and a method of producing a polyester-based fiber structure having hydrolysis-resistant properties using the terminal blocking processing agent, and is characterized in that terminal blocking treatment is carried out with the terminal blocking processing agent in which a carbodiimide-based terminal blocking agent and a carrier containing alkyl phthalimide and benzoate as essential components are emulsified or dispersed in water or a solvent using a surfactant.

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Description

TECHNICAL FIELD

[0001] The present invention is related to a finishing agent for a polyester-based fiber structure and a method of producing the polyester-based fiber structure using the finishing agent.

BACKGROUND ART

[0002] With the recent rise in environmental awareness, corporate enterprises must fulfill social responsibilities to reduce plastic waste and to cut the amount of carbon dioxide emissions in order to suppress global warming. To solve these problems, it is important to promote use of biodegradable plastics which are expected to be degraded by enzymes and microorganisms, and to promote recycling and reuse thereof.
Polylactic acids have particularly drawn attention as biodegradable plastics made of natural resources. Because lactic acid which is a raw material of the polylactic acid can be obtained from plants such as corn, it can be said that the polylactic acid is an environment-conscious plastic. However, the polylactic acid has a property of being very highly hydrolysable in water at room temperature and high temperature, and can also be degraded even by the water in air, which limits environment and application where it is used.
In a hydrolysis reaction, protons released from the terminal carboxyl group act as a self catalyst and promote break down of esters, which is a common problem not only in the polylactic acid but also polyester-based fibers.
Because polyethylene terephthalate which is general-purpose polyester has a lower hydrolysis rate as compared with polylactic acid, it has sufficient hydrolysis-resistant properties when used for application in general clothing materials. But, because uniforms used in the field of medical treatment, nursing care, food product or the like require washing in a hot water bath at 60 to 90°C or autoclaving treatment at 120°C to 130°C, there is concern on deterioration of the fabric by such treatment. Due to this, in practice, the number of years of the uniforms in the field of medical treatment being used is relatively short and disposable uniforms account for high percentage.
In view of this, by providing technique to improve hydrolysis-resistant properties of the polyester-based fiber, use of the biodegradable plastic including the polylactic acid can be promoted and extension of the durability of the uniforms requiring the washing at high temperatures or autoclaving treatment can be attained, which can solve environmental issues.

[0003] As a means of suppressing the hydrolysis of polyester, Japanese Patent Application Laid-Open Publication No. 2001-261797 and Japanese Patent Application Laid-Open Publication No. 2002-30208 disclose methods of lowering the concentration of terminal carboxyl group by adding a terminal blocking agent. However, these methods have a problem in that, because the terminal blocking agent is added to and kneaded with polymer chips before spinning, the terminal blocking agent causes fuming due to evaporation and decomposition to generate an offensive odor and toxic gas. Because of this, there is also a problem in that the terminal blocking agent must be excessively added. Further, fiber spinning properties become deteriorated and productivity becomes lower as well. As disclosed in Japanese Patent Application Laid-Open Publication No. 2010-189813, use of an inorganic substance as the terminal blocking agent can solve the problem of the poisonous gas at the time of the kneading and spinning. Yet, because it is a terminal blocking treatment in a fiber spinning stage, there is concern that the type of yarns is difficult to be changed and hydrolysis occurs in a process such as a dyeing step or post-processing step.

[0004] As an alternative method for the solution, Japanese Patent Application Laid-Open Publication No. 2009-249450 discloses a method comprising dissolving a terminal blocking agent with a solvent or emulsifying the agent with an emulsifier and bringing the resultant into direct contact with processed material to add terminal blocking onto the surface of the processed material. However, because the terminal blocking agent is present only near the surface of a treated material in this method, the amount of terminal blocking agent added is not adequate and thus the hydrolysis-resistant property against heat and humidity for a long period of time is insufficient.

[0005] As a method of improving the hydrolysis-resistant property against heat and humidity for a long period of time, it is desirable that a terminal blocking agent be added at high concentration to the surface of a structure to which the heat and humidity make a direct contact and the a terminal blocking agent also be added to the inside of the structure at a certain concentration. Japanese Patent Application Laid-Open Publication No. 2009-263840 discloses a method comprising adding a terminal blocking agent near the surface of a structure at a high concentration and adding the terminal blocking agent also to the inside of structure at a concentration equal to or higher than a certain concentration. However, there is a problem in that efficiency in taking up a terminal blocking agent into a treated product is low, resulting in high concentration of the terminal blocking agent used and high costs involved in it.

[0006] Japanese Patent Application Laid-Open Publication No. 2001-98459 discloses a method comprising, when a polyester fiber is treated with a treatment solution containing a fiber function imparting agent, letting a carrier coexist in the treatment solution. Yet, examples of a function finishing agent only include water repellent, water absorbent and

flame retardant. There is no mention of effects of the carrier on a terminal blocking agent according to the invention. The effects are not concretely examined either. In addition, Japanese Patent Application Laid-Open Publication No. 2000-226765 discloses a method comprising taking up a polymerization initiator using a phthalimide compound in a bath to carry out graft processing. Yet, there is no mention of effects of the phthalimide compound on the terminal blocking agent according to the invention. The effects are not concretely examined either.

PRIOR ART REFERENCES

PATENT DOCUMENTS

[0007]

Patent Document 1: Japanese Patent Application Laid-Open Publication No. 2001-261797
Patent Document 2: Japanese Patent Application Laid-Open Publication No. 2002-30208
Patent Document 3: Japanese Patent Application Laid-Open Publication No. 2010-189813
Patent Document 4: Japanese Patent Application Laid-Open Publication No. 2009-249450
Patent Document 5: Japanese Patent Application Laid-Open Publication No. 2009-263 840
Patent Document 6: Japanese Patent Application Laid-Open Publication No. 2001-98459
Patent Document 7: Japanese Patent Application Laid-Open Publication No. 2000-226765

SUMMARY OF THE INVENTION

PROBLEMS TO BE SOLVED BY THE INVENTION

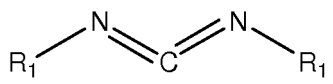
[0008] The present invention was made in view of such conventional backgrounds, and an object thereof is to provide a finishing agent for a polyester-based fiber structure allowing a treated product to efficiently take up a carbodiimide compound and a method of producing the polyester-based fiber structure with excellent hydrolysis-resistant properties using the finishing agent.

MEANS FOR SOLVING THE PROBLEMS

[0009] The present invention comprises the following in order to achieve the above object.

(1) A finishing agent for a polyester-based fiber structure, the finishing agent comprising a carbodiimide compound and a carrier agent emulsified or dispersed in water or a solvent, the carrier agent containing a benzoic acid compound and a phthalimide compound as essential components.
(2) The finishing agent for a polyester-based fiber structure described in the above (1), wherein the carbodiimide compound is a compound represented by the general formula (I):

(Chemical formula 1)



[0010] wherein R1 represents one selected from an alkyl group with 1 to 20 carbon atoms, a cycloalkyl group with 5 to 12 carbon atoms, an aryl group with 6 to 20 carbon atoms, an allyl group and an aralkyl group with 7 to 20 carbon atoms.

(3) The finishing agent for a polyester- based fiber structure described in the above (1) or (2), wherein the carbodiimide is at least one selected from N, N'- di- 2, 6- diisopropylphenyl carbodiimide, N, N'- di- cyclohexyl carbodiimide and N, N'- diisopropyl carbodiimide.
(4) The finishing agent for a polyester-based fiber structure described in any of the above (1) to (3), wherein the carrier agent comprises benzyl benzoate and N-butyl phthalimide as the essential components.
(5) A method of producing a polyester-based fiber structure, the method comprising taking up the finishing agent described in any of the above (1) to (4) into the inside of the polyester-based fiber structure.
(6) A method of producing a polyester-based fiber structure, the method comprising, in the order mentioned, applying a treatment solution containing the finishing agent described in any of the above (1) to (4) to a polyester-based fiber

(s); a drying step; and a heat treatment step.

(7) A method of producing a polyester-based fiber structure, the method comprising supplying a polyester-based fiber(s) into a treatment solution containing the finishing agent described in any of the above (1) to (4) to process the fiber(s) in a bath while circulating the treatment solution.

(8) A method of producing a polyester-based fiber structure, the method comprising applying a treatment solution containing the finishing agent described in any of the above (1) to (4) to a polyester-based fiber(s); and then subjecting the polyester-based fiber(s) to a wet heat treatment.

(9) A polyester-based fiber structure obtained by the method described in any of the above (5) to (8), wherein a concentration of the finishing agent becomes lower from an outer layer toward an inner layer in the cross-section of a single fiber of the polyester-based fiber(s).

EFFECT OF THE INVENTION

[0011] According to the present invention, a carbodiimide compound can be efficiently given to the surface and inside of a fiber structure containing a polyester-based fiber to render a high hydrolysis-resistant property.

BEST MODE FOR CARRYING OUT THE INVENTION

[0012] According to the present invention, treatment with a treatment solution in which, in conjunction with a carbodiimide compound acting as a terminal blocking agent, a carrier agent containing a phthalimide compound and benzoic acid compound as essential components are emulsified or dispersed in water or a solvent allows carbodiimide to be efficiently taken up into the inside of a fiber structure and to react with the carboxyl terminal group of a polymer composing the fiber structure to decrease the concentration of the terminal carboxyl group, thereby rendering hydrolysis-resistant properties.

[0013] A polyester-based fiber in the present invention refers to one having an ester bond in a molecular chain thereof. Aliphatic polyesters and aromatic polyesters are preferably used.

Examples of the aliphatic polyester include one obtained by fusing aliphatic dicarboxylic acid with aliphatic diol, a polymer selected from poly(D-lactic acid), poly(L-lactic acid), a copolymer of D-lactic acid and L-lactic acid, a copolymer of D-lactic acid and hydroxycarboxylic acid, a copolymer of L-lactic acid and hydroxycarboxylic acid and a copolymer of DL-lactic acid and a hydroxycarboxylic acid, and a blended product thereof. Of these, from the viewpoint of general versatility, polylactic acid containing L-lactic acid as a main component, an environment conscious polyester Apexa that has been marketed by Du Pont or the like is preferably used. Containing L-lactic acid as the main component here means that the aliphatic polyester contains 50 % by weight or more of L-lactic acid. Further, a terminal blocking agent may be added to the aliphatic polyester at the time of spinning and thereby some of the terminal carboxyl groups may be blocked.

[0014] Known methods of producing such polylactic acids include a two-step lactide method of once producing a lactide as a cyclic dimer with lactic acid as a raw material and subsequently carrying out ring-opening polymerization, and a one-step direct polymerization method of carrying out direct dehydration condensation in a solvent with lactic acid as a raw material. The polylactic acid used in the present invention may be obtained by either of the methods.

[0015] As the aromatic polyester, polyethylene terephthalate, polytrimethylene terephthalate, polybutylene terephthalate, polyethylene naphthalate, polytrimethylene naphthalate, polybutylene naphthalate or the like is used. Further, these aromatic polyesters may contain other copolymerization components and are not limited to the above polyesters.

[0016] Examples of the copolymerization component for polyester include, but are not limited to, dimer diol aimed at improving alkaline resistance hydrolysis properties, a glycol component aimed at improving chromogenic properties, a multifunctional phosphorus compound aimed at rendering flame retardant properties and a sulfoisophthalic acid salt aimed at rendering cationic dye-dyeability.

[0017] Concrete examples of the above dimer diol include Pespole HP-1000 (hydrogenated dimer diol having 36 carbon atoms with alicyclic-type/linear aliphatic-type = 75/25 (mol%)) manufactured by Toagosei Co., Ltd.

[0018] Concrete examples of the glycol component include a diol compound in which an ethylene oxide is added to a compound such as 1, 5- pentanediol, 1, 6- hexanediol, 1, 9- nonanediol, neopentylglycol, bisphenol A or bisphenol S, a copolymerization product of propylene oxide and ethylene oxide and polyoxyalkylene glycol.

[0019] As the above multifunctional phosphorus compound, to be specific, phenylphosphonic acid dimethyl ester, phenylphosphonic acid diphenyl ester or the like is preferably used. Examples of phosphinates include phosphates such as (2- carboxylethyl) methylphosphinic acid, (2- methoxycarbonylethyl) methylphosphinic acid methyl ester, (2- carboxylethyl) phenylphosphinic acid, (2- methoxycarbonylethyl) phenylphosphinic acid methyl ester, (4- methoxycarbonylphenyl) phenylphosphinic acid methyl ester or ethylene glycol ester of [2- (β- hydroxy ethoxycarbonyl) ethyl] methylphosphinic acid; and phosphine oxides such as (1, 2- dicarboxyethyl) dimethyl phosphine oxide, (2, 3- dicarboxypropyl) dimethyl phosphine oxide, (1, 2- dimethoxy carbonylethyl) dimethyl phosphine oxide, (2, 3- dimethoxy carbonylethyl) dimethyl phosphine oxide, [1, 2di (β- hydroxy ethoxycarbonyl) ethyl] dimethyl phosphine oxide or [2, 3di (β- hydroxy ethoxycarbonyl)

ethyl] dimethyl phosphine oxide.

[0020] Concrete examples of the above sulfoisophthalic acid salt include an alkali metal salt of sulfoisophthalic acid, a phosphonium salt of sulfoisophthalic acid and an ester- forming derivative derived therefrom. Concrete examples thereof include an alkali metal salt of sulfoisophthalic acid such as 5- sodium sulfoisophthalic acid or 5- lithium sulfoisophthalic acid, 5- (tetraalkyl) phosphonium sulfoisophthalic acid and an ester- forming derivative derived therefrom.

[0021] The polyester-based fiber used in the present invention may be, besides ordinary flat yarns, filament yarns such as false twisted yarns, strong twisted yarns, Taslan yarns, irregularly thick and fine yarns, mixed yarns or the like, and may also be a fiber of various modes such as staple fiber, tow, spun yarns, fabric or the like.

[0022] The polyester-based fiber used in the present invention may also form an alloy with another polymer such as a polyamide.

In the polyester-based fiber used in the present invention, a natural fiber, regenerated fiber, semi-synthetic fiber, synthetic fiber or the like can be mixed to be used. The mixing mode may be any mode of fibers-mixed spinning, threads-mixed weaving, threads-mixed knitting, or the like. Examples of the mode of the fiber structure include, but are not limited to, a filament, spun yarn, fiber structure obtained formed therefrom such as a woven fabric, knitted fabric, nonwoven fabric or other manufactured article.

Examples of the natural fiber include a cotton fiber, kapok fiber, hemp fiber, flax fiber, cannabis fiber, ramie fiber, wool fiber, alpaca fiber, cashmere fiber, mohair fiber and silk fiber. Examples of the regenerated fiber include a viscose fiber, Cupra fiber, polynosic fiber, high wet modulus rayon fiber and solvent-spun cellulose fiber. Examples of the semi-synthetic fiber include an acetate fiber, diacetate fiber and triacetate fiber. Examples of the synthetic fiber include a polyamide fiber, acrylic fiber, vinylon fiber, polypropylene fiber, polyurethane fiber, polyvinyl chloride fiber, polyethylene fiber and promix fiber.

In the present invention, a polyester-based fiber and other fibers can be mixed to use by any arbitrary method, but if the mixing rate of polyester-based fiber is small, effects of the present invention are small. It is therefore preferred that the mixing rate of the polyester-based fiber be 30% by weight or more. More preferred is 50% by weight or more.

In the present invention, a polyester-based fiber structure is treated with a treatment solution containing a finishing agent in which a carbodiimide-based terminal blocking agent and a carrier containing a phthalimide compound and benzoic acid compound as essential components are emulsified or dispersed in water or a solvent. By this treatment, the finishing agent is taken up into the inside of the fiber structure.

As a method of treating a polyester-based fiber with a treatment solution containing a finishing agent, preferably used is a method comprising placing the polyester-based fiber in a treatment solution containing a finishing agent of the present invention and then processing in a bath with the treatment solution being circulated.

When the processing in the bath is carried out while the treatment solution is circulated, examples of the mode of the treated product include, but are not limited to, a fabric, yam, other manufactured article, tow and cotton batting. As the treatment apparatus for processing in the bath, an apparatus including, but not limited to, a wince dyeing machine, jigger dyeing machine, paddle dyeing machine, drum-type dyeing machine, liquid flow dyeing machine, air flow dyeing machine, beam dyeing machine, cheese dyeing machine and obermaier can be utilized.

It is referred to immerse a fabric in a treatment solution and subject it to heat treatment at 80 to 130°C at normal pressure or under pressurization. It is preferred that the heat treatment time be 10 to 120 minutes. In the case of an aliphatic polyester, it is more preferred that the treatment be carried out at 90 to 110°C for 20 to 60 minutes. In the case of an aromatic polyester, it is more preferred that the treatment be carried out at 110 to 110°C for 20 to 60 minutes. In this case, the terminal blocking agent is attached onto the fiber and taken up to disperse in the inside of the fibers. In cases where the treatment time is short, the terminal blocking agent may be inadequately taken up to disperse in the inside of the fiber, and satisfactory hydrolysis-resistant properties can in some cases not be attained. Further, in cases where the treatment time is too long, the polyester ends up being hydrolyzed during the treatment.

In such a method, after the treatment in the solution, dehydration and drying are carried out. As long as water can be dried, the drying may be carried out in any condition with 100 to 140°C being preferred. As for a heat treatment that is carried out after the drying, it is preferred to treat at 80 to 200°C. It is preferred that the treatment time be 15 seconds to 8 minutes. In the case of an aliphatic polyester, it is more preferred to treat at 90 to 140°C for 30 seconds to 5 minutes. In the case of an aromatic polyester, it is more preferred to treat at 130 to 190°C for 30 seconds to 5 minutes. If the treatment solution is mixed with a hydrophobic dye typified by a disperse dye, terminal blocking treatment and dyeing can be concurrently carried out. For the dehydration, drying, and heat treatment after the treatment in the solution, conditions for drying and finishing sets employed in an ordinary dyeing step may be applied.

As a heat treatment apparatus, a tenter, short loop dryer, shrink surfer, steamer or cylinder dryer, or the like can be utilized, but the apparatus is not limited thereto as long as it can give heat uniformly to the fiber.

Also, examples of the method of treating a polyester-based fiber with a treatment solution containing a finishing agent includes a method of giving the treatment solution containing the finishing agent to the polyester-based fiber using a device apparatus such as a mangle, followed by drying and heat treatment.

As an apparatus for giving the treatment solution containing the finishing agent of the present invention to the polyester-

based fiber, an ordinary mangle can be suitably used as a liquid-giving apparatus, but the apparatus is not restricted as long as it can give the solution uniformly to the fiber. A foam processing machine or print method, ink jet, spray method, coating method or the like may also be used for giving the solution.

As a drying or heat treatment apparatus, a tenter, short loop dryer, shrink surfer, steamer or cylinder dryer, or the like can be utilized, but the apparatus is not limited thereto as long as it can give heat uniformly to the fiber. A fabric is immersed in the treatment solution containing a finishing agent and squeezed uniformly, followed by dry heat treatment. It is preferred that the drying temperature be 80°C to 150°C. It is preferred that the treatment time be 15 seconds to 5 minutes. In the case of an aliphatic polyester, it is more preferred to treat at 90 to 110°C for 30 seconds to 3 minutes. In the case of an aromatic polyester, it is more preferred to treat at 100 to 140°C for 30 seconds to 3 minutes. In cases where the drying temperature is too high, the terminal blocking agent may in some cases react with water in a drying step and become deactivated.

It is preferred that heat treatment after the drying be carried out at 80 to 200°C. It is preferred that the treatment time be 15 seconds to 8 minutes. In the case of an aliphatic polyester, it is more preferred to treat at 90 to 140°C for 30 seconds to 5 minutes. In the case of an aromatic polyester, it is more preferred to treat at 130 to 190°C for 30 seconds to 5 minutes. In cases where the treatment temperature is too high, polyester ends up melting. Also, in cases where the treatment time is too long, the polyester ends up being hydrolyzed during the treatment.

Further, As a method of treating a polyester-based fiber in a treatment solution containing the finishing agent, a method of wet heat treatment after giving the treatment solution containing the finishing agent of the present invention to the polyester-based fiber is preferably used. The wet heat treatment, as compared with the above two methods of the dry heat treatment, has better heat conduction and allows the terminal blocking agent to more efficiently react with the polyester-based fiber.

Similarly to the above, the wet heat treatment is carried out after giving the treatment solution using a mangle or the like. As a wet heat treatment apparatus, an atmospheric steamer, high pressure steamer or the like can be utilized, but the apparatus is not limited thereto as long as it can give heat uniformly to the fiber. It is preferred that a fabric is immersed in the treatment solution containing a terminal blocking agent and squeezed uniformly, followed by wet heat treatment at 80 to 130°C. It is preferred that the treatment time be 15 seconds to 8 minutes. In the case of an aliphatic polyester, it is more preferred to treat at 90 to 105°C for 30 seconds to 5 minutes. In the case of an aromatic polyester, it is more preferred to treat at 105 to 130°C for 30 seconds to 5 minutes. In cases where the treatment time is too long, the polyester ends up being hydrolyzed during the treatment.

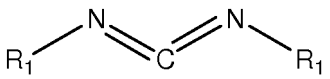
If a treatment solution containing a terminal blocking agent is mixed with a hydrophobic dye typified by a disperse dye, terminal blocking treatment and dyeing can be concurrently carried out. When the terminal blocking treatment and dyeing are concurrently carried out, the dye concentration is enhanced. Further, the number of times of undergoing a wet heat treatment step decreases and the hydrolysis of polyester is thus inhibited.

As the hydrophobic dye, vat dye, indigo dye, naphthol dye or the like can also be used.

A carbodiimide compound used as a terminal blocking agent in the present invention may be any compound as long as it has at least one carbodiimide group. For instance, a compound represented by the following general formula (I) is used.

[0023]

(Chemical formula 2)



[0024] In the general formula (I), R1 represents one selected from an alkyl group with 1 to 20 carbon atoms, a cycloalkyl group with 5 to 12 carbon atoms, an aryl group with 6 to 20 carbon atoms, an allyl group and an aralkyl group with 7 to 20 carbon atoms.

[0025] Concrete examples thereof include N, N'- di- o- tolylcarbodiimide, N, N'- diphenylcarbodiimide, N, N'- dioctyldecylcarbodiimide, N, N'- di- 2, 6- dimethylphenylcarbodiimide, N- tolyl- N'- cyclohexylcarbodiimide, N, N'- di- 2, 6- diisopropylphenylcarbodiimide, N, N'- di- 2, 6- di- tert.- butylphenylcarbodiimide, N, N'- di- p- nitrophenylcarbodiimide, N, N'- di- p- aminophenylcarbodiimide, N, N'- di- p- hydroxyphenylcarbodiimide, N, N'- di- cyclohexylcarbodiimide, N, N'- di- p- tolylcarbodiimide, p- phenylene- bis- di- o- tolylcarbodiimide, p- phenylene- bis- dicyclohexylcarbodiimide, hexamethylene- bis- dicyclohexylcarbodiimide, ethylene- bis- diphenylcarbodiimide, N, N'- benzylcarbodiimide, N- octadecyl- N'- phenylcarbodiimide, N- benzyl- N'- phenylcarbodiimide, N- octadecyl- N'- tolylcarbodiimide, N- phenyl- N'- tolylcarbodiimide, N- benzyl- N'- tolylcarbodiimide, N, N'- di- o- ethylphenylcarbodiimide, N, N'- di- p- ethylphenylcarbodiimide, N, N'- di- o- isopropylphenylcarbodiimide, N, N'- di- p- isopropylphenylcarbodiimide, N, N'- di- o- isobutylphenylcarbodiimide, N, N'- di- p- isobutylphenylcarbodiimide, N, N'- di- 2, 6- diethylphenylcarbodiimide, N, N'- di- 2- ethyl-

6- isopropylphenylcarbodiimide, N, N'- di- 2- isobutyl- 6- isopropylphenylcarbodiimide, N, N'- di- 2, 4, 6- trimethylphenylcarbodiimide, N, N'- di- 2, 4, 6- triisopropylphenylcarbodiimide, N, N'- di- 2, 4, 6- triisobutylphenylcarbodiimide and N, N'- diisopropylcarbodiimide.

[0026] Further, one or more compounds may be arbitrarily selected from these carbodiimide compounds to block the carboxyl terminus of the polyester.

[0027] Furthermore, as an industrially available carbodiimide compound, N, N'- di- 2, 6- diisopropylphenylcarbodiimide (TIC) and N, N'- di- cyclohexylcarbodiimide (DCC), N, N'- diisopropylcarbodiimide (DIC) can also be suitably used. Suitable examples are "Stabaxol" I, "Stabaxol" I LF, "Stabaxol" P, and "Stabaxol" P- 100, all of which are marketed under the trade name of "Stabaxol" by Rhein Chemie Japan Ltd.

[0028] A carrier agent used in the present invention is added for the purpose of swelling a polyester chain and efficiently taking up a terminal blocking agent into the inside of a fiber. Trichlorobenzene, methylnaphthalene and the like, which have been conventionally used, have strong odor and readily cause carrier spots. Thus, there is concern about the odor and quality of a final manufactured article in conjunction with worsened working environment. As a result of intensive studied conducted for solving such problems, it has been found that use of a phthalimide compound and benzoic acid compound enables a terminal blocking agent to be efficiently taken up into the inside of a polyester fiber.

The phthalimide compound used in the present invention is a compound having a phthalimide group. Preferred is phthalimide having an aliphatic or aromatic alkyl group or the like in the N group of phthalimide. Examples of substituent group include methyl, ethyl, propyl, isopropyl, butyl, isobutyl, benzyl and naphthal. From the viewpoint of remained amount in a processed manufactured article, odor, safety, handling workability or the like, more preferred is N-phthalimide having low molecular weight aliphatic alkyl group such as methyl, ethyl, propyl, isopropyl, butyl or isobutyl. Of these, N-butylphthalimide is preferably used in that it has excellent compatibility with the carbodiimide compound.

A benzoic acid compound used in the present invention refers to a benzoic acid derivative. Preferred is a benzoate formed from benzoic acid and aliphatic or aromatic alcohol. Examples of the benzoate include ethyl benzoate, methyl benzoate, propyl benzoate, butyl benzoate, benzyl benzoate and phenyl benzoate.

In the viewpoint of compatibility with the carbodiimide compound or swelling effects of a polyester chain, benzyl benzoate, phenyl benzoate or the like is more preferred. Of these, preferably used is benzyl benzoate that has molecular weight close to TIC, which is a terminal blocking agent, and can be inexpensively obtained.

A mixing ratio of the phthalimide compound with the benzoic acid compound is, based on 50 parts by weight of phthalimide compound, preferably 10 parts by weight to 50 parts by weight of benzoic acid compound, more preferably 15 parts by weight to 40 parts by weight of benzoic acid compound, and still more preferably 20 parts by weight to 30 parts by weight of benzoic acid compound. In addition, two or more types of the phthalimide compounds and two or more types of the benzoic acid compounds may be used.

[0029] As a mixture ofN-phthalimide and benzoate, UNIVADINE PB has been marketed by Huntsman Corporation and can be suitably used.

[0030] The ratio of the carbodiimide compound with the mixed carrier agent is, based on 25 parts by weight of the carbodiimide compound, preferably 20 parts by weight to 35 parts by weight of the mixed carrier agent and more preferably 25 to 30parts by weight of the mixed carrier agent.

The finishing agent for a polyester-based fiber structure by the present invention is made by emulsifying or dispersing the above carbodiimide compound and a carrier agent containing a benzoic acid compound and phthalimide compound as essential components in water or a solvent. It is preferred that at least one surfactant selected from either a nonionic surfactant or anionic surfactant be used as an emulsifying agent or dispersing agent.

As for a carbodiimide compound used as a terminal blocking agent in the present invention, the terminal blocking agent can, for example, be mixed with the above-mentioned carrier agent and surfactant and, as necessary, an organic solvent, and heated to form a homogeneous molten product, which is allowed to cool to obtain a self emulsification-type finishing agent which is in a liquid form at normal temperature. At the time of carrying out the terminal blocking processing of polyester-based fiber structure, if the above self emulsification-type finishing agent can be added with water and stirred, an emulsified product of the terminal blocking agent with water being a dispersion medium can be obtained.

[0031] On the other hand, without using the organic solvent, the terminal blocking agent can, for example, be mixed with the above-mentioned aliphatic hydrocarbon-based compatibilizing agent and surfactant, heated to form a homogeneous molten product which is then gradually added to heated water while stirred, to emulsify and allow to cool, thereby obtaining, similarly to the above, an emulsified product of the terminal blocking agent with water being a dispersion medium.

[0032] In the present invention, when a emulsified product of a carbodiimide compound which is a terminal blocking agent is obtained, for the purpose of keeping the resulting emulsified product homogeneous or improving the emulsifying property of the carbodiimide compound, an organic solvent can, as described above, be used as necessary to the extent where the rate of taking up the carbodiimide compound is not affected. Examples of this organic solvent include aromatic hydrocarbons such as toluene, xylene or alkyl naphthalene; ketones such as acetone or methyl ethyl ketone; alcohols such as methyl alcohol or ethyl alcohol; glycols such as ethylene glycol or propylene glycol; ethers such as dioxane;

alkylene glycol alkyl ethers such as ethylene glycol monomethyl ether, ethylene glycol monobutyl ether or ethylene glycol monoisobutyl ether; amides such as dimethylformamide; sulfoxides such as dimethyl sulfoxide; and halogenated hydrocarbons such as methylene chloride or chloroform. These organic solvents may be used solely or two or more types thereof may be used in combination as necessary.

[0033] It is preferred in the present invention that, as described above, at least one type of the surfactant selected from either a nonionic surfactant or anionic surfactant be combined to use when a terminal blocking agent composed of the carbodiimide compound is emulsified in water together with the carrier agent. Examples of the above nonionic surfactant include polyoxyalkylene-type nonionic surfactant such as higher alcohol alkylene oxide adduct, alkyl phenol alkylene oxide adduct, styrenated phenol alkylene oxide adduct, fatty acid alkylene oxide adduct, polyol aliphatic ester alkylene oxide adduct, higher alkylamine alkylene oxide adduct or fatty acid amide alkylene oxide adduct; and polyol-type nonionic surfactant such as alkyl glycoside or sucrose fatty acid ester. These nonionic surfactants may be used solely or two or more types thereof may be used in combination as necessary.

[0034] Meanwhile, examples of the above anionic surfactant include carboxylate such as fatty acid soap; sulfate ester salt such as higher alcohol sulfate ester salt, higher alkyl polyalkylene glycol ether sulfate ester salt, sulfate ester salt of styrenated phenol alkylene oxide adduct, sulfate ester salt of alkyl phenol alkylene oxide adduct, sulfated oil, sulfated fatty acid ester, sulfated fatty acid or sulfated olefin; formalin fusion product such as alkyl benzene sulfonate, alkyl naphthalenesulfonate, naphthalenesulfonate or naphthalenesulfonic acid; sulfonate such as α- olefin sulfonate, paraffin sulfonate or sulfosuccinic acid diester salt; and higher alcohol phosphate ester salt. These anionic surfactants may be used solely or two or more types thereof may be used in combination as necessary.

In addition, the nonionic surfactant and anionic surfactant may be combined as necessary.

The amount of the above surfactant added is, based on 25 parts by weight of the carbodiimide compound, preferably 0.1 to 3.5 parts by weight, more preferably 0.1 to 3.0 parts by weight, and still more preferably 0.1 to 2.5 parts by weight. If the surfactant is less than 0.1 parts by weight the carbodiimide compound is not adequately emulsified and dispersed, whereas if it is more than 3.5 parts by weight a rate of taking up the carbodiimide compound might decrease.

[0035] As equipment used emulsified dispersion for obtaining the emulsified product in the present invention, a propeller-type stirring machine, piston-type high pressure emulsification machine, homo mixer, ultrasonic emulsified dispersion machine, pressurized nozzle-type emulsification machine, high-speed rotating high shear-type stirring disperser or the like can be used. Two or more types of these equipment can be used in combination.

[0036] The amount of terminal blocking agent used in the present invention may be just determined in accordance with the amount of the terminal carboxyl group of a polyester-based fiber to be subjected.

[0037] The polyester-based fibers structure obtained by the present invention have excellent hydrolysis resistance and can be preferably used as dress shirts, blouses, pants, skirts, polo shirts, T shirts, training wear, coats, sweaters, pajamas, school uniforms, work clothes, white robes, clean room wear, unlined kimonos, underwear, linings, interlinings or the like. In particular, ones subjected to the terminal blocking treatment with polyethylene terephthalate can be preferably used for uniforms for medical treatment, nursing care, food products, which uniforms require autoclaving treatment at 120°C to 130°C.

EXAMPLES

[0038] By way of examples, the present invention will now be described more specifically below but the present invention is by no means limited thereto. Meanwhile, the physical properties in the examples were measured in the following manner.

(Measurement of Various Physical Properties)

[0039]

(1) Terminal carboxyl group concentration (equivalents/10^3 kg) of polylactic acid: An accurately weighed sample was dissolved into an o-cresol solution (water content 5%), and an adequate amount of dichloromethane was added to the solution. Subsequently, a 0.02 N potassium hydroxide methanol solution was used for titration, to measure the concentration.

(2) Terminal carboxyl group concentration (equivalents/10^3 kg) of polyethylene terephthalate, polytrimethylene terephthalate, and polybutylene terephthalate: An accurately weighed sample was dissolved into benzyl alcohol, and chloroform was added to the solution. Subsequently, a 0.1 N potassium hydroxide benzyl alcohol solution was used for titration to measure the concentration.

(3) Molecular weight of polylactic acid: A sample is immersed in chloroform to obtain a measurement solution which is a chloroform solution in which only PLA portion is dissolved. This was measured by gel permeation chromatography (GPC) to determine weight average molecular weight in terms of polystyrene.

(4) Strength (cN/dtex): Measurement was carried out at a sample length of 20 cm and at a stress rate of 20 cm/min using Shimadzu Autograph AG-1S.
(5) Hydrolysis test: A hydrolysis test was carried out by the following three types of methods.

[0040]

(i) Using a thermo-hygrostat tester THN064PB manufactured by Toyo Engineering Works, Ltd., a sample was placed in the thermo-hygrostat at 70°C and 90% RH and hydrolysis treatment was carried out for seven days.

[0041]

(ii) Using UR-MINI-COLOR (infrared ray Mini-Color (manufactured by Texam Co., Ltd.)), hydrolysis treatment was carried out in conditions of a liquor ratio of 1:50 at 130°C and for 48 hours.

[0042]

(iii) Using UR-MINI-COLOR (infrared ray Mini-Color (manufactured by Texam Co., Ltd.)), hydrolysis treatment was carried out in conditions of a liquor ratio of 1:50 at 130°C for 16 hours.

(6) Strength retaining rate (%): Let the strength of sample after the terminal blocking treatment be A and let the strength of sample subjected to the hydrolysis treatment in the conditions of the above (i), (ii) or (iii) be B, the strength retaining rate was calculated from the following formula:

$$\text{Strength retaining rate (\%)} = \{(\text{Strength after hydrolysis treatment A}) / (\text{Strength after terminal blocking treatment B})\} \times 100$$

The following describes a fabric used in the present example.

(Production of polylactic acid fabric)

[0043] L- polylactic acid chips with a melting point of 166°C were dried in a vacuum dryer that was set at 105°C for 12 hours. The dried chips were charged into a melt spinning machine and melt- spun at a melting temperature of 210°C, at a spinning temperature of 220°C and at a spinning speed of 4500 m/min to obtain product type 100 dtex- 26 filament undrawn yarns. The undrawn yarns were stretched at a preheating temperature of 100°C, at a heat set temperature of 130°C and at a draw ratio of 1.2 times, to obtain 84 dtex- 26 filament drawn yarns. The obtained drawn yarns were used to weave taffeta that was scoured at 80°C and subjected to dry heat set at 130°C for 1 minute to obtain a polylactic acid woven fabric.

(Production of polyethylene terephthalate fabric)

[0044] A known method was used to obtain 84 dtex-26 filament polyethylene terephthalate (PET) drawn yarns. The obtained filament was used to weave taffeta that was scoured at 80°C for 20 minutes and subjected to dry heat set at 170°C for 1 minute to obtain a PET woven fabric.

(Production of fabric of polylactic acid/polytrimethylene terephthalate core-sheath yarns)

[0045] Poly L-lactic acid (optical purity 97% L-lactic acid) (PLA) with a weight average molecular weight of 165,000, a melting point of 170°C, and an residual lactide amount of 0.085% by weight was used as a core part A and polytrimethylene terephthalate (PTT) (melting point 228°C) containing 0.3% by weight titanium oxide with an average secondary particle size of 0.4 μm was used as a sheath part. They were separately melted at a spinning temperature of 250°C, at a core-sheath composition ratio (% by weight) of 70:30 and at a spinning speed of 3000 m/min to obtain 110 decitex, 36 filament undrawn yarns with a core-sheath complex structure.

[0046] The undrawn yarns were further stretched at a drawing speed of 800m/min, at a draw ratio of 1.3 times, at a drawing temperature of 90°C, and at a heat set temperature of 130°C to obtain 84 decitex, 48 filament drawn yarns. The obtained drawn yarns were used to weave taffeta that was scoured at 80°C for 20 minutes and subjected to dry heat set at 130°C for 2 minutes to obtain a PET woven fabric.

(Production of polybutylene terephthalate fabric)

[0047] A known method was used to obtain 84 dtex-24 filament polybutylene terephthalate (PBT) drawn yarns. The obtained filament was used to weave taffeta that was scoured at 80°C for 20 minutes and subjected to dry heat set at 170°C for 1 minute to obtain a PBT woven fabric.

(Production of "Apexa"/cotton fabric)

[0048] "Apexa" that is an environment conscious polyester Du Pont had been marketed and cotton were mixed and spun at a ratio of 45/55 by a known method to obtain No. 45 count (131.2 dtex) spun yarns (A/C). The obtained spun yarns were used to weave taffeta that was subjected to desizing at 100°C for 30 minutes, bleached and scoured at 90°C for 30 minutes, and subjected to dry heat set at 190°C for 1 minute to obtain an A/C woven fabric.

(Production of polyethylene terephthalate/cotton fabric)

[0049] Polyethylene terephthalate (PET) and cotton were mixed and spun at a ratio of 40/60 by a known method to obtain No. 42 count (140.6 dtex) spun yam A. In addition, PET and cotton were mixed and spun at a ratio of 65/35 to obtain No. 45 count (131.2 dtex) spun yam B. The obtained spun yam A and spun yam B were used as warp and one obtained by aligning 84 dtex-26 filament PET yarns that were obtained by a known method with stretching was used as weft to obtain twill. The obtained twill was subjected to desizing at 100°C for 30 minutes, bleached and scoured at 90°C for 30 minutes, and subjected to dry heat set at 190°C for 1 minute to obtain a polyethylene terephthalate/cotton (PET/C) woven fabric. The mixing rate of the PET/C woven fabric was 63/37.

(Production of cationic dye-dyeable polyester fabric)

[0050] A method disclosed in Japanese Patent Application Laid-Open Publication No. 2007-169856 was used to make a 24-gauge circular knitting of a weight of 150 g/m^2 using a cationic dye-dyeable polyester fiber composed of 84 dtex-26 filament sulfonated aromatic dicarboxylic acid modified polyethylene terephthalate. The obtained knit was scoured at 80°C for 20 minutes and subjected to dry heat set at 170°C for 1 minute to obtain a cationic dye-dyeable polyester (CDP) knit.

[0051] A method of producing a terminal blocking finishing agent used in the present examples will be described below.

(Preparation of terminal blocking finishing agent 1)

[0052] Twenty (20.0) parts by weight of bis (2, 6- diisopropylphenyl) carbodiimide (Stabaxol I LF; Rhein Chemie Japan Ltd.,), 5.0 parts by weight of benzyl benzoate (Nacalai Tesque, Inc.), 10.0 parts by weight of N- butylphthalimide (Nacalai Tesque, Inc.), and 2.0 parts by weight of sulfated castor oil (Turkey red oil; Miyoshi Oil & Fat Co., Ltd.) were mixed, heated to 60°C and uniformly melted. The resultant was gradually added, while stirred by a propeller- type stirring machine, to 50.0 parts by weight of 70°C water for emulsified dispersion and allowed to cool to obtain terminal blocking finishing agent 1.

(Preparation of terminal blocking finishing agent 2)

[0053] The terminal blocking finishing agent 2 was obtained in the same manner except that the N-butylphthalimide of the terminal blocking finishing agent 1 was altered to N-butylphthalimide (Nacalai Tesque, Inc.).

(Preparation of terminal blocking finishing agent 3)

[0054] The terminal blocking finishing agent 3 was obtained in the same manner except that the N-butylphthalimide of the terminal blocking finishing agent 1 was altered to N-propylphthalimide (Nacalai Tesque, Inc.).

(Preparation of terminal blocking finishing agent 4)

[0055] The terminal blocking finishing agent 4 was obtained in the same manner except that the N-butylphthalimide of the terminal blocking finishing agent 1 was altered to N-benzylphthalimide (Nacalai Tesque, Inc.).

(Preparation of terminal blocking finishing agent 5)

[0056] The terminal blocking finishing agent 5 was obtained in the same manner except that the benzyl benzoate of the terminal blocking finishing agent 1 was altered to phenyl benzoate (Nacalai Tesque, Inc.).

(Preparation of terminal blocking finishing agent 6)

[0057] The terminal blocking finishing agent 6 was obtained in the same manner except that the benzyl benzoate of the terminal blocking finishing agent 2 was altered to phenyl benzoate.

(Preparation of terminal blocking finishing agent 7)

[0058] The terminal blocking finishing agent 7 was obtained in the same manner except that the benzyl benzoate of the terminal blocking finishing agent 3 was altered to phenyl benzoate.

(Preparation of terminal blocking finishing agent 8)

[0059] The terminal blocking finishing agent 8 was obtained in the same manner except that the benzyl benzoate of the terminal blocking finishing agent 4 was altered to phenyl benzoate.

(Preparation of terminal blocking finishing agent 9)

[0060] The terminal blocking finishing agent 9 was obtained in the same manner except that the benzyl benzoate of the terminal blocking finishing agent 1 was altered to butyl benzoate (Nacalai Tesque, Inc.).

(Preparation of terminal blocking finishing agent 10)

[0061] The terminal blocking finishing agent 10 was obtained in the same manner except that the benzyl benzoate of the terminal blocking finishing agent 2 was altered to butyl benzoate.

(Preparation of terminal blocking finishing agent 11)

[0062] The terminal blocking finishing agent 11 was obtained in the same manner except that the benzyl benzoate of the terminal blocking finishing agent 3 was altered to butyl benzoate.

(Preparation of terminal blocking finishing agent 12)

[0063] The terminal blocking finishing agent 12 was obtained in the same manner except that the benzyl benzoate of the terminal blocking finishing agent 4 was altered to butyl benzoate.

(Preparation of terminal blocking finishing agent 13)

[0064] The terminal blocking finishing agent 13 was obtained in the same manner except that the bis (2, 6- diisopropylphenyl) carbodiimide of the terminal blocking finishing agent 1 was altered to diisopropylcarbodiimide (Tokyo Chemical Industry Co., Ltd.) .

(Preparation of terminal blocking finishing agent 14)

[0065] The terminal blocking finishing agent 14 was obtained in the same manner except that the bis (2, 6- diisopropylphenyl) carbodiimide of the terminal blocking finishing agent 1 was altered to dicyclohexylcarbodiimide (Tokyo Chemical Industry Co., Ltd.) .

(Preparation of terminal blocking finishing agent 15)

[0066] The terminal blocking 15 was obtained in the same manner as the terminal blocking finishing agent 1 using 20.0 parts by weight of bis (2, 6- diisopropylphenyl) carbodiimide, 15.0 parts by weight of trichlorobenzene (Nacalai Tesque, Inc.), 2.0 parts by weight of sulfated castor oil and 20.0 parts by weight of water.

(Preparation of terminal blocking finishing agent 16)

[0067] The terminal blocking finishing agent 16 was obtained in the same manner except that the trichlorobenzene of the terminal blocking finishing agent 15 was altered to methylnaphthalene.

(Preparation of terminal blocking finishing agent 17)

[0068] The terminal blocking finishing agent 17 was obtained in the same manner except that the trichlorobenzene of the terminal blocking finishing agent 15 was altered to benzyl benzoate.

(Preparation of terminal blocking finishing agent 18)

[0069] The terminal blocking finishing agent 18 was obtained in the same manner except that the trichlorobenzene of the terminal blocking finishing agent 15 was altered to phenyl benzoate.

(Preparation of terminal blocking finishing agent 19)

[0070] The terminal blocking finishing agent 19 was obtained in the same manner except that the trichlorobenzene of the terminal blocking finishing agent 15 was altered to butyl benzoate.

(Preparation of terminal blocking finishing agent 20)

[0071] The terminal blocking finishing agent 20 was obtained in the same manner except that the trichlorobenzene of the terminal blocking finishing agent 15 was altered to N-butylphthalimide.

(Preparation of terminal blocking finishing agent 21)

[0072] The terminal blocking finishing agent 21 was obtained in the same manner except that the trichlorobenzene of the terminal blocking finishing agent 15 was altered to N-ethylphthalimide.

(Preparation of terminal blocking finishing agent 22)

[0073] The terminal blocking finishing agent 22 was obtained in the same manner except that the trichlorobenzene of the terminal blocking finishing agent 15 was altered to N-propylphthalimide.

(Preparation of terminal blocking finishing agent 23)

[0074] The terminal blocking finishing agent 23 was obtained in the same manner except that the trichlorobenzene of the terminal blocking finishing agent 15 was altered to N-benzylphthalimide.

(Preparation of terminal blocking finishing agent 24)

[0075] The terminal blocking finishing agent 24 was obtained in the same manner except that the amount of benzyl benzoate added was altered to 1.0 part by weight and the amount of N-butylphthalimide added was altered to 14 parts by weight in the terminal blocking finishing agent 1.

(Preparation of terminal blocking finishing agent 25)

[0076] The terminal blocking finishing agent 25 was obtained in the same manner except that the amount of benzyl benzoate added was altered to 9.0 parts by weight and the amount of N-butylphthalimide added was altered to 6.0 parts by weight in the terminal blocking finishing agent 1.

(Preparation of terminal blocking finishing agent 26)

[0077] The terminal blocking finishing agent 26 was obtained in the same manner except that the amount of phenyl benzoate added was altered to 1.0 part by weight and the amount of N-butylphthalimide added was altered to 14 parts by weight in the terminal blocking finishing agent 5.

(Preparation of terminal blocking finishing agent 27)

[0078] The terminal blocking finishing agent 27 was obtained in the same manner except that the amount of phenyl benzoate added was altered to 9.0 parts by weight and the amount of N-butylphthalimide added was altered to 6.0 parts by weight in the terminal blocking finishing agent 5.

(Preparation of terminal blocking finishing agent 28)

[0079] The terminal blocking finishing agent 28 was obtained in the same manner except that the amount of benzyl benzoate added was altered to 1.0 part by weight and the amount of N-benzylphthalimide added was altered to 14 parts by weight in the terminal blocking finishing agent 4.

(Preparation of terminal blocking finishing agent 29)

[0080] The terminal blocking finishing agent 29 was obtained in the same manner except that the amount of benzyl benzoate added was altered to 9.0 parts by weight and the amount of N-benzylphthalimide added was altered to 6.0 parts by weight in the terminal blocking finishing agent 4.

(Preparation of terminal blocking finishing agent 30)

[0081] The terminal blocking finishing agent 30 was obtained in the same manner except that the amount of phenyl benzoate added was altered to 1.0 part by weight and the amount of N-benzylphthalimide added was altered to 14 parts by weight in the terminal blocking finishing agent 8.

(Preparation of terminal blocking finishing agent 31)

[0082] The terminal blocking finishing agent 31 was obtained in the same manner except that the amount of phenyl benzoate added was altered to 9.0 parts by weight and the amount of N-benzylphthalimide added was altered to 6.0 parts by weight in the terminal blocking finishing agent 8.

(Preparation of terminal blocking finishing agent 32)

[0083] The terminal blocking finishing agent 32 was obtained in the same manner except that the amount of benzyl benzoate added was altered to 11 parts by weight and the amount of N-butylphthalimide added was altered to 22 parts by weight in the terminal blocking finishing agent 1.

(Preparation of terminal blocking finishing agent 33)

[0084] The terminal blocking finishing agent 33 was obtained in the same manner except that the amount of benzyl benzoate added was altered to 3.0 parts by weight and the amount of N-butylphthalimide added was altered to 6.0 parts by weight in the terminal blocking finishing agent 1.

(Preparation of terminal blocking finishing agent 34)

[0085] The terminal blocking finishing agent 34 was obtained in the same manner except that the amount of phenyl benzoate added was altered to 11 parts by weight and the amount of N-butylphthalimide added was altered to 22 parts by weight in the terminal blocking finishing agent 5.

(Preparation of terminal blocking finishing agent 35)

[0086] The terminal blocking finishing agent 35 was obtained in the same manner except that the amount of phenyl benzoate added was altered to 3.0 parts by weight and the amount of N-butylphthalimide added was altered to 6.0 parts by weight in the terminal blocking finishing agent 5.

(Preparation of terminal blocking finishing agent 36)

[0087] The terminal blocking finishing agent 36 was obtained in the same manner except that the amount of benzyl benzoate added was altered to 11 parts by weight and the amount of N-benzylphthalimide added was altered to 22 parts

by weight in the terminal blocking finishing agent 4.

(Preparation of terminal blocking finishing agent 37)

[0088] The terminal blocking finishing agent 37 was obtained in the same manner except that the amount of benzyl benzoate added was altered to 3.0 parts by weight and the amount of N-benzylphthalimide added was altered to 6.0 parts by weight in the terminal blocking finishing agent 4.

(Preparation of terminal blocking finishing agent 38)

[0089] The terminal blocking finishing agent 38 was obtained in the same manner except that the amount of phenyl benzoate added was altered to 11 parts by weight and the amount of N-benzylphthalimide added was altered to 22 parts by weight in the terminal blocking finishing agent 8.

(Preparation of terminal blocking finishing agent 39)

[0090] The terminal blocking finishing agent 38 was obtained in the same manner except that the amount of phenyl benzoate added was altered to 3.0 parts by weight and the amount of N-benzylphthalimide added was altered to 6.0 parts by weight in the terminal blocking finishing agent 8.

(Preparation of terminal blocking finishing agent 40)

[0091] Twenty (20.0) parts by weight of bis (2, 6- diisopropylphenyl) carbodiimide (Stabaxol I LF; Rhein Chemie Japan Ltd.,), benzyl benzoate, N- butylphthalimide, and 20.0 parts by weight of carrier agent for polyester with sulfated castor oil as a major component (Huntsman Corporation UNIVADINE PB) were mixed, heated to 60°C and uniformly melted. The resultant was gradually added, while stirred by a propeller- type stirring machine, to 40.0 parts by weight of 70°C water for emulsified dispersion and allowed to cool to obtain the terminal blocking finishing agent 40.

[0092] Examples of processing using the terminal blocking finishing agent obtained by the above method will be shown below.

(Example 1) The PET woven fabric was immersed in a treatment solution in which the terminal blocking finishing agent 1 was used in an amount of 2% owf as the solid content of the terminal blocking agent at a liquor ratio of 1: 20 using a high pressure dyeing tester and was processed in conditions of 130°C for 30 minutes using UR-MINI-COLOR (infrared ray Mini-Color (manufactured by Texam Co., Ltd.)) while circulating the treatment solution. Subsequently, reduction cleaning was carried out in conditions of nonionic surfactant Gran-up US-20 (Sanyo Chemical Industries, Ltd.) 0.5 g/L, 30% (in concentration) aqueous sodium hydroxide solution 1.0 g/L and hydrosulfite 2.0 g/L at a liquor ratio of 1:20 at 80°C for 20 minutes. After centrifugal dehydration, drying was carried out in a pin tenter that was set at 130°C. Dry heat set was then carried out without changing the width of the fabric for one minute in the pin tenter that was set at 170°C. After the set, hydrolysis treatment was carried out in conditions of 130°C for 48 hours at a liquor ratio of 1:50 using UR·MINI-COLOR.

(Example 2) The same treatment as described in Example 1 was carried out except that the terminal blocking finishing agent 1 was altered to the terminal blocking finishing agent 2.

(Example 3) The same treatment as described in Example 1 was carried out except that the terminal blocking finishing agent 1 was altered to the terminal blocking finishing agent 3.

(Example 4) The same treatment as described in Example 1 was carried out except that the terminal blocking finishing agent 1 was altered to the terminal blocking finishing agent 4.

(Example 5) The same treatment as described in Example 1 was carried out except that the terminal blocking finishing agent 1 was altered to the terminal blocking finishing agent 5.

(Example 6) The same treatment as described in Example 1 was carried out except that the terminal blocking finishing agent 1 was altered to the terminal blocking finishing agent 6.

(Example 7) The same treatment as described in Example 1 was carried out except that the terminal blocking finishing agent 1 was altered to the terminal blocking finishing agent 7.

(Example 8) The same treatment as described in Example 1 was carried out except that the terminal blocking finishing agent 1 was altered to the terminal blocking finishing agent 8.

(Example 9) The same treatment as described in Example 1 was carried out except that the terminal blocking finishing agent 1 was altered to the terminal blocking finishing agent 9.

(Example 10) The same treatment as described in Example 1 was carried out except that the terminal blocking finishing agent 1 was altered to the terminal blocking finishing agent 10.

(Example 11) The same treatment as described in Example 1 was carried out except that the terminal blocking finishing agent 1 was altered to the terminal blocking finishing agent 11.
(Example 12) The same treatment as described in Example 1 was carried out except that the terminal blocking finishing agent 1 was altered to the terminal blocking finishing agent 12.
(Example 13) The same treatment as described in Example 1 was carried out except that the terminal blocking finishing agent 1 was altered to the terminal blocking finishing agent 13.
(Example 14) The same treatment as described in Example 1 was carried out except that the terminal blocking finishing agent 1 was altered to the terminal blocking finishing agent 14.
(Comparative example 1) The same treatment as described in Example 1 was carried out except that the terminal blocking finishing agent 1 was altered to the terminal blocking finishing agent 15.
(Comparative example 2) The same treatment as described in Example 1 was carried out except that the terminal blocking finishing agent 1 was altered to the terminal blocking finishing agent 16.
(Comparative example 3) The same treatment as described in Example 1 was carried out except that the terminal blocking finishing agent 1 was altered to the terminal blocking finishing agent 17.
(Comparative example 4) The same treatment as described in Example 1 was carried out except that the terminal blocking finishing agent 1 was altered to the terminal blocking finishing agent 18.
(Comparative example 5) The same treatment as described in Example 1 was carried out except that the terminal blocking finishing agent 1 was altered to the terminal blocking finishing agent 19.
(Comparative example 6) The same treatment as described in Example 1 was carried out except that the terminal blocking finishing agent 1 was altered to the terminal blocking finishing agent 20.
(Comparative example 7) The same treatment as described in Example 1 was carried out except that the terminal blocking finishing agent 1 was altered to the terminal blocking finishing agent 21.
(Comparative example 8) The same treatment as described in Example 1 was carried out except that the terminal blocking finishing agent 1 was altered to the terminal blocking finishing agent 22.
(Comparative example 9) The same treatment as described in Example 1 was carried out except that the terminal blocking finishing agent 1 was altered to the terminal blocking finishing agent 23.
(Comparative example 10) The same treatment as described in Example 1 was carried out except that the terminal blocking finishing agent 1 was altered to the terminal blocking finishing agent 24.
(Comparative example 11) The same treatment as described in Example 1 was carried out except that the terminal blocking finishing agent 1 was altered to the terminal blocking finishing agent 25.
(Comparative example 12) The same treatment as described in Example 1 was carried out except that the terminal blocking finishing agent 1 was altered to the terminal blocking finishing agent 26.
(Comparative example 13) The same treatment as described in Example 1 was carried out except that the terminal blocking finishing agent 1 was altered to the terminal blocking finishing agent 27.
(Comparative example 14) The same treatment as described in Example 1 was carried out except that the terminal blocking finishing agent 1 was altered to the terminal blocking finishing agent 28.
(Comparative example 15) The same treatment as described in Example 1 was carried out except that the terminal blocking finishing agent 1 was altered to the terminal blocking finishing agent 29.
(Comparative example 16) The same treatment as described in Example 1 was carried out except that the terminal blocking finishing agent 1 was altered to the terminal blocking finishing agent 30.
(Comparative example 17) The same treatment as described in Example 1 was carried out except that the terminal blocking finishing agent 1 was altered to the terminal blocking finishing agent 31.
(Comparative example 18) The same treatment as described in Example 1 was carried out except that the terminal blocking finishing agent 1 was altered to the terminal blocking finishing agent 32.
(Comparative example 19) The same treatment as described in Example 1 was carried out except that the terminal blocking finishing agent 1 was altered to the terminal blocking finishing agent 33.
(Comparative example 20) The same treatment as described in Example 1 was carried out except that the terminal blocking finishing agent 1 was altered to the terminal blocking finishing agent 34.
(Comparative example 21) The same treatment as described in Example 1 was carried out except that the terminal blocking finishing agent 1 was altered to the terminal blocking finishing agent 35.
(Comparative example 22) The same treatment as described in Example 1 was carried out except that the terminal blocking finishing agent 1 was altered to the terminal blocking finishing agent 36.
(Comparative example 23) The same treatment as described in Example 1 was carried out except that the terminal blocking finishing agent 1 was altered to the terminal blocking finishing agent 37.
(Comparative example 24) The same treatment as described in Example 1 was carried out except that the terminal blocking finishing agent 1 was altered to the terminal blocking finishing agent 38.
(Comparative example 25) The same treatment as described in Example 1 was carried out except that the terminal blocking finishing agent 1 was altered to the terminal blocking finishing agent 39.

(Reference example 1) The same treatment as described in Example 1 was carried out except that the terminal blocking finishing agent was not added.

As shown in Reference example 1, in cases where the terminal blocking treatment was not carried out, the strength of the fabric after the hydrolysis test markedly decreased so much that the strength could not be measured strength. In contrast, as shown in Examples 1 to 14, treatment with the terminal blocking finishing agent containing N-phthalimide and benzoate as essential components was found to be able to render good wet heat durability (Table 1).

Meanwhile, as shown in Comparative examples 1 to 9, single application of each of trichlorobenzene, methylnaphthalene, N-phthalimide and benzoate was able to render a certain level of wet heat durability, which was not as good as that in the case of combined use of N-phthalimide and benzoate, indicating that the combined use of N-phthalimide and benzoate developed an excellent effect (Table 2).

In addition, as shown Comparative examples 10 to 25, a particularly excellent effect was shown to be developed when the content ratio of the terminal blocking agent, N-phthalimide and benzoate was within a certain range (Table 3).

(Example 15) The PLA woven fabric was immersed in a treatment solution in which the terminal blocking finishing agent 1 was used in an amount of 2% owf as the solid content of the terminal blocking agent at a liquor ratio of 1:20 using a high pressure dyeing tester and was processed in conditions of 110°C for 30 minutes using UR-MINI-COLOR (infrared ray Mini-Color (manufactured by Texam Co., Ltd.)) while circulating the treatment solution. Subsequently, reduction cleaning was carried out in conditions of nonionic surfactant Gran-up US-20 (Sanyo Chemical Industries, Ltd.) 0.5 g/L, soda ash 1.5 g/L, and hydrosulfite 2.0 g/L at a liquor ratio of 1:20 at 60°C for 20 minutes. After centrifugal dehydration, drying was carried out in a pin tenter that was set at 110°C. Dry heat set was then carried out without changing the width of the fabric for one minute in the pin tenter that was set at 130°C. After the set, using a thermo-hygrostat tester THN064PB manufactured by Toyo Engineering Works, Ltd., a sample was placed in the thermo-hygrostat at 70°C and 90% RH and hydrolysis treatment was carried out for seven days.

(Example 16) The same treatment as described in Example 15 was carried out except that the treated fabric was altered to PLA/PTT woven fabric from PLA woven fabric and the temperature of the terminal blocking treatment was altered to 130°C for 30 minutes from 110°C for 30 minutes.

(Example 17) The same treatment as described in Example 1 was carried out except that the treated fabric was altered to PBT woven fabric from PET woven fabric.

(Example 18) The same treatment as described in Example 1 was carried out except that the treated fabric was altered to PET/C woven fabric from PET woven fabric. After the hydrolysis treatment was carried out, a weft was taken out from the woven fabric and the tensile strength of the weft was measured to calculate a strength retaining rate.

(Example 19) The same terminal blocking treatment and set as described in Example 1 were carried out in the same conditions as in Example 1 except that the treated fabric was altered to CDP knit from PET woven fabric. After the set, hydrolysis treatment was carried out in conditions of 130°C for 16 hours at a liquor ratio of 1:50 using UR·MINI-COLOR.

(Example 20) An A/C woven fabric was immersed in a treatment solution in which the terminal blocking finishing agent 1 was used in an amount of 2% owf as the solid content of the terminal blocking agent at a liquor ratio of 1:20 using a high pressure dyeing tester and was processed in conditions of 100°C for 30 minutes using UR-MINI-COLOR (infrared ray Mini-Color (manufactured by Texam Co., Ltd.)) while circulating the treatment solution. Subsequently, reduction cleaning was carried out in conditions of nonionic surfactant Gran-up US-20 (Sanyo Chemical Industries, Ltd.) 0.5 g/L, soda ash 1.5 g/L, and hydrosulfite 2.0 g/L at a liquor ratio of 1:20 at 60°C for 20 minutes. After centrifugal dehydration, drying was carried out in a pin tenter that was set at 110°C. After the drying, using a thermo-hygrostat tester THN064PB manufactured by Toyo Engineering Works, Ltd., a sample was placed in the thermo-hygrostat at 70°C and 90% RH and hydrolysis treatment was carried out for seven days.

(Comparative example 26) The same treatment as described in Example 15 was carried out except that the terminal blocking finishing agent 1 was altered to the terminal blocking finishing agent 16.

(Comparative example 27) The same treatment as described in Example 16 was carried out except that the terminal blocking finishing agent 1 was altered to the terminal blocking finishing agent 16.

(Comparative example 28) The same treatment as described in Example 17 was carried out except that the terminal blocking finishing agent 1 was altered to the terminal blocking finishing agent 16.

(Comparative example 29) The same treatment as described in Example 20 was carried out except that the terminal blocking finishing agent 1 was altered to the terminal blocking finishing agent 16.

(Reference example 2) The same treatment as described in Example 15 was carried out except that the terminal blocking finishing agent was not added.

(Reference example 3) The same treatment as described in Example 16 was carried out except that the terminal blocking finishing agent was not added.

(Reference example 4) The same treatment as described in Example 17 was carried out except that the terminal blocking finishing agent was not added.

(Reference example 5) The same treatment as described in Example 18 was carried out except that the terminal

blocking finishing agent was not added.

(Reference example 6) The same treatment as described in Example 19 was carried out except that the terminal blocking finishing agent was not added.

(Reference example 7) The same treatment as described in Example 20 was carried out except that the terminal blocking finishing agent was not added.

[0093] As shown in Reference examples 2 to 7, in cases where the terminal blocking treatment was not carried out, the strength of the fabric drastically decreased in the hydrolysis test. In contrast, as shown in Examples 15 to 20, carrying out the terminal blocking treatment was shown to allow good wet heat durability to be rendered (Table 4).

[0094] Meanwhile, as shown in Comparative examples 26 to 29, the use of methylnaphthalene as the carrier agent led to enhanced wet heat durability, which was found to not be as good as the synergistic effect of benzyl benzoate and N- butylphthalimide (Table 4) .

[0095] From the above results, it has been shown that the present invention has a beneficial effect on polyesters other than PET.

(Example 21) The PET fabric is immersed in the treatment solution below, squeezed out excess treatment solution using a mangle (pick up rate: 82%), dried for two minutes in a tenter that was set at 130°C, followed by heat treatment for another three minutes in a tenter that was set at 170°C. The PET fabric after the heat treatment was cleaned in warm water of 60°C containing a nonionic surfactant, Gran-up US-20 (Sanyo Chemical Industries, Ltd.) 0.5 g/L for 10 minutes. The PET fabric after the cleaning was dried for two minutes in a tenter that was set at 130°C. After the drying, the hydrolysis test was carried out by the same method as described in Example 1.

(Treatment solution)	Terminal blocking agent	:25 g/L
	Benzyl benzoate	:5.2 g/L
	N-butylphthalimide	:10.5 g/L
	Sulfated castor oil	:2.5 g/L

[0096] (Example 22) In Example 21, the PET fabric was altered to the PLA fabric; the drying temperature was altered to 110°C from 130°C and the temperature of the dry heat treatment was altered to 130°C from 170°C. The hydrolysis test was carried out by the same method as described in Example 15.

[0097] (Example 23) The same treatment as described in Example 22 was carried out except that the PLA fabric was altered to the PLA/PTT fabric.

[0098] (Example 24) The same treatment as described in Example 21 was carried out except that the PET fabric was altered to the PBT fabric.

[0099] (Example 25) The same treatment as described in Example 21 was carried out except that the PET fabric was altered to the PET/C fabric.

[0100] (Example 26) The same treatment as described in Example 22 was carried out except that the PLA fabric was altered to the A/C fabric.

[0101] (Example 27) The same treatment as described in Example 21 was carried out except that the heat treatment method was altered to steaming treatment by saturated steam of 102°C from the dry heat treatment.

[0102] (Example 28) The same treatment as described in Example 22 was carried out except that the heat treatment method was altered to steaming treatment by saturated steam of 102°C from the dry heat treatment.

[0103] (Example 29) The same treatment as described in Example 23 was carried out except that the heat treatment method was altered to steaming treatment by saturated steam of 102°C from the dry heat treatment.

[0104] (Example 30) The same treatment as described in Example 24 was carried out except that the heat treatment method was altered to steaming treatment by saturated steam of 102°C from the dry heat treatment.

[0105] (Example 31) The same treatment as described in Example 25 was carried out except that the heat treatment method was altered to steaming treatment by saturated steam of 102°C from the dry heat treatment.

[0106] (Example 32) The same treatment as described in Example 26 was carried out except that the heat treatment method was altered to steaming treatment by saturated steam of 102°C from the dry heat treatment.

[0107] As shown in Examples 21 to 26, the dry heat treatment was also found to be able to render good wet heat durability. In addition, as shown in Examples 27 to 32, the wet heat treatment was also found to be able to render good wet heat durability (Table 5).

[0108] (Example 33) The same treatment as described in Example 1 was carried out except that Dianix TuxedoBlack Fconc. liq was added in an amount of 8.0% owf in addition to the terminal blocking finishing agent 1.

[0109] (Example 34) The same treatment as described in Example 15 was carried out except that DENAPLA Black GS was added in an amount of 4.5% owf in addition to the terminal blocking finishing agent 1.

[0110] (Example 35) The same treatment as described in Example 16 was carried out except that DENAPLA Black

GS was added in an amount of 4.5% owf in addition to the terminal blocking finishing agent 1.

[0111] (Example 36) The same treatment as described in Example 17 was carried out except that Dianix TuxedoBlack Fconc. liq was added in an amount of 8.0% owf in addition to the terminal blocking finishing agent 1.

[0112] (Example 37) The PET woven fabric was, using a liquid flow dyeing machine, placed in a treatment solution in which the terminal blocking finishing agent 1 was used in an amount of 2% owf as solid content of the terminal blocking agent and further processed in conditions of 8.0% owf Dianix TuxedoBlack Fconc. liq at a liquor ratio 1: 25 at 130°C for 30 minutes while circulating the treatment solution. The clothing fabric placed was 30 m in length, which was processed at a clothing fabric rate of 42 m/ minute. Subsequently, reduction cleaning was carried out in conditions of nonionic surfactant Gran- up US- 20 (Sanyo Chemical Industries, Ltd.) 0.5 g/L, 30% (in concentration) aqueous sodium hydroxide solution 1.0 g/L and hydrosulfite 2.0 g/L at a liquor ratio of 1: 20 at 80°C for 20 minutes. After centrifugal dehydration, drying was carried out in a pin tenter that was set at 130°C. Dry heat set was then carried out without changing the width of the fabric for one minute in the pin tenter that was set at 170°C. After the set, hydrolysis treatment was carried out in conditions of 130°C for 48 hours at a liquor ratio of 1: 50 using UR·MINI- COLOR.

[0113] As shown in Examples 33 to 36, it was shown that, even when the dye was placed in a bath to process the dyeing and terminal blocking in the same bath, good wet heat durability could be rendered (Table 6).

[0114] In addition, as shown in Example 37, the long fabric was confirmed to have good wet heat durability when processed using the liquid flow dyeing machine. No problem such as listing or ending was observed (Table 6).

(Example 38) The same treatment as described in Example 33 was carried out except that the terminal blocking finishing agent 1 was altered to the terminal blocking finishing agent 40.

(Example 39) The same treatment as described in Example 34 was carried out except that the terminal blocking finishing agent 1 was altered to the terminal blocking finishing agent 40.

(Example 40) The same treatment as described in Example 35 was carried out except that the terminal blocking finishing agent 1 was altered to the terminal blocking finishing agent 40.

(Example 41) The same treatment as described in Example 36 was carried out except that the terminal blocking finishing agent 1 was altered to the terminal blocking finishing agent 40.

(Example 42) The same treatment as described in Example 37 was carried out except that the terminal blocking finishing agent 1 was altered to the terminal blocking finishing agent 40.

[0115] As shown in Examples 38 to 42, the fabric was confirmed to have good wet heat durability when processed with the terminal blocking finishing agent 40 (Table 7).

[0116]

[Table 1]

					Example 1	Example 2	Example 3	Example 4	Example 5	Example 6	Example 7
Conditions	Fabric treated				PET	PET	PET	PET	PET	PET	PET
	Treatment solution	Composition of treatment solution	Terminal blocking agent		TIC	TIC	TIC	TIC	TIC	TIC	TIC
			Carrier agent	Benzoate	Benzyl benzoate	Benzyl benzoate	Benzyl benzoate	Benzyl benzoate	Phenyl benzoate	Phenyl benzoate	Phenyl benzoate
				N-alkyl phthalimide	N-butyl phthalimide	N-ethyl phthalimide	N-propyl phthalimide	N-benzyl phthalimide	N-butyl phthalimide	N-ethyl phthalimide	N-propyl phthalimide
				Others							
			Emulsifying agent		Sulfated castor oil	Sulfated castor oil	Sulfated castor oil	Sulfated castor oil	Sulfated castor oil	Sulfated castor oil	Sulfated castor oil
		Concentration of agent in treatment solution (%owf)	Terminal blocking agent		2.0	2.0	2.0	2.0	2.0	2.0	2.0
			Carrier agent	Benzoate	0.5	0.5	0.5	0.5	0.5	0.5	0.5
				N-alkyl phthalimide	1.0	1.0	1.0	1.0	1.0	1.0	1.0
				Others							
			Emulsifying agent		0.2	0.2	0.2	0.2	0.2	0.2	0.2
	Treatment method				In a bath	In a bath	In a bath	In a bath	In a bath	In a bath	In a bath
Physical properties	At the time of completion of processing	Strength (cN/dtex)			3.40	3.42	3.41	3.39	3.38	3.39	3.41
		Concentration of terminal group (equivalent/10^3kg)			5.3	5.4	6.5	5.8	5.6	5.6	5.2
	After hydrolysis treatment	Method of hydrolysis test			(ii)	(ii)	(ii)	(ii)	(ii)	(ii)	(ii)
		Strength (cN/dtex)			2.73	2.74	2.75	2.73	2.69	2.71	2.76
		Concentration of terminal group (equivalent/10^3kg)			45.2	43.4	45.6	46.9	52.3	43.6	45.6
		Strength retaining rate (%)			80	80	81	81	80	80	81

					Example 8	Example 9	Example 10	Example 11	Example 12	Example 13	Example 14
Conditions	Fabric treated				PET	PET	PET	PET	PET	PET	PET
	Treatment solution	Composition of treatment solution	Terminal blocking agent		TIC	TIC	TIC	TIC	TIC	DIC	DCC
			Carrier agent	Benzoate	Phenyl benzoate	Butyl benzoate	Butyl benzoate	Butyl benzoate	Butyl benzoate	Benzyl benzoate	Benzyl benzoate
				N-alkyl phthalimide	N-benzyl phthalimide	N-butyl phthalimide	N-ethyl phthalimide	N-propyl phthalimide	N-benzyl phthalimide	N-butyl phthalimide	N-butyl phthalimide
				Others							
			Emulsifying agent		Sulfated castor oil	Sulfated castor oil	Sulfated castor oil	Sulfated castor oil	Sulfated castor oil	Sulfated castor oil	Sulfated castor oil
		Concentration of agent in treatment solution (%owf)	Terminal blocking agent		2.0	2.0	2.0	2.0	2.0	2.0	2.0
			Carrier agent	Benzoate	0.5	0.5	0.5	0.5	0.5	0.5	0.5
				N-alkyl phthalimide	1.0	1.0	1.0	1.0	1.0	1.0	1.0
				Others							
			Emulsifying agent		0.2	0.2	0.2	0.2	0.2	0.2	0.2
	Treatment method				In a bath	In a bath	In a bath	In a bath	In a bath	In a bath	In a bath
Physical properties	At the time of completion of processing	Strength (cN/dtex)			3.42	3.42	3.43	3.43	3.42	3.39	3.38
		Concentration of terminal group (equivalent/10^3kg)			5.6	5.8	5.6	5.6	5.6	5.2	5.4
	After hydrolysis treatment	Method of hydrolysis test			(ii)	(ii)	(ii)	(ii)	(ii)	(ii)	(ii)
		Strength (cN/dtex)			2.65	2.69	2.64	2.71	2.71	2.67	2.63
		Concentration of terminal group (equivalent/10^3kg)			43.4	43.1	42.7	39.5	47.5	46.4	44.4
		Strength retaining rate (%)			77	79	77	79	79	79	78

[0117]

[Table 2]

					Comparative example 1	Comparative example 2	Comparative example 3	Comparative example 4	Comparative example 5
Conditions	Fabric treated				PET	PET	PET	PET	PET
	Treatment solution	Composition of treatment solution	Terminal blocking agent		TIC	TIC	TIC	TIC	TIC
			Carrier agent	Benzoate			Benzyl benzoate	Phenyl benzoate	Butyl benzoate
				N-alkyl phthalimide					
				Others	Trichlorobenzene	Methylnaphthalene			
			Emulsifying agent		Sulfated castor oil	Sulfated castor oil	Sulfated castor oil	Sulfated castor oil	Sulfated castor oil
		Concentration of agent in treatment solution (%owf)	Terminal blocking agent		2.0	2.0	2.0	2.0	2.0
			Carrier agent	Benzoate			1.5	1.5	1.5
				N-alkyl phthalimide					
				Others	1.5	1.5			
			Emulsifying agent		0.2	0.2	0.2	0.2	0.2
	Treatment method				In a bath	In a bath	In a bath	In a bath	In a bath
Physical properties	At the time of completion of processing	Strength (cN/dtex)			3.39	3.38	3.39	3.39	3.41
		Concentration of terminal group (equivalent/10^3kg)			18.2	16.4	17.5	15.3	18.9
	After hydrolysis treatment	Method of hydrolysis test			(ii)	(ii)	(ii)	(ii)	(ii)
		Strength (cN/dtex)			1.64	1.45	1.52	1.49	1.32
		Concentration of terminal group (equivalent/10^3kg)			78.9	83.4	85.6	79.3	87.3
		Strength retaining rate (%)			48	43	45	44	39

					Comparative example 6	Comparative example 7	Comparative example 8	Comparative example 9
Conditions	Fabric treated				PET	PET	PET	PET
	Treatment solution	Composition of treatment solution	Terminal blocking agent		TIC	TIC	TIC	TIC
			Carrier agent	Benzoate				
				N-alkyl phthalimide	N-butyl phthalimide	N-ethyl phthalimide	N-propyl phthalimide	N-benzyl phthalimide
				Others				
			Emulsifying agent		Sulfated castor oil	Sulfated castor oil	Sulfated castor oil	Sulfated castor oil
		Concentration of agent in treatment solution (%owf)	Terminal blocking agent		2.0	2.0	2.0	2.0
			Carrier agent	Benzoate				
				N-alkyl phthalimide	1.5	1.5	1.5	1.5
				Others				
			Emulsifying agent		0.2	0.2	0.2	0.2
	Treatment method				In a bath	In a bath	In a bath	In a bath
Physical properties	At the time of completion of processing	Strength (cN/dtex)			3.42	3.42	3.42	3.42
		Concentration of terminal group (equivalent/10^3kg)			14.4	14.3	14.1	16.7
	After hydrolysis treatment	Method of hydrolysis test			(ii)	(ii)	(ii)	(ii)
		Strength (cN/dtex)			1.42	1.43	1.47	1.68
		Concentration of terminal group (equivalent/103kg)			86.4	72.3	74.4	74.3
		Strength retaining rate (%)			42	42	43	49

[0118]

[Table 3]

					Comparative example 10	Comparative example 11	Comparative example 12	Comparative example 13	Comparative example 14	Comparative example 15	Comparative example 16	Comparative example 17	Comparative example 18
Conditions	Fabric treated				PET	PET	PET	PET	PET	PET	PET	PET	PET
	Treatment solution	Composition of treatment solution	Terminal blocking agent		TIC	TIC	TIC	TIC	TIC	TIC	TIC	TIC	TIC
			Carrier agent	Benzoate	Benzyl benzoate	Benzyl benzoate	Phenyl benzoate	Phenyl benzoate	Benzyl benzoate	Benzyl benzoate	Phenyl benzoate	Phenyl benzoate	Benzyl benzoate
				N-alkyl phthalimide	N-butyl phthalimide	N-butyl phthalimide	N-butyl phthalimide	N-butyl phthalimide	N-benzyl phthalimide	N-benzyl phthalimide	N-benzyl phthalimide	N-benzyl phthalimide	N-butyl phthalimide
				Others									
			Emulsifying agent		Sulfated castor oil	Sulfated castor oil	Sulfated castor oil	Sulfated castor oil	Sulfated castor oil	Sulfated castor oil	Sulfated castor oil	Sulfated castor oil	Sulfated castor oil
		Concentration of agent in treatment solution (%owf)	Terminal blocking agent		2.0	2.0	2.0	2.0	2.0	2.0	2.0	2.0	2.0
			Carrier agent	Benzoate	0.1	0.9	0.1	0.9	0.1	0.9	0.1	0.9	1.1
				N-alkyl phthalimide	1.4	0.6	1.4	0.6	1.4	0.6	1.4	0.6	2.2
				Others									
			Emulsifying agent		0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.5
	Treatment method				In a bath	In a bath	In a bath	In a bath	In a bath	In a bath	In a bath	In a bath	In a bath
Physical properties	At the time of completion of processing	Strength (cN/dtex)			3.41	3.39	3.38	3.36	3.38	3.39	3.40	3.41	3.42
		Concentration of terminal group (equivalent/10^3kg)			16.9	16.5	16.4	17.4	15.8	16.4	14.6	15.3	20.8
	After hydrolysis treatment	Method of hydrolysis test			(ii)	(ii)	(ii)	(ii)	(ii)	(ii)	(ii)	(ii)	(ii)
		Strength (cN/dtex)			1.70	1.98	1.65	1.92	1.69	1.89	1.67	1.76	1.45
		Concentration of terminal group (equivalent/10^3kg)			74.5	72.5	76.1	74.6	73.9	69.5	70	71.2	74.5
		Strength retaining rate (%)			50	58	49	57	50	56	49	52	42

					Comparative example 19	Comparative example 20	Comparative example 21	Comparative example 22	Comparative example 23	Comparative example 24	Comparative example 25	Reference example 1
Conditions	Fabric treated				PET	PET	PET	PET	PET	PET	PET	PET
	Treatment solution	Composition of treatment solution	Terminal blocking agent		TIC	TIC	TIC	TIC	TIC	TIC	TIC	None
			Carrier agent	Benzoate	Benzyl benzoate	Phenyl benzoate	Phenyl benzoate	Benzyl benzoate	Benzyl benzoate	Phenyl benzoate	Phenyl benzoate	
				N-alkyl phthalimide	N-butyl phthalimide	N-butyl phthalimide	N-butyl phthalimide	N-benzyl phthalimide	N-benzyl phthalimide	N-benzyl phthalimide	N-benzyl phthalimide	
				Others								
			Emulsifying agent		Sulfated castor oil	Sulfated castor oil	Sulfated castor oil	Sulfated castor oil	Sulfated castor oil	Sulfated castor oil	Sulfated castor oil	
		Concentration of agent in treatment solution (%owf)	Terminal blocking agent		2.0	2.0	2.0	2.0	2.0	2.0	2.0	0
			Carrier agent	Benzoate	0.3	1.1	0.3	1.1	0.3	1.1	0.3	
				N-alkyl phthalimide	0.6	2.2	0.6	2.2	0.6	2.2	0.6	
				Others								
			Emulsifying agent		0.1	0.5	0.1	0.5	0.1	0.5	0.1	
	Treatment method				In a bath	In a bath	In a bath	In a bath	In a bath	In a bath	In a bath	In a bath
Physical properties	At the time of completion of processing	Strength (cN/dtex)			3.43	3.42	3.42	3.42	3.43	3.42	3.42	3.56
		Concentration of terminal group (equivalent/10^3kg)			22.3	21.3	22.4	23.4	21.3	23.4	23.4	29.8
	After hydrolysis treatment	Method of hydrolysis test			(ii)	(ii)	(ii)	(ii)	(ii)	(ii)	(ii)	(ii)
		Strength (cN/dtex)			1.79	1.23	1.34	1.34	1.45	1.39	1.45	0.63
		Concentration of terminal group (equivalent/10^3kg)			72.3	83.5	80.7	79.8	78.8	78.1	85.4	155.8
		Strength retaining rate (%)			52	36	39	39	42	41	42	18

[Table 4]

					Example 15	Example 16	Example 17	Example 18	Example 19	Example 20	Comparative example 26	Comparative example 27
Conditions	Fabric treated				PLA	PLA/PTT	PBT	PET/C	CDP	A/C	PLA	PLA/PTT
	Treatment solution	Composition of treatment solution	Terminal blocking agent		TIC	TIC	TIC	TIC	TIC	TIC	TIC	TIC
			Carrier agent	Benzoate	Benzyl benzoate	Benzyl benzoate	Benzyl benzoate	Benzyl benzoate	Benzyl benzoate	Benzyl benzoate		
				N-alkyl phthalimide	N-butyl phthalimide	N-butyl phthalimide	N-butyl phthalimide	N-butyl phthalimide	N-butyl phthalimide	N-butyl phthalimide		
				Others							Methyl-naphthalene	Methyl-naphthalene
			Emulsifying agent		Sulfated castor oil	Sulfated castor oil	Sulfated castor oil	Sulfated castor oil	Sulfated castor oil	Sulfated castor oil	Sulfated castor oil	Sulfated castor oil
		Concentration of agent in treatment solution (%owf)	Terminal blocking agent		2.0	2.0	2.0	2.0	2.0	2.0	2.0	2.0
			Carrier agent	Benzoate	0.5	0.5	0.5	0.5	0.5	0.5		
				N-alkyl phthalimide	1.0	1.0	1.0	1.0	1.0	1.0		
				Others							1.5	1.5
			Emulsifying agent		0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2
	Treatment method				In a bath	In a bath	In a bath	In a bath	In a bath	In a bath	In a bath	In a bath
Physical properties	At the time of completion of processing	Strength (cN/dtex)			2.53	2.51	2.91	3.39	2.45	1.64	2.49	2.61
		Molecular weight of polylactic acid portion (ten thousand)			12.3	12.5					11.9	12.8
		Concentration of terminal group (equivalent/10^3kg)			8.9	7.8	6.3	6.1			7.8	7.6
	After hydrolysis treatment	Method of hydrolysis test			(i)	(i)	(ii)	(ii)	(iii)	(i)	(i)	(i)
		Strength (cN/dtex)			2.29	2.48	2.45	2.55	1.49	1.59	1.43	1.89
		Molecular weight of polylactic acid portion (ten thousand)			11.9	12.1						
		Concentration of terminal group (equivalent/10^3kg)			14.6	12.4	38.5	48.0			45.7	32.2
		Strength retaining rate (%)			91	99	84	75	61	97	57	72

					Comparative example 28	Comparative example 29	Reference example 2	Reference example 3	Reference example 4	Reference example 5	Reference example 6	Reference example 7
Conditions	Fabric treated				PBT	A/C	PLA	PLA/PTT	PBT	PET/C	CDP	A/C
	Treatment solution	Composition of treatment solution	Terminal blocking agent		TIC	TIC	None	None	None	None	None	None
			Carrier agent	Benzoate								
				N-alkyl phthalimide								
				Others	Methyl-naphthalene	Methyl-naphthalene						
			Emulsifying agent		Sulfated castor oil	Sulfated castor oil						
		Concentration of agent in treatment solution (%owf)	Terminal blocking agent		2.0	2.0	0	0	0	0	0	0
			Carrier agent	Benzoate								
				N-alkyl phthalimide								
				Others	1.5	1.5						
			Emulsifying agent		0.2	0.2						
	Treatment method				In a bath	In a bath	In a bath	In a bath	In a bath	In a bath	In a bath	In a bath
Physical properties	At the time of completion of processing	Strength (cN/dtex)			2.98	1.71	2.43	2.56	2.89	3.31	2.45	1.72
		Molecular weight of polylactic acid portion (ten thousand)					11.9	12.2				
		Concentration of terminal group (equivalent/10^3kg)			6.9		23.4	28.9	28.9	28.5		
	After hydrolysis treatment	Method of hydrolysis test			(ii)	(i)	(i)	(i)	(ii)	(ii)	(iii)	(i)
		Strength (cN/dtex)			1.92	1.32	0.54	1.67	0.79	0.49	0.41	0.69
		Molecular weight of polylactic acid portion (ten thousand)					1.3	1.9				
		Concentration of terminal group (equivalent/10^3kg)			32.5		109.3	112.2	154.6	160.2		
		Strength retaining rate (%)			64	77	22	65	27	15	17	40

[Table 5]

					Example 21	Example 22	Example 23	Example 24	Example 25	Example 26
Conditions	Fabric treated				PET	PLA	PLA/PTT	PBT	PET/C	A/C
	Treatment solution	Composition of treatment solution	Terminal blocking agent		TIC	TIC	TIC	TIC	TIC	TIC
			Carrier agent	Benzoate	Benzyl benzoate	Benzyl benzoate	Benzyl benzoate	Benzyl benzoate	Benzyl benzoate	Benzyl benzoate
				N-alkyl phthalimide	N-butyl phthalimide	N-butyl phthalimide	N-butyl phthalimide	N-butyl phthalimide	N-butyl phthalimide	N-butyl phthalimide
				Others						
			Emulsifying agent		Sulfated castor oil	Sulfated castor oil	Sulfated castor oil	Sulfated castor oil	Sulfated castor oil	Sulfated castor oil
		Concentration of agent in treatment solution(g/L)	Terminal blocking agent		25.0	25.0	25.0	25.0	25.0	25.0
			Carrier agent	Benzoate	5.2	5.2	5.2	5.2	5.2	5.2
				N-alkyl phthalimide	10.5	10.5	10.5	10.5	10.5	10.5
				Others						
			Emulsifying agent		2.5	2.5	2.5	2.5	2.5	2.5
	Treatment method				Dry heat	Dry heat	Dry heat	Dry heat	Dry heat	Dry heat
Physical properties	At the time of completion of processing	Strength (cN/dtex)			3.42	2.49	2.62	2.93	3.37	1.62
		Molecular weight of polylactic acid portion (ten thousand)				12.2	12.1			
		Concentration of terminal group (equivalent/10^3kg)			7.6	9.4	8.5	8.9	9.3	
	After hydrolysis treatment	Method of hydrolysis test			(ii)	(i)	(i)	(ii)	(ii)	(i)
		Strength (cN/dtex)			2.39	2.19	2.32	2.21	1.87	1.39
		Molecular weight of polylactic acid portion (ten thousand)				10.8	10.3			
		Concentration of terminal group (equivalent/10^3kg)			50.2	16.8	16.8	21.7	58.2	
		Strength retaining rate (%)			70	88	89	75	55	86

					Example 27	Example 28	Example 29	Example 30	Example 31	Example 32
Conditions	Fabric treated				PET	PLA	PLA/PTT	PBT	PET/C	A/C
	Treatment solution	Composition of treatment solution	Terminal blocking agent		TIC	TIC	TIC	TIC	TIC	TIC
			Carrier agent	Benzoate	Benzyl benzoate	Benzyl benzoate	Benzyl benzoate	Benzyl benzoate	Benzyl benzoate	Benzyl benzoate
				N-alkyl phthalimide	N-butyl phthalimide	N-butyl phthalimide	N-butyl phthalimide	N-butyl phthalimide	N-butyl phthalimide	N-butyl phthalimide
				Others						
			Emulsifying agent		Sulfated castor oil	Sulfated castor oil	Sulfated castor oil	Sulfated castor oil	Sulfated castor oil	Sulfated castor oil
		Concentration of agent in treatment solution(g/L)	Terminal blocking agent		25.0	25.0	25.0	25.0	25.0	25.0
			Carrier agent	Benzoate	5.2	5.2	5.2	5.2	5.2	5.2
				N-alkyl phthalimide	10.5	10.5	10.5	10.5	10.5	10.5
				Others						
			Emulsifying agent		2.5	2.5	2.5	2.5	2.5	2.5
	Treatment method				Wet heat	Wet heat	Wet heat	Wet heat	Wet heat	Wet heat
Physical properties	At the time of completion of processing	Strength (cN/dtex)			3.45	2.42	2.53	2.82	3.42	1.65
		Molecular weight of polylactic acid portion (ten thousand)				12.1	12.5			
		Concentration of terminal group (equivalent/10^3kg)			6.2	8.9	8.2	7.8	8.6	
	After hydrolysis treatment	Method of hydrolysis test			(ii)	(i)	(i)	(ii)	(ii)	(i)
		Strength (cN/dtex)			2.59	2.31	2.41	2.42	2.53	1.42
		Molecular weight of polylactic acid portion (ten thousand)				11.9	12.1			
		Concentration of terminal group (equivalent/10^3kg)			45.3	14.6	12.4	38.5	51.4	
		Strength retaining rate (%)			75	95	95	86	74	86

[Table 6]

					Example 33	Example 34	Example 35	Example 36	Example 37
Conditions	Fabric treated				PET	PLA	PLA/PTT	PBT	PET
	Treatment solution	Composition of treatment solution	Terminal blocking agent		TIC	TIC	TIC	TIC	TIC
			Carrier agent	Benzoate	Benzyl benzoate	Benzyl benzoate	Benzyl benzoate	Benzyl benzoate	Benzyl benzoate
				N-alkyl phthalimide	N-butyl phthalimide	N-butyl phthalimide	N-butyl phthalimide	N-butyl phthalimide	N-butyl phthalimide
				Others					
			Emulsifying agent		Sulfated castor oil	Sulfated castor oil	Sulfated castor oil	Sulfated castor oil	Sulfated castor oil
			Dye		Dianix Tuxedo Black Fconc. Liq.	DENAPLA Black GS	DENAPLA Black GS	Dianix Tuxedo Black Fconc. Liq.	Dianix Tuxedo Black Fconc. Liq.
		Concentration of agent in treatment solution	Terminal blocking agent		2.0	2.0	2.0	2.0	2.0
			Carrier agent	Benzoate	0.5	0.5	0.5	0.5	0.5
				N-alkyl phthalimide	1.0	1.0	1.0	1.0	1.0
				Others					
			Emulsifying agent		0.2	0.2	0.2	0.2	0.2
			Dye		8.0	4.5	4.5	8.0	8.0
	Treatment method				In a bath	In a bath	In a bath	In a bath	In a bath
Physical properties	At the time of completion of processing	Strength (cN/dtex)			3.45	2.43	2.54	2.91	3.46
		Molecular weight of polylactic acid portion (ten thousand)				11.8	11.2		
		Concentration of terminal group (equivalent/10^3kg)			5.6	5.2	5.6	4.9	5.2
	After hydrolysis treatment	Method of hydrolysis test			(ii)	(i)	(i)	(ii)	(ii)
		Strength (cN/dtex)			2.78	2.21	2.34	2.46	2.81
		Molecular weight of polylactic acid portion (ten thousand)				10.8	12.1		
		Concentration of terminal group (equivalent/10^3kg)			42.4	18.9	15.6	39.0	40.4
		Strength retaining rate (%)			81	91	92	85	81

[0119]

[Table 7]

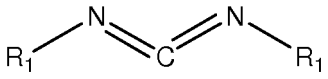
				Example 38	Example 39	Example 40	Example 41	Example 42
Conditions	Fabric treated			PET	PLA	PLA/PTT	PBT	A/C
	Treatment solution	Composition of treatment solution	Terminal blocking agent	TIC	TIC	TIC	TIC	TIC
			Carrier agent·Emulsifying agent	UNIVADINE PB	UNIVADINE PB	UNIVADINE PB	UNIVADINE PB	UNIVADINE PB
			Dye	Dianix Tuxedo Black Fconc. Liq.	DENAPLA Black GS	DENAPLA Black GS	Dianix Tuxedo Black Fconc. Liq.	None
		Concentration of agent in treatment solution	Terminal blocking agent	2.0	2.0	2.0	2.0	2.0
			Carrier agent·Emulsifying agent	2.0	2.0	2.0	2.0	2.0
			Dye	8.0	4.5	4.5	8.0	0
	Treatment method			In a bath	In a bath	In a bath	In a bath	In a bath
Physical properties	At the time of completion of processing	Strength (cN/dtex)		3.47	2.48	2.55	2.93	1.69
		Molecular weight of polylactic acid portion (ten thousand)			11.4	11.9		
		Concentration of terminal group (equivalent/10^3kg)		5.3	5.5	5.4	5.3	
	After hydrolysis treatment	Method of hydrolysis test		(ii)	(i)	(i)	(ii)	(i)
		Strength (cN/dtex)		2.81	2.19	2.36	2.46	1.46
		Molecular weight of polylactic acid portion (ten thousand)			10.3	10.7		
		Concentration of terminal group (equivalent/10^3kg)		41.4	23.4	19.8	36.8	
		Strength retaining rate (%)		81	88	93	84	86

Claims

1. A finishing agent for a polyester-based fiber structure, said finishing agent comprising a carbodiimide compound and a carrier agent emulsified or dispersed in water or a solvent, said carrier agent containing a benzoic acid compound and a phthalimide compound as essential components.

2. The finishing agent for a polyester-based fiber structure according to claim 1, wherein said carbodiimide compound is a compound represented by the general formula (I):

(Chemical formula 1)



wherein R1 represents one selected from an alkyl group with 1 to 20 carbon atoms, a cycloalkyl group with 5 to 12 carbon atoms, an aryl group with 6 to 20 carbon atoms, an allyl group and an aralkyl group with 7 to 20 carbon atoms.

3. The finishing agent for a polyester- based fiber structure according to claim 1 or 2, wherein said carbodiimide is at least one selected from N, N'- di- 2, 6- diisopropylphenyl carbodiimide, N, N'- di- cyclohexyl carbodiimide and N, N'- diisopropyl carbodiimide.

4. The finishing agent for a polyester-based fiber structure according to any of claims 1 to 3, wherein said carrier agent comprises benzyl benzoate and N-butyl phthalimide as said essential components.

5. A method of producing a polyester-based fiber structure, said method comprising taking up said finishing agent according to any of claims 1 to 4 into the inside of said polyester-based fiber structure.

6. A method of producing a polyester-based fiber structure, said method comprising, in the order mentioned, applying a treatment solution containing said finishing agent according to any of claims 1 to 4 to a polyester-based fiber(s); a drying step; and a heat treatment step.

7. A method of producing a polyester-based fiber structure, said method comprising supplying a polyester-based fiber

(s) into a treatment solution containing said finishing agent according to any of claims 1 to 4 to process the fiber(s) in a bath while circulating said treatment solution.

8. A method of producing a polyester-based fiber structure, said method comprising applying a treatment solution containing said finishing agent according to any of claims 1 to 4 to a polyester-based fiber(s); and then subjecting said polyester-based fiber(s) to a wet heat treatment.

9. A polyester-based fiber structure obtained by said method according to any of claims 5 to 8, wherein a concentration of said finishing agent becomes lower from an outer layer toward an inner layer in the cross-section of a single fiber of said polyester-based fiber(s).

INTERNATIONAL SEARCH REPORT

International application No.
PCT/JP2011/078857

A. CLASSIFICATION OF SUBJECT MATTER
D06M13/322(2006.01)i, *D06M13/188*(2006.01)i, *D06M101/32*(2006.01)n

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)
D06M13/00-15/715

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched
Jitsuyo Shinan Koho 1922-1996 Jitsuyo Shinan Toroku Koho 1996-2012
Kokai Jitsuyo Shinan Koho 1971-2012 Toroku Jitsuyo Shinan Koho 1994-2012

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.
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Y A	JP 2009-263840 A (Toray Industries, Inc.), 12 November 2009 (12.11.2009), claims; paragraphs [0033] to [0035], [0044] & US 2011/0015310 A1 & EP 2199456 A1 & WO 2009/041518 A1	1-3,5-9 4
A	JP 2010-174219 A (Teijin Ltd.), 12 August 2010 (12.08.2010), paragraphs [0067] to [0072], [0078] (Family: none)	1-9

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

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Date of the actual completion of the international search	Date of mailing of the international search report
17 January, 2012 (17.01.12)	24 January, 2012 (24.01.12)
Name and mailing address of the ISA/ Japanese Patent Office Facsimile No.	Authorized officer Telephone No.

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INTERNATIONAL SEARCH REPORT

International application No.
PCT/JP2011/078857

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
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A	JP 2010-203022 A (Toray Industries, Inc.), 16 September 2010 (16.09.2010), claims; paragraphs [0036] to [0058] (Family: none)	1-9
A	JP 2009-52150 A (Toray Industries, Inc.), 12 March 2009 (12.03.2009), paragraphs [0044] to [0050] (Family: none)	1-9

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REFERENCES CITED IN THE DESCRIPTION

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- JP 2001098459 A **[0006] [0007]**
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- JP 2007169856 A **[0050]**