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(54) FIBER-TREATING AGENT

(57) The present invention discloses a fiber treatment agent containing an ester compound represented by the formula (1):

$$R^1\text{-O-}((EO)_l\text{-}(PO)_m)\text{-}(EO)_n\text{-CO-}R^2 \qquad (1)$$

wherein, R^1 represents a hydrocarbon group containing 8 to 24 carbon atoms; R^2 represents an alkyl, alkenyl or cycloalkyl group having 7 to 23 carbon atoms; EO represents an ethyleneoxy group; PO represents a propyleneoxy group; l and n each represent an average reacted mole number of ethyleneoxy groups, 1 ranging from 0 to 15 and n ranging from 1 to 20; m represents an average reacted mole number of propyleneoxy groups, ranging from 0.5 to 20; and $((EO)_l\text{-}(PO)_m)$ and $(EO)_n$ are arranged in block in this order, while $(EO)_l$ and $(PO)_m$ in $((EO)_l\text{-}(PO)_m)$ may be arranged at random or in block.

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Description

Field of the invention

[0001] The present invention relates to a fiber treatment agent, and particularly to a fiber treatment agent used in steps of producing and processing fibers with a thermal treatment.

Background of the invention

[0002] Fiber manufacturing uses fiber treatment agents such as for imparting smoothness, a focusing property, and an antistatic property according to a purpose to facilitate spinning and/or drawing fibers. For example, there are conventionally known lubricating components for fibers including paraffin hydrocarbons such as mineral oils, oleyl oleate, dioleyl adipate, and dioleyl dipropionate.

[0003] In recent years, the spinning speed has been increasing. Therefore, there is a demand for a fiber treatment agent that reduces the occurrence of a broken fiber, a fluff, or the like in a step of spinning and has good heat resistance causing little decomposition and small fuming.

[0004] With respect to a lubricating component for fibers, JP- B 47- 29474 discloses an ester of a bisphenol- alkylene oxide adduct as a lubricating component that has good heat resistance causing little decomposition and small fuming. JP- A 06- 228866 discloses a technology using an ester compound of 1, 2, 3, 4- butenetetracarboxylic acid as a fiber treatment agent having good heat resistance. JP- A 10- 245781 discloses a technology using an ester compound having a phenol moiety and an ester of thiodipropionic acid together as a fiber treatment agent having a good heat resistance.

[0005] JP- A 6- 010264 discloses a method for sizing carbon fibers, including treating carbon fibers with an aqueous solution containing an ester compound and a nonionic surfactant.

JP- A 10- 251974 discloses a synthetic fiber treatment agent containing two compounds.

Summary of the invention

[0006] The present invention provides a fiber treatment agent containing an ester compound represented by the formula (1) :

$$R^1\text{- O- }((EO)_l\text{- }(PO)_m)\text{- }(EO)_n\text{- CO- }R^2 \qquad (1)$$

wherein, R^1 represents a hydrocarbon group having 8 to 24 carbon atoms; R^2 represents an alkyl group, an alkenyl group or a cycloalkyl group, having 7 to 23 carbon atoms; EO represents an ethyleneoxy group; PO represents a propyleneoxy group; 1 and n each represent an average reacted mole number of ethyleneoxy groups, l ranging from 0 to 15 and n ranging from 1 to 20; m represents an average reacted mole number of propyleneoxy groups, ranging from 0.5 to 20; and $((EO)_l\text{- }(PO)_m)$ and $(EO)_n$ are arranged in block in this order, while $(EO)_l$ and $(PO)_m$ in $((EO)_l\text{- }(PO)_m)$ may be arranged at random or in block.

The present invention also provides use of the ester compound as a fiber treatment agent in steps of producing, processing and spinning fiber with a thermal treatment, and a method for treating fibers using the ester compound.

Detailed description of the invention

[0007] An aqueous fiber treatment agent is required to have enough stability in an emulsion state for uniform attachment of a lubricating component on fibers. With regard to this, as described in JP-A 6-228866, an emulsifying component (e.g., a surfactant) may be a cause of a broken fiber due to hydrolytic thickening or of a fuming phenomenon due to low heat resistance of the emulsifying component itself. There is thus a demand for a treating agent that can form an emulsion having good stability with a small amount of emulsifying component. In addition, a fiber treatment agent containing a lubricating component having a high melting point reduces lubricating performance, stability, and/or handling property. There is thus a demand for a fiber treatment agent containing a lubricating component having a low melting point and being in the liquid state at an ambient temperature (20°C)

[0008] However, none of conventional fiber treatment agents including those described in JP- B 47- 29474, JP- A 6- 228866, and JP- A 10- 245781 can fulfill all these demands.

[0009] Therefore, the present invention provides a fiber treatment agent that contains a lubricating component with a low melting point, is easy to handle, and has all good properties in lubricating performance, heat resistance, and emulsion stability in an aqueous system.

[0010] The present inventors have extensively investigated for solving the above-mentioned problems, and found that an ester compound produced by reacting an alkylene oxide to an alcohol having a specific chain length in a specified manner and reacting with a fatty acid can solve these problems by being used as a lubricating component. The present invention thus has been accomplished.

[0011] According to the present invention, a fiber treatment agent is provided, that contains a lubricating component with a low melting point and is in the liquid state at an ambient temperature, is easy to handle, and has all good properties in lubricating performance, heat resistance, and emulsion stability in an aqueous system. The fiber treatment agent is easy to handle at a low temperature, can coat a fiber yarn uniformly, and is easy to remove in scouring and washing before a step of staining or the like, and therefore contributes significantly to facilitating a producing process of fibers.

[0012] The fiber treatment agent of the present invention contains the ester compound represented by the formula (1).

[0013] In the formula (1), R^1 represents a hydrocarbon group having 8 to 24 carbon atoms, preferably 10 to 22 carbon atoms, and more preferably 12 to 18 carbon atoms. Examples of the hydrocarbon group for R^1 include linear and branched alkyl and alkenyl groups and aromatic hydrocarbon groups that may be substituted with a linear or branched alkyl or alkenyl group. From the viewpoint of lubricating performance in high-speed winding, R^1 preferably represents a linear primary alkyl or alkenyl group, and more preferably a linear primary alkyl group. From the viewpoints of lubricating performance and environment safety, R^1 preferably represents a residual group of a natural higher alcohol such as octyl alcohol, decyl alcohol, lauryl alcohol, myristyl alcohol, cetyl alcohol, stearyl alcohol, eicosyl alcohol, behenyl alcohol or oleyl alcohol by removing a hydroxy group therefrom. Particularly from the viewpoint of lubricating performance in high-speed winding, R^1 more preferably represents a residual group of an alcohol selected from lauryl alcohol, myristyl alcohol, cetyl alcohol, stearyl alcohol, eicosyl alcohol, behenyl alcohol and oleyl alcohol by removing a hydroxy group therefrom, even more preferably represents a residual group of an alcohol selected from lauryl alcohol, myristyl alcohol, cetyl alcohol, stearyl alcohol and oleyl alcohol by removing a hydroxy group therefrom, and still even more preferably represents a residual group of an alcohol selected from lauryl alcohol, myristyl alcohol, cetyl alcohol and stearyl alcohol by removing a hydroxy group therefrom.

[0014] In the formula (1), R^2 represents an alkyl, alkenyl or cycloalkyl group having 7 to 23 carbon atoms, preferably a linear alkyl or alkenyl group having 7 to 23 carbon atoms, more preferably a linear alkyl or alkenyl group having 9 to 21 carbon atoms, and even more preferably a linear alkyl or alkenyl group having 11 to 17 carbon atoms. Specific examples of preferred R^2 include residual groups of linear and branched, saturated and unsaturated fatty acids by removing a carboxy group. Particularly from the viewpoint of lubricating performance, R^2 preferably represents a residual group of a fatty acid selected from octanoic acid, decanoic acid, lauric acid, myristic acid, palmitic acid, stearic acid, arachidic acid, behenic acid, palmitoleic acid, petroselinic acid, oleic acid, elaidic acid, linoleic acid, linolenic acid, erucic acid, isopalmitic acid (2-hexyldecanoic acid), 2-heptylundecanoic acid, citronellic acid, and isostearic acid by removing a carboxy group, more preferably represents a residual group of a fatty acid selected from decanoic acid, lauric acid, myristic acid, palmitic acid, stearic acid, arachic acid, behenic acid, palmitoleic acid, petroselinic acid, oleic acid, elaidic acid, and erucic acid by removing a carboxy group, and even more preferably represents a residual group of a fatty acid selected from lauric, myristic, palmitic, stearic, and oleic acids by removing a carboxy group.

[0015] In the formula (1), l represents the number ranging from 0 to 15, preferably 0 to 5, more preferably 0 to 3, and from the viewpoint of lubricating performance, even more preferably being equal to 0.

[0016] In the formula (1), n represent the number ranging from 1 to 20, preferably 2 to 15, more preferably 3 to 12, and from the viewpoint of emulsion stability, even more preferably 4 to 10.

[0017] In the formula (1), m represent the number ranging from 0.5 to 20, preferably 0.8 to 15, more preferably 1 to 10, and from the viewpoint of lubricating performance, even more preferably 1.2 to 8.

[0018] In the formula (1), $(EO)_l$ and $(PO)_m$ in $((EO)_l\text{-}(PO)_m)$ may be arranged at random or in block. For achieving a low melting point, $(EO)_l$ and $(PO)_m$ are preferably arranged in block.

[0019] For achieving the effects of the present invention, a key feature is an arrangement of $((EO)_l\text{-}(PO)_m)$ and $(EO)_n$ in block in this order in the formula (1) . The arrangement of $((EO)_l\text{-}(PO)_m)$ and $(EO)_n$ in block in this order makes the ester compound represented by the formula (1) have a low temperature and contribute to improving easiness in handling. Further, the fiber treatment agent containing the ester compound having the arrangement can have all good properties in lubricating performance, heat resistance, and emulsion stability in an aqueous system.

[0020] The ester compound represented by the formula (1) can be obtained, for example, by the following method. Or the ester compound represented by the formula (1) is preferably produced by the following steps (I) to (III):

step (I): reacting an EO/PO mixture to one or more alcohols collectively represented by the formula R^1-OH (2) (wherein, R^1 has the same meanings as above);
step (II): reacting EO to the resulting alkoxylate; and
step (III): esterifying the resulting alkoxylate.

[0021] Specifically, the method is conducted as follows. One or more alcohols collectively represented by the formula (2) :

R^1- OH (2)

(wherein, R^1 has the same meanings as above) are mixed with a known acid or base catalyst, and if needed, dehydrated at 20 to 140°C under reduced pressure. With the alcohol is reacted (1) propylene oxide alone, (2) ethylene oxide and then propylene oxide, or (3) ethylene oxide and propylene oxide randomly, and then reacted ethylene oxide to give a corresponding alkylene oxide adduct. The addition reaction of an alkylene oxide is preferably conducted by introducing the alkylene oxide in a predetermined amount and reacting until a pressure is decreased to a constant level (aging operation) at a temperature of 80 to 180°C. After obtaining the alkylene oxide adduct, the catalyst is neutralized if needed, and removed by filtration with the aid of an alkali adsorbent if additionally needed. The resultant alkylene oxide adduct is subjected to esterification or transesterification with one or more fatty acids or fatty acid esters collectively represented by the formula (3) :

R^2- COOM (3)

(wherein, R^2 has the same meanings as above; and M represents a hydrogen atom or an alkyl group having 1 to 3 carbon atoms) by a standard method in the presence of a known catalyst such as a metal, acid or base catalyst if needed to obtain ester compound (s) represented by the formula (1), which may be purified by removing the catalyst with an adsorbent or a steam treatment if needed.

[0022] The fiber treatment agent of the present invention may be in the form of liquid oily agent containing the ester compound represented by the formula (1) [hereinafter, referred to as ester compound (1)], or aqueous emulsion prepared by emulsifying the ester compound (1) in an aqueous medium selected from water and mixtures of water with water-miscible organic solvents (e.g., ethanol and isopropanol). The ester compound (1) has a tendency of self-emulsifying in an aqueous medium, and thus can reduce other emulsifying agent used together and can prevent separation of other functional agents to provide uniform coating on fibers. Considering the tendency and easiness in controlling an amount of the fiber treatment agent coating on fivers, the fiber treatment agent is preferably used in the form of aqueous emulsion to treat fibers.

[0023] For imparting sufficient smoothness to fibers, the fiber treatment agent of the present invention preferably contains the ester compound (1) in an amount of 3 to 70% by mass, more preferably 4 to 50% by mass, and even more preferably 5 to 25% by mass.

[0024] The fiber treatment agent of the present invention can preferably further contain one or more components selected from emulsifying component and other functional agents.

[0025] Known emulsifying components can be adequately used, including nonionic surfactants, anionic surfactants, and nitrogen-containing compounds. A known nonionic surfactant can be used without limitation. Examples of the nonionic surfactant include hardened castor oil-alkylene oxide adducts, esters of higher alcohols, alkylphenols, and polyhydric alcohols, and alkylene oxide adducts of these esters. A known anionic surfactant can be used without limitation. Examples of the anionic surfactant include fatty acid salts such as stearates, oleates, and erucates; alkylsulfonate salts, alkylbenzenesulfonate salts, alkylsulfate ester salts, and alkylphosphate ester salts. Examples of the nitrogen-containing compound include quaternary alkylammonium salts, alkylhydroxyethylammonium salts, cationic surfactants such as imidazolinium salts, and amphoteric surfactants such as aliphatic alkanolamides, aliphatic amine-ethylene oxide adducts, and alkyl betaines.

[0026] The fiber treatment agent containing the ester compound (1) of the present invention can form a stable emulsion with a less amount of emulsifying component than usual due to a self-emulsifying property of the ester compound (1). In other words, a content of the emulsifying component in the fiber treatment agent of the present invention can be adjusted within the range of 0 to 50% by mass, and preferably 0.1 to 20% by mass. Even in cases of lowering the content of the emulsifying component to, for example, the range of 0.1 to 5% by mass, particularly to the range of 0.1 to 3% by mass, and more particularly to the range of 0.1 to 2% by mass, the fiber treatment agent of the present invention can form an emulsion having good stability.

[0027] Other function-imparting agents to use suitably with the fiber treatment agent of the present invention are common components used in a fiber treatment agent, including, for example, lubricants such as ester oils, such as isostearyl isostearate, isostearyl adipate and trimethylolpropane trilaurate, and mineral oils, such as liquid paraffin; adhesives such as unmodified, ether-modified, amino-modified, epoxy-modified or glycol-modified silicon resins, polyacrylic esters, polyvinyl alcohols, terminal esterified or etherified adducts of bisphenol A with ethylene oxide; extreme pressure additives such as alkylamines having 8 to 22 carbon atoms and ethylene oxide adducts thereof; anticorrosives; preservatives, and antioxidants. A content of such other functional agent can be adequately determined according to a purpose within the range that does not impair the effects of the present invention.

[0028] Fiber can be coated (lubricated) with the fiber treatment agent of the present invention by a known method, without limitation, such as dipping and spraying. For dipping, fiber is preferably treated in a solution containing the fiber treatment agent at a concentration of 1 to 150 g/L as the ester compound represented by the formula (1).

[0029] For imparting sufficient smoothness to fibers, fibers are preferably coated with the fiber treatment agent in an amount of 0.02 to 5 parts by mass, more preferably 0.1 to 3 parts by mass, and even more preferably 0.3 to 2 parts by mass, as the ester compound (1), to 100 parts by mass of fibers.

[0030] Examples of fibers to which the fiber treatment agent of the present invention is applicable include synthetic fibers such as polyester, polyamide, polyacrylic, polypropylene, polyvinyl alcohol, poly(vinyl chloride), poly(vinylidene chloride) and polyurethane fibers, semisynthetic fibers such as acetate fibers, blended yarns of synthetic or semisynthetic fibers and natural fibers such as cotton, linen, rayon, and wool, and modified synthetic fibers containing inorganic compounds such as titania, zirconia, alumina, silica, zeolite in a dispersed state. These fibers may contain a delustering agent, a fluorescent brightening agent, an antistatic agent, and/or a flame retardant. Among these fibers, synthetic fibers receive much benefit from the fiber treatment agent of the present invention, because synthetic fibers are generally subjected to a heat treatment at high temperature. The fiber treatment agent of the present invention is also applicable to materials such as cotton, linen fabric, and woolen goods treated with conventional lubricants.

[0031] The fiber treatment agent of the present invention can be applied at any timing after a spun yarn is solidified. In general, the fiber treatment agent of the present invention is applied onto a yarn at a point before a taking-off roller. The fiber treatment agent of the present invention can coat a yarn uniformly when applied, and can be easily washed off in scouring and washing before a staining step or the like.

[0032] The resultant yarn can be used in woven and non-woven fabrics for industrial materials such as clothing fabric, adhesive tape-reinforcing fabric, covering material of cable, fabric for a tent, and tire cord.

[0033] The fiber treatment agent of the present invention can be suitably used as a lubricant for synthetic fiber in high-speed spinning or for tire cord, or may be used as a base oil for lubricating oil used in drawing, direct spin drawing, and spinning fiber to manufacture a fabric, coning oils, and lubricating oil used in entangling fiber to form a fabric. In this case, the fiber treatment agent of the present invention can be blended with appropriate additive(s) to provide a composition.

[0034] The present invention includes the following aspects:

aspect 1. a fiber treatment agent containing an ester compound represented by the formula (1) :

$$R^1\text{- O- }((EO)_l\text{- }(PO)_m)\text{- }(EO)_n\text{- CO- }R^2 \qquad (1)$$

wherein, R^1 represents a hydrocarbon group having 8 to 24 carbon atoms; R^2 represents an alkyl, alkenyl or cycloalkyl group having 7 to 23 carbon atoms; EO represents an ethyleneoxy group; PO represents a propyleneoxy group; l and n each represent an average reacted mole number of ethyleneoxy groups, l ranging from 0 to 15 and n ranging from 1 to 20; m represents an average number of reacted propyleneoxy groups, ranging from 0.5 to 20; and $((EO)_l\text{- }(PO)_m)$ and $(EO)_n$ are arranged in block in this order, while $(EO)_l$ and $(PO)_m$ in $((EO)_l\text{- }(PO)_m)$ may be arranged at random or in block;

aspect 2. the fiber treatment agent according to aspect 1, wherein R^1 in the formula (1) represents a linear primary alkyl or alkenyl group having 8 to 24 carbon atoms, more preferably having 12 to 18 carbon atoms, more preferably a residual group of a natural higher alcohol selected from the group containing octyl alcohol, decyl alcohol, lauryl alcohol, myristyl alcohol, cetyl alcohol, stearyl alcohol, eicosyl alcohol, behenyl alcohol, and oleyl alcohol by removing a hydroxy group therefrom, and even more preferably a residual group of a natural higher alcohol selected from the group containing stearyl alcohol and oleyl alcohol by removing a hydroxy group therefrom;

aspect 3. the fiber treatment agent according to aspect 1 or 2, wherein R^2 in the formula (1) represents a linear alkyl or alkenyl group having 7 to 23 carbon atoms, preferably having 11 to 17 carbon atoms, more preferably a residual group of a fatty acid selected from the group containing octanoic acid, decanoic acid, lauric acid, myristic acid, palmitic acid, stearic acid, arachidic acid, behenic acid, palmitoleic acid, petroselinic acid, oleic acid, elaidic acid, linoleic acid, linolenic acid, erucic acid, isopalmitic (2- hexyldecanoic) acid, 2- heptylundecanoic acid, citronellic acid, and isostearic acid by removing a carboxy group therefrom, and even more preferably a residual group of a fatty acid selected from the group containing lauric acid, stearic acid, and oleic acid by removing a carboxy group therefrom;

aspect 4. the fiber treatment agent according to any one of aspects 1 to 3, wherein l in the formula (1) preferably ranges from 0 to 5, more preferably 0 to 3, and even more preferably is equal to 0;

aspect 5. the fiber treatment agent according to any one of aspects 1 to 4, wherein m in the formula (1) preferably ranges from 0.8 to 15, more preferably 1 to 10, and even more preferably 1.2 to 8;

aspect 6. the fiber treatment agent according to any one of aspects 1 or 5, wherein n in the formula (1) preferably ranges from 2 to 15, more preferably 3 to 12, and even more preferably 4 to 10;

aspect 7.the fiber treatment agent according to any one of aspects 1 to 6, wherein $(EO)_l$ and $(PO)_m$ in $((EO)_l\text{- }(PO)_m)$ in the formula (1) are arranged in block;

aspect 8. the fiber treatment agent according to any one of aspects 1 to 7, further containing an emulsifying component;

aspect 9. the fiber treatment agent according to aspect 8, wherein a content of the emulsifying component is 0 to 50% by mass, preferably 0.1 to 20% by mass, and more preferably 0.1 to 5% by mass of the fiber treatment agent;
aspect 10. the fiber treatment agent according to any one of aspect 8 or 9, wherein a proportion of the emulsifying component to the ester compound represented by the formula (1) in the fiber treatment agent is 0.4 to 10% by mass;
aspect 11. the fiber treatment agent according to any one of aspects 1 to 10, wherein a content of the ester compound represented by the formula (1) is 3 to 70% by mass, and the fiber treatment agent further contains water;
aspect 12. the fiber treatment agent according to any one of aspects 1 to 11, which is for synthetic fibers;
aspect 13. the fiber treatment agent according to any one of aspects 1 to 12, which is produced through steps (I) to (III);
step (I): reacting an EO/PO mixture to one or two or more alcohols collectively represented by the formula R^1-OH (2) (wherein, R^1 has the same meanings as above);
step (II): reacting EO to the resulting alkoxylate; and
step (III): esterifying the resulting alkoxylate;
aspect 14. a method for treating fibers, including coating fibers with the fiber treatment agent according to any one of aspects 1 or 13 in an amount of 0.02 to 5 parts by mass, preferably 0.1 to 3 parts by mass, and more preferably 0.3 to 2 parts by mass, as the ester compound represented by the formula (1), to 100 parts by mass of fibers, and then drawing and rolling fibers;
aspect 15. the method for treating fibers according to aspect 15, wherein fibers are treated in a solution of the fiber treatment agent at a concentration of 1 to 150 g/L as the ester compound represented by the formula (1); and
aspect 16. use of the ester compound according to any one of aspects 1 or 13 as a fiber treatment agent in steps of producing, processing and spinning fiber with a thermal treatment.

Examples

[0035] The following Examples demonstrate the present invention. Examples are intended to illustrate the present invention and not to limit the present invention.
The present invention will be described in detail by Examples. Ester compounds used in Examples were prepared according to following procedure. In the following description, unless otherwise cited, "%" refers to "% by mass".

Preparation Example 1: Preparation of an ester compound (A)

[0036] In an autoclave equipped with a stirring device, a temperature controller, and an automatic feeder, 1082 g (4.0 mol) of starting stearyl alcohol (Kalcol 8098, Kao Corporation) and 3.2 g (0.08 mol) of potassium hydroxide were fed and dehydrated for 30 minutes at 110°C under 1.3 kPa. The inner atmosphere of the autoclave was replaced with nitrogen. Then, for addition reaction of propylene oxide, it was heated at an increased temperature of 125°C. Into the reaction mixture was introduced 580.8 g (10.0 mol) of propylene oxide and subjected to the aging operation. Next, for reacting ethylene oxide, it was heated at a further increased temperature of 150°C. Into the reaction mixture was introduced 881 g (20 mol) of ethylene oxide and subjected to the aging operation. After these reactions completed, the reaction mixture was cooled to 80°C. Then, to the autoclave was added 9.6 g of 90% lactic acid. The mixture was stirred for 30 minutes at 80°C and extracted to obtain an alkylene oxide adduct. A hydroxyl value of the product ensured that the moles of propylene oxide and ethylene oxide, reacted with the alcohol, were equal to the moles introduced. In addition, a residual pressure during each addition reaction of propylene oxide and ethylene oxide reached to a constant value nearly equal to 0 to ensure exhaustion of propylene oxide and ethylene oxide. According to these conditions, the product indicated a block arrangement in its structure.
Next, in a four-neck flask equipped with a stirring device, a temperature controller, and a distilling trap, 636 g (1 mol) of the alkylene oxide adduct, 282.5 g of oleic acid (Lunac OLL-V, Kao Corporation), and 0.18 g of tin monoxide (0.02% by mass to the total amount of ingredients used) as a catalyst were reacted for 10 hours at 170 to 240°C for esterification. The esterified product was treated with activated charcoal (Carboraffin, Takeda Pharmaceutical Company Limited.) and activated clay each in the amount corresponding to 0.4% by mass for 2 hours with stirring at 90°C under 5.33 kPa, and filtered to obtain an ester compound. A rate of esterification was 97% (from the starting fatty acid). Structural features of the ester compound are shown in Table 1.

Preparation Examples 2 to 18: Preparation of ester compounds (B) to (K) and comparative ester compounds (a) to (g)

[0037] Ester compounds (B) to (K) and comparative ester compounds (a) to (g) were prepared in the same reactor, operations, and conditions as in Preparation Example 1, except for reacting an average l moles of ethylene oxide, an average m moles of propylene oxide, and an average n moles of ethylene oxide in this order to starting alcohols shown in Table 1, respectively, under the same conditions as in Preparation Example 1 and then esterifying the resultant with respective fatty acids shown in Table 1. Respective rates of esterification (from the starting fatty acid) and structural features of ester compounds are shown in Table 1.

[0038]

[Table 1]

Production example	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18
Ester compound	(A)	(B)	(C)	(D)	(E)	(F)	(G)	(H)	(I)	(J)	(K)	(a)	(b)	(c)	(d)	(e)	(f)	(g)
Starting alcohol (R^1-OH)	Stearyl alcohol	Stearyl alcohol	Stearyl alcohol	Stearyl alcohol	Stearyl alcohol	Lauryl alcohol	Stearyl alcohol	Stearyl alcohol	Oleyl alcohol	Stearyl alcohol	Stearyl alcohol	Stearyl alcohol	Oleyl alcohol	Oleyl alcohol	Stearyl alcohol	Stearyl alcohol	Stearyl alcohol	Stearyl alcohol
Starting fatty acid (R^2-COOH)	Oleic acid	Oleic acid	Oleic acid	Oleic acid	Oleic acid	Oleic acid	Lauric acid	Stearyl acid	Oleic acid	Oleic acid	Oleic acid	Oleic acid	Oleic acid	Oleic acid	Oleic acid	Oleic acid	Oleic acid	Oleic acid
Average reacted mole number "l" of EO_s	-	-	-	-	-	-	-	-	-	2.5	2.5	-	-	-	-	-	5	-
Average reacted number of "m" of POs	2.5	1.5	6	10	2.5	2.5	2.5	10	2.5	2.5	2.5	-	-	-	25	2.5	2.5	2.5
Average reacted number of "n" of EOs	5	7	7	7	12	5	5	7	5	2.5	2.5	7	7	-	7	25	-	5
Arrangement of $(EO)_l$ and $(PO)_m$	-	-	-	-	-	-	-	-	-	Block	Random	-	-	-	-	-	Block	-
Arrangement of $((EO)_l$-$(PO)_m)$ and $(EO)_n$	Block	Block	Block	Block	Block	Block	Block	Block	Block	Block	Block	-	-	-	Block	Block	-	Random
Rate of esterification (%)	97	98	97	97	98	97	98	98	97	96	95	98	97	98	95	97	83	87

Examples 1 to 11 and Comparative Examples 1 to 8

[0039] Ester compounds shown in Table 1 were evaluated for appearance and heat resistance according to the following methods. These ester compounds shown in Table 1 were used to prepare fiber treatment agents according to the following procedure. Fiber treatment agents were evaluated for lubricating performance and emulsion stability according to the following methods. Results are collectively shown in Table 2.

[Evaluation for appearance]

[0040] An ester compound used as a lubricating component in a fiber treatment agent should be liquid at a treatment temperature to offer a sufficient lubricating performance and be easy to handle at an ambient temperature (20°C). This evaluation graded ester compounds shown in Table 1 on the following scale in terms of appearance at 20°C (ambient temperature). Results are shown in Table 2.
Scale of appearance:

○: being clear liquid
Δ: having fluidity while containing precipitation
x: being solid

[Evaluation for heat resistance]

[0041] An ester compound used as a lubricating component in a fiber treatment agent causes problems such as fuming and a reduced lubricating performance, if it is volatilized or thermally decomposed at a treatment temperature during spinning. Such an ester compound thus should not be volatilized or thermally decomposed at a temperature of spinning (generally about 200°C). In this evaluation, ester compounds shown in Table 1 were evaluated in term of volatilization and thermal decomposition behavior in a high-temperature region by thermogravimetric analysis. Using simultaneous thermogravimetry/differential thermal analyzer TG/DTA7200 (Seiko Instruments Inc.), ester compounds shown in Table 1 were heated in air flowing at an increasing rate of 10°C/min from an initial temperature 20°C and a temperature (T5) when the ester compound has a reduction of weight corresponding to 5% by weight of the initial weight was determined. Rating was made with the following scale. Results are shown in Table 2.

Scales of heat resistance:

[0042]

○: T5 is not lower than 200°C
×: T5 is lower than 200°C

[Evaluation for lubricating performance]

[0043] In a 1000 ml glass beaker, to deionized water in a required amount to provide 500 g of finished fiber treatment agent were added 42.5 g (8.5% by mass of the fiber treatment agent) of ester compound shown in Table 1, 5 g (1.0% by mass of the fiber treatment agent) of polyoxyethylene (7) alkyl (sec- C_{11-15}) ether [Emulgen 707, Kao Corporation] as an emulsifying component, and 2.5 g (0.5% by mass of the fiber treatment agent) of lauryl sulfate, and stirred at a room temperature (25°C) to obtain the fiber treatment agent in an aqueous emulsion form.
Sample yarn (polyester filament yarn: 83 dtex, 36 filaments) was immersed in the fiber treatment agent to be coated with the fiber treatment agent in an amount of 1.5 parts by mass, as the ester compound, to 100 parts by mass of yarn, and dried to give a test yarn.
The test yarn was tested by a running yarn method with a friction meter (micro tension meter TWD- 4, Eiko Sokki Co., Ltd.,), including contacting a running yarn with a metal pin (stainless steel) under conditions of an initial tension of 20 gf, a yarn speed of 500 m/min, and a friction angle of 180°. A dynamic friction between the yarn and the metal during the yarn passing through the metal pin was measured five times to calculate an average value. From the average value, a coefficient of dynamic friction was determined. Results are shown in Table 2. The smaller coefficient of dynamic friction refers to the smaller friction of the yarn or the better lubricating performance of the fiber- treatment agent.

[Evaluation for emulsion stability]

[0044] In a 500 ml glass beaker, deionized water was introduced in a required amount to provide 300 g of a finished fiber treatment agent. Then were added 30 g (10% by mass of the fiber treatment agent) of ester compound shown in Table 1, and either polyoxyethylene (7) alkyl (sec- C_{11-15}) ether [Emulgen 707, Kao Corporation] or polyoxyethylene (7) alkyl (C_{12-13}) ether [Emulgen507, Kao Corporation, proportion of a branched alkyl with β- methyl: 20%]] as an emulsifying component in an amount of 0.5 to 2.0% by mass of the fiber treatment agent. They were stirred at a room temperature (25°C) to obtain the fiber treatment agent in an aqueous emulsion form.

The fiber treatment agent was stored at 25°C and evaluated for its emulsified state after one day and seven days storages according to the following scales. Results are shown in Table 2.

Scales of emulsified state:

○: keeping a uniformly emulsified state
x: separating into two layers

[0045] [Table 2]

【Table 2】

			Example											Comparative example							
			1	2	3	4	5	6	7	8	9	10	11	1	2	3	4	5	6	7	8
	Ester compound		(A)	(B)	(C)	(D)	(E)	(F)	(G)	(H)	(I)	(J)	(K)	Not used	(a)	(b)	(c)	(d)	(e)	(f)	(g)
Results of evaluation	Appearance	20°C	○	○	○	○	○	○	○	○	○	○	○	-	×	○	○	△	×	△	△
	Heat resistance	at temperature not higher than T5 (200°C)	○	○	○	○	○	○	○	○	○	○	○	-	○	○	○	○	○	×	×
	Lubricating performance	Coefficient of dynamic friction	1.3	1.3	1.3	1.4	1.3	1.3	1.3	1.4	1.4	1.4	1.4	Too large a friction to measure (yarn broke)	1.4	1.4	1.4	1.5	1.6	1.5	1.5
	Emulsion stability (1)	ester compound/emulsifying component (surfactantA *1)/water =10/0.5/balance (% by mass) At 25°C, one day after	○	○	○	○	○	○	○	○	○	○	○	-	×	×	×	×	○	×	×
		At 25°C, seven days after	○	○	○	×	○	○	○	○	○	○	○	-	×	×	×	×	○	×	×
		Ester compound/emulsifying component (surfactant A *1) /water =10/1.0/balance (% by mass) At 25°C, one day after	○	○	○	○	○	○	○	○	○	○	○	-	×	×	×	×	○	○	○
		At 25°C, seven days after	○	○	○	○	○	○	○	○	○	○	○	-	×	×	×	×	○	×	×
	Emulsion stability (2)	Ester compound/emulsifying component (surfactantB *2)/water =10/1.0/balance(% by mass) At 25°C, one day after	○	○	○	○	○	○	○	○	○	○	○	-	×	×	×	×	○	×	×
		At 25°C, seven days after	○	○	○	○	○	○	○	○	○	○	○	-	×	×	×	×	○	×	×

[*1] :polyoxyethylene (7) alkyl (sec-C_{11-15}) ether 〔Emulgen 707, manufactured by Kao Corporation〕

[*2] :polyoxyethylene (7) alkyl (C_{12-13}) ether 〔Emulgen 507, Kao Corporation, proportion of branched alkyl with β-methyl: about 20%〕

[0046] Fiber treatment agents containing ester compounds having a specific structure represented by the formula (1) according to the present invention have all good properties in appearance, heat resistance, lubricating performance and emulsion stability in an aqueous system. Furthermore, the fiber treatment agent of the present invention can be easily removed during scouring and washing, and therefore significantly contribute to facilitating a manufacturing process of fiber.

Claims

1. A fiber treatment agent, comprising an ester compound represented by the formula (1) :

$$R^1\text{- O- }((EO)_l\text{- }(PO)_m)\text{- }(EO)_n\text{- CO- }R^2 \qquad (1)$$

wherein, R^1 represents a hydrocarbon group comprising 8 to 24 carbon atoms; R^2 represents an alkyl group, an alkenyl group or a cycloalkyl group, having 7 to 23 carbon atoms; EO represents an ethyleneoxy group; PO represents a propyleneoxy group; 1 and n each represent an average reacted mole number of ethyleneoxy groups, 1 ranging from 0 to 15 and n ranging from 1 to 20; m represents an average reacted mole number of propyleneoxy groups, ranging from 0.5 to 20; and $((EO)_l\text{- }(PO)_m)$ and $(EO)_n$ are arranged in block in this order, while $(EO)_l$ and $(PO)_m$ in $((EO)_l\text{- }(PO)_m)$ may be arranged at random or in block.

2. The fiber treatment agent according to claim 1, wherein R^1 in the formula (1) represents a linear primary alkyl or alkenyl group having 8 to 24 carbon atoms.

3. The fiber treatment agent according to claim 1 or 2, wherein R^1 in the formula (1) represents a linear primary alkyl group comprising 12 to 18 carbon atoms.

4. The fiber treatment agent according to any one of claims 1 to 3, wherein R^2 in the formula (1) represents a linear alkyl or alkenyl group having 7 to 23 carbon atoms.

5. The fiber treatment agent according to any one of claims 1 to 4, wherein R^2 in the formula (1) represents a linear primary alkyl or alkenyl group having 11 to 17 carbon atoms.

6. The fiber treatment agent according to any one of claims 1 to 5, wherein 1 in the formula (1) ranges from 0 to 3.

7. The fiber treatment agent according to any one of claims 1 to 6, wherein m in the formula (1) ranges from 1 to 10.

8. The fiber treatment agent according to any one of claims 1 or 7, wherein n in the formula (1) ranges from 3 to 12.

9. The fiber treatment agent according to any one of claims 1 or 8, further comprising an emulsifying agent component.

10. The fiber treatment agent according to any one of claims 1 to 9, wherein $(EO)_l$ and $(PO)_m$ in $((EO)_l\text{- }(PO)_m)$ in the formula (1) are arranged in block in this order.

11. The fiber treatment agent according to any one of claims 1 to 10, wherein a content of the ester compound represented by the formula (1) is 3 to 70% by mass, and the fiber treatment agent further contains water.

12. The fiber treatment agent according to any one of claims 1 to 11, which is for synthetic fibers.

13. A method for treating fibers, comprising coating fiber with the fiber treatment agent according to any one of claims 1 or 12 in an amount of 0.1 to 3 parts by mass of the ester compound represented by the formula (1) to 100 parts by mass of fibers, and then drawing it and rolling it.

14. The fiber treatment agent according to any one of claims 9 to 12, wherein a content of the emulsifying component is 0.1 to 5% by mass of the fiber treatment agent.

15. The fiber treatment agent according to any one of claims 9 to 12 and 14, wherein a ratio of the emulsifying agent component to the ester compound represented by the formula (1) in the fiber treatment agent is 0.4 to 10% by mass.

16. The fiber treatment agent according to any one of claims 9 to 12, 14 and 15, wherein the ester compound represented by the formula (1) is produced through steps (I) to (III);

step (I): reacting an EO/PO mixture to one or two or more alcohols represented by the formula R^1-OH (2) (wherein, R^1 has the same meanings as above);
step (II): reacting EO to the resulting alkoxylate; and
step (III): esterifying the resulting alkoxylate.

17. The method for treating fibers according to claim 13, wherein fiber is treated in a solution of the fiber treatment agent having a concentration of the ester compound represented by the formula (1) of 1 to 150 g/L.

18. Use of the ester compound according to any one of claims 9 to 12 and 14 to 16 as a fiber treatment agent in steps of producing, processing and spinning fiber with a thermal treatment.

INTERNATIONAL SEARCH REPORT

International application No.
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A. CLASSIFICATION OF SUBJECT MATTER
D06M15/507(2006.01)i, *D06M13/165*(2006.01)i, *D06M13/224*(2006.01)i, *D06M15/513*(2006.01)i

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, C08K3/00-13/08, C08L1/00-101/14

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)
JSTPlus(JDreamII)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	JP 2000-282363 A (Sanyo Chemical Industries, Ltd.), 10 October 2000 (10.10.2000), entire text (Family: none)	1-18
A	JP 9-59869 A (Toray Industries, Inc.), 04 March 1997 (04.03.1997), entire text (Family: none)	1-18
A	JP 3-213577 A (Toray Industries, Inc.), 18 September 1991 (18.09.1991), entire text (Family: none)	1-18

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

* Special categories of cited documents:

"A"	document defining the general state of the art which is not considered to be of particular relevance	"T"	later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
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"O"	document referring to an oral disclosure, use, exhibition or other means	"&"	document member of the same patent family
"P"	document published prior to the international filing date but later than the priority date claimed		

Date of the actual completion of the international search 05 March, 2012 (05.03.12)	Date of mailing of the international search report 13 March, 2012 (13.03.12)
Name and mailing address of the ISA/ Japanese Patent Office Facsimile No.	Authorized officer Telephone No.

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INTERNATIONAL SEARCH REPORT

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C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	JP 2004-353115 A (Sanyo Chemical Industries, Ltd.), 16 December 2004 (16.12.2004), entire text (Family: none)	1-18
A	JP 2007-262588 A (Teijin Fibers Ltd.), 11 October 2007 (11.10.2007), entire text (Family: none)	1-18

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- JP 10245781 A **[0004] [0008]**
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- JP 10251974 A **[0005]**