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(54) **Titre : Polyester-based fiber for artificial hair,method for producing the same, and, fiber bundle for hair and hair ornament product including the same.**

(57) **Abrégé :**

A polyester-based fiber for artificial hair having excellent flame retardance and also a smooth touch similar to that of human hair, a method for producing the same, and, a fiber bundle for hair and a hair ornament product including the same, are provided. The polyester-based fiber for artificial hair of the present invention includes a polyester resin, a brominated epoxy flame retardant and an antimony compound. The polyester resin is polyalkylene terephthalate and/or a copolymerized polyester containing polyalkylene terephthalate as a main component. The antimony compound is a compound containing pentavalent antimony and having a PH of 3 to 10, and the antimony compound has an average diagonal width of 0.5 um or less in the fiber cross section. A method for producing the polyester-based fiber for artificial hair includes a step of melt kneading a polyester resin composition including a polyester resin, a brominated epoxy flame retardant and antimony compound by an extruder, and during the melt kneading step, a ratio Q/R of a discharge amount Q (kg/hour) to a screw rotation number R (rpm) of the extruder is 1.8 or less.

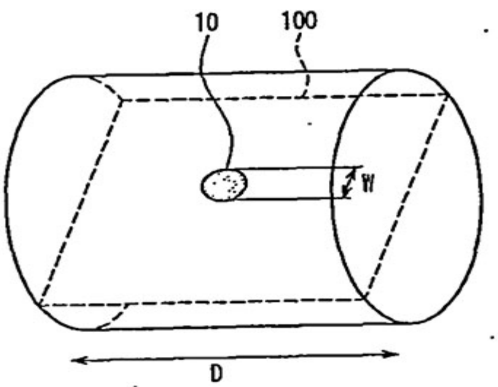


Fig. 1

KANEKA CORPORATION

POLYESTER-BASED FIBER FOR ARTIFICIAL HAIR, METHOD FOR PRODUCING THE SAME, AND, FIBER BUNDLE FOR HAIR AND HAIR ORNAMENT PRODUCT INCLUDING THE SAME

BACKGROUND OF THE INVENTION

1. Field of the Invention

The present invention relates to a polyester-based fiber for artificial hair that can be used as a substitute for human hair, a method for producing the same, and a fiber bundle for hair and a hair ornament product including the same. Specifically, the present invention relates to a polyester-based fiber for artificial hair including a brominated epoxy flame retardant and an antimony compound, a method for producing the same, and a fiber bundle for hair and a hair ornament product including the same.

2. Description of Related Art

Conventionally, human hair has been used for hair ornament products such as hairpieces, hair wigs, hair extensions, headbands, and doll hair. In recent years, however, it becomes more and more difficult to obtain the human hair, and thus, demand for fibers for artificial hair to supplant human hair has been increased. For example, use of a polyester-based fiber containing polyethylene terephthalate as a main component having excellent heat resistance has been proposed for the material of fibers for artificial hair. In such a case of using the fiber as the fiber for artificial hair, flame retardance is required from the viewpoint of safety, and thus providing flame retardance to the polyester-based fiber has been studied. For example, JP 2007-131982 A, JP 2007-84951 A and JP 2006-97185 A propose addition of brominated epoxy flame retardant and an antimony compound to the polyester-based fiber for the purpose of providing flame retardance.

Although the flame retardance of these polyester-based fibers described in these patent documents is improved as a result of addition of the brominated epoxy flame retardant and the antimony compound, in some cases the brominated epoxy flame retardant and the antimony compound cause roughness in the fiber, thereby degrading the touch.

SUMMARY OF THE INVENTION

Therefore, with the foregoing in mind, it is an object of the present invention to provide a polyester-based fiber for artificial hair having excellent flame retardance and further having a smooth touch similar to that of human hair, a method for producing the same, and a fiber bundle for hair and a hair ornament product including the same.

The present invention relates to a polyester-based fiber for artificial hair that includes a polyester resin, a brominated epoxy flame retardant and an antimony compound. The polyester resin is polyalkylene terephthalate and/or a copolymerized polyester containing polyalkylene terephthalate as a main component. The antimony compound is a compound containing pentavalent antimony and having a pH of 3 to 10, and the antimony compound has an average diagonal width of 0.5 μm or less in the fiber cross section.

It is preferable that in the fiber cross section of the polyester-based fiber for artificial hair of the present invention, the average diagonal width of the antimony compound is 0.1 to 0.5 μm, and the standard deviation of the diagonal width is 0.1 to 0.3. Alternatively, it is preferable that in the fiber cross section of the polyester-based fiber for artificial hair of the present invention, the average diagonal width of the antimony compound is less than 0.1 μm. It is preferable that the antimony compound is antimony pentoxide having a pH of 3 to 10.

The present invention relates also to a method for producing the polyester-based fiber for artificial hair as described above. The method includes a step of melt kneading a polyester resin composition including a polyester resin, a brominated epoxy flame retardant and an antimony compound by an extruder. During the melt kneading step, a ratio Q/R of a discharge amount Q (kg/hour) to a screw rotation number R (rpm) of the extruder is set to 1.8 or less.

The present invention relates also to a fiber bundle for hair that includes the above-described polyester-based fiber for artificial hair, and at least one fiber selected from the group consisting of human hair, animal hair, a polyvinyl chloride-based fiber, a modacrylic fiber, a polyamide-based fiber, a polyolefin-based fiber, a regenerated protein fiber and another polyester-based fiber.

The present invention relates also to a hair ornament product including the polyester-based fiber for artificial hair as described above.

The hair ornament product may include further at least one fiber selected from the group consisting of human hair, animal hair, a polyvinyl chloride-based fiber, a modacrylic fiber, a polyamide-based fiber, a polyolefin-based fiber, a regenerated protein fiber and another polyester-based fiber.

The present invention provides a polyester-based fiber for artificial hair, a fiber bundle for hair and a hair ornament product having an excellent flame retardance and further a smooth touch similar to that of human hair, by using a compound containing pentavalent antimony and having a pH of 3 to 10 as an antimony compound in a polyester-based fiber for artificial hair including a polyester resin, a brominated epoxy flame retardant and the antimony compound, and by setting the average diagonal width of the antimony compound in the fiber cross section to 0.5 μm or less.

According to the producing method of the present invention, during the step of melt kneading a polyester resin composition including a polyester resin, a brominated epoxy flame retardant and an antimony compound by an extruder, the ratio Q/R of the discharge amount Q (kg/hour) to the screw rotation number R (rpm) of the extruder is set to 1.8 or less, thereby making it possible to set the average diagonal width of the antimony compound in the fiber cross section to 0.5 μm or less. As a result, it is possible to produce a polyester-based fiber having excellent flame retardance and further a smooth touch similar to that of human hair.

BRIEF DESCRIPTION OF THE DRAWINGS

FIG. 1 is a schematic diagram showing the diagonal width of an antimony compound in the fiber cross section (a cross section in the direction parallel to the fiber axis direction) of a polyester-based fiber for artificial hair.

DETAILED DESCRIPTION OF THE INVENTION

The present inventors have conducted numerous studies to solve the above problems and found out that in a polyester-based fiber for artificial hair provided with flame retardance by blending a brominated epoxy flame retardant and an antimony compound in a polyester resin, by using a compound containing pentavalent antimony and having a pH of 3 to 10 as the antimony compound and also by setting the average diagonal width of the antimony compound in the fiber cross section to 0.5 μm or less, substantially no roughness is caused by the brominated epoxy flame retardant and the antimony compound, thereby a smooth touch similar to that of human hair is obtained, and thus the present inventors have reached the present invention. Further by setting the ratio Q/R of the discharge amount Q (kg/hour) to the screw rotation number R (rpm) of an extruder to 1.8 or less, during melt kneading a polyester resin composition including the brominated epoxy flame retardant and the antimony compound by the extruder, the average diagonal width of the antimony compound in the fiber cross section is 0.5 μm or less in a polyester-based fiber obtained by melt spinning the polyester resin composition after the melt kneading.

The polyester-based fiber for artificial hair of the present invention is formed of a polyester resin composition including a polyester resin, a brominated epoxy flame retardant and an antimony compound.

The polyester resin is polyalkylene terephthalate and/or a copolymerized polyester containing polyalkylene terephthalate as a main component. The polyalkylene terephthalate is not particularly limited and may be, e.g., polyethylene terephthalate, polypropylene terephthalate, polybutylene terephthalate, or polycyclohexane dimethylene terephthalate. The copolymerized polyester containing polyalkylene terephthalate as the main component is not particularly limited and may be, e.g., a copolymerized polyester containing polyalkylene terephthalate (such as polyethylene terephthalate, polypropylene terephthalate, polybutylene terephthalate, or polycyclohexane dimethylene terephthalate) as the main component and other copolymerizable components. In the present invention, the term "main component" means a component contained in an amount of 50 mol% or more. Therefore, the "copolymerized polyester containing polyalkylene terephthalate as the main component" refers to the copolymerized polyester containing 50 mol% or more of polyalkylene terephthalate. Preferably, the "copolymerized polyester containing polyalkylene terephthalate as the main component" contains 60 mol% or more, more preferably 70 mol% or more, and further preferably 80 mol% or more of polyalkylene terephthalate.

Examples of the other copolymerizable components include the following: polycarboxylic acids such as isophthalic acid, orthophthalic acid, naphthalenedicarboxylic acid, paraphenylenedicarboxylic acid, trimellitic acid, pyromellitic acid, succinic acid, glutaric acid, adipic acid, suberic acid, azelaic acid, sebacic acid, and dodecanedioic acid, and their derivatives; dicarboxylic acids including a sulfonic acid salt such as 5-sodiumsulfoisophthalic acid and dihydroxyethyl 5-sodiumsulfoisophthalate, and their derivatives; 1,2-propanediol; 1,3-propanediol; 1,4-butanediol; 1,6-hexanediol; neopentyl glycol; 1,4-cyclohexanedimethanol; diethylene glycol; polyethylene glycol; trimethylolpropane; pentaerythritol; 4-hydroxybenzoic acid; and ε-caprolactone.

The specific examples of the copolymerized polyester containing polyalkylene terephthalate as the main component include a copolymerized polyester obtained by copolymerization of polyethylene terephthalate as the main component with one kind of compound selected from the group consisting of ethylene glycol ether of bisphenol A, 1,4-cyclohexanedimethanol, isophthalic acid, and dihydroxyethyl 5-sodiumsulfoisophthalate.

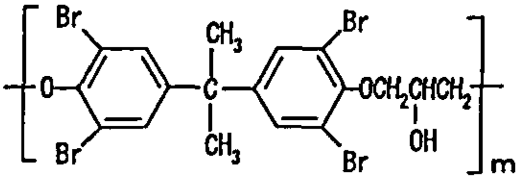
The polyalkylene terephthalate and the copolymerized polyester containing polyalkylene terephthalate as the main component may be used individually or in combinations of two or more. In particular, it is preferable that polyethylene terephthalate, polypropylene terephthalate, polybutylene terephthalate, a copolymerized polyester obtained by copolymerization of polyethylene terephthalate as the main component with ethylene glycol ether of bisphenol A, a copolymerized polyester obtained by copolymerization of polyethylene terephthalate as the main component with 1,4-cyclohexanedimethanol, a copolymerized polyester obtained by copolymerization of polyethylene terephthalate as the main component with isophthalic acid, and a copolymerized polyester obtained by copolymerization of polyethylene terephthalate as the main component with dihydroxyethyl 5-sodiumsulfoisophthalate are used individually or in combinations of two or more.

Though there is no particular limitation on the material of the brominated epoxy flame retardant, for example, a brominated epoxy flame retardant having an epoxy group, an alkyltribromo phenoxy group or the like at the end of the molecule can be used. From the viewpoint that the antimony compound is dispersed easily in the brominated epoxy flame retardant, it is preferable that the brominated epoxy flame retardant has an epoxy group at the end of the molecule.

The antimony compound has a refractive index higher than the refractive index of the polyester resin, and the brominated epoxy flame retardant has a refractive index approximate to the refractive index of the polyester resin. Therefore, when the antimony compound is dispersed in the brominated epoxy flame retardant dispersed in the polyester resin, the color development (transparency) of the polyester-based fiber for artificial hair is improved further in comparison with a case of dispersing the antimony compound directly in the polyester resin. Namely, when the rate of the antimony compound dispersed in the brominated epoxy flame retardant is higher, the color development of the polyester-based fiber for artificial hair is better.

For the brominated epoxy flame retardant, specifically, a compound including a structural formula represented by the general formula (1) in the molecules below can be used.

[Chemical formula 1]

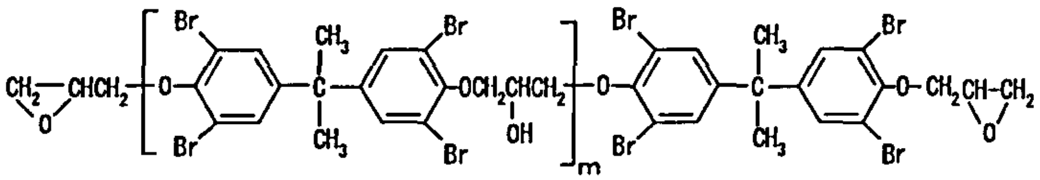


(1)

In the general formula (1), m is 1 to 1000.

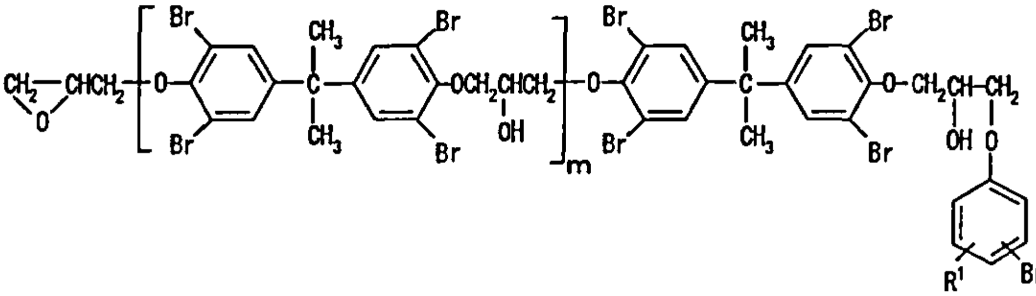
For a compound represented by the general formula (1), specifically, the compounds represented by the general formulae (2) to (4) below can be used.

[Chemical formula 2]



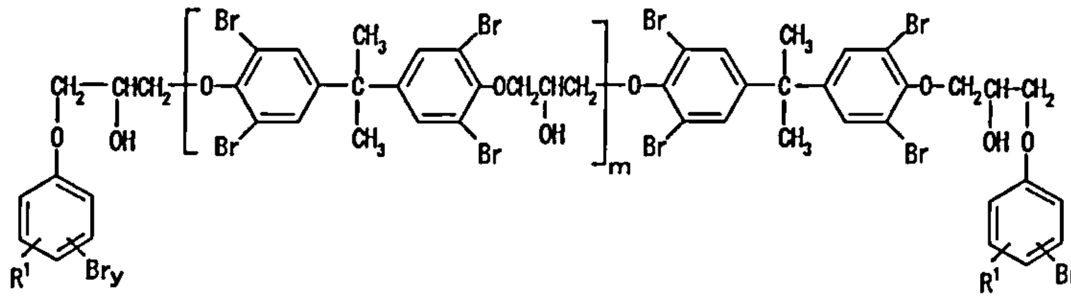
(2)

[Chemical formula 3]



(3)

[Chemical formula 4]



(4)

In the general formulae (2) to (4), m is 1 to 1000, R^1 is a C_1 to C_{10} alkyl group, and y is 0 to 5.

In the present invention, a brominated epoxy flame retardant having the above-mentioned structure at the end of the molecule is used as the material. However, the structure of the brominated epoxy flame retardant after the melt kneading and/or melt spinning (in the fiber) is not limited in particular. For example, the end of the molecule may be substituted by an epoxy group, a hydroxyl group, a phosphate group or a phosphonic acid group. Alternatively, the end of the molecule may be coupled with the polyester resin via the ester group.

The brominated epoxy flame retardant may be used individually or in combination of two or more kinds.

It is preferable that the polyester-based fiber for artificial hair includes 5 to 40 parts by weight of the brominated epoxy flame retardant with respect to 100 parts by weight of the polyester resin. It is more preferable that the lower limit of the content of the brominated epoxy flame retardant with respect to 100 parts by weight of the polyester resin is 6 parts by weight or more. It is more preferable that the upper limit of the content of the brominated epoxy flame retardant with respect to 100 parts by weight of the polyester resin is 30 parts by weight or less, and further preferably, 25 parts by weight or less. When the content of the brominated epoxy flame retardant is within the above-mentioned range, the polyester-based fiber for artificial hair exhibits excellent flame retardance, color development and spinning property.

The polyester-based fiber for artificial hair includes an antimony compound. The antimony compound functions as a flame retardant auxiliary and improves the flame retardance of the polyester-based fiber for artificial hair. It is preferable that the polyester-based fiber for artificial hair includes 0.5 to 10 parts by weight of the antimony compound with respect to 100 parts by weight of the polyester resin. It is more preferable that the lower limit of the content of the antimony compound with respect to 100 parts by weight of the polyester resin is 1 part by weight or more, and further preferably, 1.5 parts by weight or more. It is more preferable that the upper limit of the content of the antimony compound with respect to 100 parts by weight of the polyester resin is 7 parts by weight or less, and further preferably, 5 parts by weight or less.

The antimony compound is a compound containing pentavalent antimony, and its pH is in the range of 3 to 10. Since a compound containing pentavalent antimony and having a pH of 3 to 10 is used, substantially no roughness occurs and the polyester-based fiber for artificial hair has a smooth touch. Furthermore, the compound containing the pentavalent antimony is dispersed easily in the brominated epoxy flame retardant, thereby the color development of the polyester-based fiber for artificial hair is improved. When a compound containing trivalent antimony is used, the brominated epoxy flame retardant aggregates, thereby roughness occurs in the fiber so as to degrade the touch, and the color development also deteriorates. Similarly, when a compound containing pentavalent antimony and having a pH of less than 3 is used, the brominated epoxy flame retardant aggregates, thereby roughness occurs in the fiber so as to degrade the touch, and the color development also deteriorates. When the pH of the antimony compound is 10 or less, hydrolysis does not occur and the workability is improved. The lower limit of the pH of the antimony compound (the compound containing pentavalent antimony) is preferably 4 or more, and more preferably, 5 or more. The upper limit of the pH of the antimony compound (the compound containing pentavalent antimony) is preferably 9.5 or less, and more preferably, 9 or less. Here, the pH of the antimony compound is a value measured after dispersing 1 g of the target compound in 10 g of pure water at room temperature (20°C).

For the antimony compound, for example, pentavalent antimony having a pH of 3 to 10, sodium antimonate (antimony sodiate), potassium antimonate and the like can be used. From the viewpoint of ensuring the fiber physical properties such as strength and suppressing gelation of the brominated epoxy flame retardant, it is preferable that the antimony compound is pentavalent antimony having a pH of 3 to 10. These antimony compounds may be used individually or in combination of two or more.

In the fiber cross section of the polyester-based fiber for artificial hair, the average diagonal width of the antimony compound is 0.5 μm or less, preferably 0.4 μm or less, and more preferably 0.3 μm or less. When the average diagonal width of the antimony compound in the fiber cross section of the polyester-based fiber for artificial hair is within the above-mentioned range, substantially no roughness occurs and the fiber has a smooth touch similar to that of human hair.

In the fiber cross section of the polyester-based fiber for artificial hair, it is preferable that the average diagonal width of the antimony compound is in the range of 0.1 to 0.5 μm, and the standard deviation of the diagonal width is in the range of 0.1 to 0.3. More preferably, the average diagonal width is 0.1 to 0.4 μm, and the standard deviation of the diagonal width is 0.1 to 0.3. And further preferably, the average diagonal width is 0.1 to 0.3 μm, and the standard deviation of the diagonal width is 0.1 to 0.25. As described above, since the antimony compound has a certain dimension and the dimension of the antimony compound varies within a predetermined range, the polyester-based fiber for artificial hair has a smooth and dry touch with substantially no stickiness, and the color development also is favorable.

In the fiber cross section of the polyester-based fiber for artificial hair, it is preferable that the average diagonal width of the antimony compound is less than 0.1 μm. When the average diagonal width of the antimony compound is within the above-mentioned range, the dispersibility is high, and the color development is improved remarkably. When the average diagonal width of the antimony compound is smaller, it is dispersed more easily in the brominated epoxy flame retardant, and the color development is improved. From the viewpoint of stickiness, it is preferable that the lower limit of the average diagonal width of the antimony compound is 0.005 μm or more, and more preferably 0.01 μm or more.

From the viewpoint of setting the average diagonal width of the antimony compound in the fiber cross section of the polyester-based fiber for artificial hair to 0.5 μm or less, it is preferable that the particle diameter of the antimony compound used as the material is 3.5 μm or less, more preferably 3.0 μm or less, and further preferably 2.5 μm or less. Though there is no particular lower limit for the particle diameter of the antimony compound used as the material, from the viewpoint of workability, it is preferably 0.02 μm or more, more preferably 0.1 μm or more, and further preferably 0.2 μm or more. The particle diameter of the antimony compound used as the material indicates a median diameter by grain size distribution measurement. The grain size distribution measurement can be performed by using a laser diffraction particle size analyzer (model "SALD-7000" manufactured by Shimadzu Corporation) and by using distilled water as a dispersion medium.

In the present invention, the fiber cross section indicates a cross section parallel to the fiber axis direction, and the diagonal width of the antimony compound in the fiber cross section indicates the largest length of the antimony compound in the direction perpendicular to the fiber axis direction in the cross section parallel to the fiber axis direction. The average diagonal width of the antimony compound in the fiber cross section is the average of the diagonal widths of the antimony compounds present in the area of 360 μm^2 of the cross section of the fiber parallel to the fiber axis direction. The standard deviation of the diagonal width of the antimony compound in the fiber cross section indicates the standard deviation of the diagonal width of the antimony compounds present in the area of 360 μm^2

of the cross section parallel to the fiber axis direction. This will be explained specifically hereinafter with reference to FIG. 1 that schematically shows the diagonal width of the antimony compound in the fiber cross section (a cross section in the direction parallel to the fiber axis direction) of the polyester-based fiber for artificial hair of the present invention. As shown in FIG. 1, in the cross section 100 parallel to the fiber axis direction D, the largest length W in the direction perpendicular to the fiber axis direction D of the antimony compound 10 is the diagonal width.

In the polyester-based fiber for artificial hair, the antimony compound in the fiber cross section (a cross section parallel to the fiber axis direction) can be observed by a scanning electron microscope (SEM) or the like. Further in the present invention, it is possible to measure the diagonal width of the antimony compound in the fiber cross section and calculate the average diagonal width and the standard deviation of the diagonal width by using a SEM image of the fiber cross section of the polyester-based fiber for artificial hair. Preparation of the fiber cross section of the polyester-based fiber for artificial hair can be conducted for example by preparation of a cross section (ion milling) using a cross section polisher (CP). The morphological observation can be performed by using a field emission scanning electron microscope (FE-SEM) ("ULTRA plus" manufactured by Carl Zeiss Co., Ltd.) at an acceleration voltage of 2 kV. Since a composition image of a sample depends on the average atomic number, the image becomes bright in a portion of the sample that includes heavy elements and becomes dark in a portion of the sample that includes light elements.

The polyester-based fiber for artificial hair may include a flame retardant other than the brominated epoxy flame retardant, a flame retardant auxiliary other than the antimony compound, a heat-resistant agent, a stabilizer, a fluorescent agent, an antioxidant, an antistatic agent and the like as required, as long as they do not interfere with the effects of the present invention.

The polyester-based fiber for artificial hair of the present invention is obtained by for example, melt kneading a polyester resin composition including a polyester resin, a brominated epoxy flame retardant, an antimony compound and the like, and melt spinning the melt kneaded polyester resin composition by an conventional melt spinning method.

It is preferable that the polyester resin composition includes 5 to 40 parts by weight of the brominated epoxy flame retardant with respect to 100 parts by weight of the polyester resin. The more preferable lower limit of the content of the brominated epoxy flame retardant with respect to 100 parts by weight of the polyester resin is 6 parts by weight or more. The more preferable upper limit of the content of the brominated epoxy flame retardant with respect to 100 parts by weight of the polyester resin is 30 parts by weight or less, and further preferably, 25 parts by weight or less. It is preferable that the polyester resin composition includes 0.5 to 10 parts by weight of the antimony compound (the compound containing pentavalent antimony and having a pH of 3 to 10) with respect to 100 parts by weight of the polyester resin. The more preferable lower limit of the content of the antimony compound (the compound containing pentavalent antimony and having a pH of 3 to 10) with respect to 100 parts by weight of the polyester resin is 1 part by weight or more, and further preferably, 1.5 parts by weight or more. The further preferable upper limit of the content of the antimony compound (the compound containing pentavalent antimony and having a pH of 3 to 10) with respect to 100 parts by weight of the polyester resin is 7 parts by weight or less, and more preferably, 5 parts by weight or less.

The polyester resin composition can be obtained by dry blending the above-mentioned respective components such as the polyester resin, the brominated epoxy flame retardant and the antimony compound, and then by melt kneading the components by using various conventional kneaders. Examples of the kneaders include a single-screw extruder, a twin-screw extruder, a roll, a Banbury mixer, and a kneader. In particular, the single-screw extruder and the twin-screw extruder are preferred in terms of the adjustment of the degree of kneading and the ease of operation.

It is preferable that the melt kneading of the polyester resin composition is performed by using an extruder under a condition that the ratio Q/R of the discharge amount Q (kg/hour) to the screw rotation number R (rpm) of the extruder (hereinafter, this may be recited also as Q/R simply) is 1.8 or less. It is preferable that the melt kneading of the polyester resin composition is performed at a temperature not lower than the melting point of the polyester resin, for example, at temperature in the range of 250 to 280°C. For the extruder, a twin-screw extruder is preferred. When the Q/R is 1.8 or less, the dispersion of the brominated epoxy flame retardant and the antimony compound in the polyester resin composition after melt kneading is favorable, and furthermore, by melt spinning the polyester resin composition after the melt kneading, it is possible to make the average diagonal width of the antimony compound in the fiber cross section 0.5 μm or less. More preferably, the upper limit of the Q/R is 1.5 or less, and further preferably. 0.8 or less. It is preferable that the lower limit of the Q/R is 0.05 or more, and more preferably, 0.2 or more. When the Q/R is 0.05 or more, there is no risk of decomposition of the polyester resin due to excessive kneading, and the spinning stability is favorable.

The polyester-based fiber for artificial hair of the present invention may be produced by melt spinning the polyester resin composition with a conventional melt spinning method. In such a case, e.g., the polyester resin composition is melt spun into yarns while the temperatures of a gear pump, a spinneret, etc. are set to 250 to 310°C. Then, the spun yarns are cooled to a temperature of not more than the glass transition point of the polyester resin, and wound up at a speed of 50 to 5000 m/min, thereby spun yarns (undrawn yarns) are obtained. Alternatively, the spun yarns may be cooled in a water bath containing cooling water so as to control the fineness. The temperature and amount of the cooling air applied, the temperature of the cooling water bath, the cooling time, and the winding speed can be adjusted appropriately in accordance with the discharge amount of the polymer and the number of holes of the spinneret.

In the present invention, it is preferable that the resultant spun yarns (undrawn yarns) are hot drawn. The drawing may be performed by either a two-step method or a direct drawing method. In the two-step method, the spun yarns are once wound, and then drawn. In the direct drawing method, the spun yarns are drawn continuously without winding. The hot drawing may be performed by a single-stage drawing method or a multi-stage drawing method that includes two or more stages. The heating means for the hot drawing may be, e.g., a heating roller, a heat plate, a steam jet apparatus, or a hot water bath, and they can be used in combination as desired.

It is preferable that the polyester-based fiber for artificial hair of the present invention is a non-crimped fiber. From the viewpoint of suitability to artificial hair, the fineness of the polyester-based fiber is preferably in the range of 10 to 100 dtex. It is more preferable that the lower limit of the fineness of the polyester-based fiber for artificial hair is 20 dtex or more, and further preferably 35 dtex or more. It is more preferable that the upper limit of the fineness of the polyester-based fiber for artificial hair is 90 dtex or less, and further preferably 80 dtex or less.

The polyester-based fiber for artificial hair of the present invention has an excellent flame retardance, and furthermore, it has a smooth touch with substantially no stickiness. Preferably, it also has excellent color development.

The flame retardance of the polyester-based fiber for artificial hair can be checked with reference to the LOI value and occurrence of dripping in a combustion test. The measurement of the LOI value and the combustion test can be performed as described later. From the viewpoint of excellent flame retardance, it is preferable that the polyester-based fiber for artificial hair has a LOI value of 23 or more, and dripping does not occur in the combustion test. It is more preferable that the LOI value is 25 or more and dripping does not occur in the combustion test.

The polyester-based fiber for artificial hair of the present invention can be directly used alone as artificial hair. Alternatively, the polyester-based fiber for artificial hair can be blended with at least one fiber selected from the group consisting of human hair, animal hair, polyvinyl chloride-based fiber, modacrylic fiber, polyamide-based fiber, polyolefin-based fiber, regenerated protein fiber and another polyester-based fiber so as to be used as a fiber bundle for hair.

The hair ornament product of the present invention may be formed only of the polyester-based fiber for artificial hair of the present invention. Alternatively, the hair ornament product may be formed by combining the polyester-based fiber for artificial hair of the present invention with at least one fiber selected from the group consisting of human hair, animal hair, polyvinyl chloride-based fiber, modacrylic fiber, polyamide-based fiber, polyolefin-based fiber, regenerated protein fiber and another polyester-based fiber.

No particular limitation is imposed on the hair ornament product, and examples thereof include a hairpiece, a hair wig, a weaving, a hair extension, braid hair, a hair accessory, and doll hair.

Examples

Hereinafter, the present invention will be described in more detail based on Examples. However, the present invention is not limited to the Examples.

The compounds (materials) used in Examples and Comparative examples are stated below.

Polyethylene terephthalate (hereinafter, this is recited also as "PET"): manufactured by Mitsubishi Chemical Corporation, trade name: "BK–2180"

Brominated epoxy flame retardant (hereinafter, this is recited also simply as "flame retardant"): manufactured by Sakamoto Yakuhin Kogyo Co., Ltd., trade name: "SRT–20000", number-average molecular weight: 40000, epoxy-terminated brominated epoxy flame retardant

Flame retardant auxiliary I: sodium antimonate (V) manufactured by Nihon Seiko Co., Ltd. trade name: "SA–A", particle diameter: 2.5 μm, pH: 9

Flame retardant auxiliary II: antimony pentoxide manufactured by

NYACOL Nano Technologies, Inc., trade name: "ZTA", particle diameter: 2 μm, pH: 9.5

Flame retardant auxiliary III: antimony pentoxide manufactured by NYACOL Nano Technologies, Inc., trade name: "6220", particle diameter: 2 μm, pH: 7

Flame retardant auxiliary IV: antimony pentoxide manufactured by NYACOL Nano Technologies, Inc., trade name: "ADP494", particle diameter: 2 μm, pH: 5

Flame retardant auxiliary V: antimony pentoxide manufactured by NYACOL Nano Technologies, Inc., trade name: "A1588LP", particle diameter: 2 μm, pH: 2.5

Flame retardant auxiliary VI: antimony trioxide manufactured by Nihon Seiko Co., Ltd. trade name: "PATOX-M", particle diameter: 1 μm, pH: 5

Flame retardant auxiliary VII: antimony pentoxide manufactured by Nissan Chemical Industries, LTD., trade name: "Sunepoch NA-1030", particle diameter: 4 μm, pH: 9

In the above description, the pH of the antimony compound is a value measured at room temperature (20°C) after dispersing 1g of a target compound in 10 g of pure water. The particle diameter of the antimony compound is a median diameter measured by using a laser diffraction particle size analyzer (model "SALD-7000" manufactured by Shimadzu Corporation) and by using distilled water as a dispersion medium.

(Examples 1-7, Comparative examples 1-4)

The respective compounds (materials) as described above were dried to a moisture content of 100 ppm or less, and then the materials were dry blended in their proportions as shown in Table 1 below. The polyester resin composition thus obtained was supplied to a twin-screw extruder (trade name: "TEX44" manufactured by Japan Steel Works, LTD.) and melt kneaded at a barrel temperature of 270°C under the conditions for the discharge amount and the screw rotation number as indicated in Table 1, and then were formed into pellets. The pellets were dried to a moisture content of 100 ppm or less. Next, the dried pellets were supplied to a melt spinning machine (trade name: "SV30" manufactured by SHINKO MACHINERY CO., LTD.), and a molten polymer was discharged through a spinneret with nozzle holes having a cocoon-shaped cross section (with an aspect ratio of 1.4 : 1) at a barrel temperature of 270°C. The molten polymer was air-cooled with a cooling air at 20°C and wound up at a speed of 100 m/min, thereby providing undrawn yarns. The resultant undrawn yarns were drawn to 3.5 times using a heating roller heated at 75°C, heat-treated using the heating roller heated at 180°C, and wound up at a speed of 30 m/min. Thus, a polyester-based fiber (multifilament) with a single fiber fineness of about 60 dtex was produced.

For each of the polyester-based fibers obtained in Examples 1-7 and Comparative examples 1-4, the average diagonal width and the standard deviation of the diagonal width of the antimony compound (Sb compound) in the fiber cross section were measured in the following manner, and the results are shown in Table 1 below. Furthermore, the flame retardance, the touch (smoothness), the stickiness and the color development for each of the polyester-based fibers obtained in Examples 1-7 and Comparative examples 1-4 were measured and evaluated in the following manner, and the results are shown in Table 1 below. Further, the spinning stability in Examples 1-7 and Comparative examples 1-4 were measured and evaluated in the following manner, and the results are shown in Table 1 below.

(Average diagonal width and standard deviation of diagonal width of antimony compound)

The diagonal width of an antimony compound in the fiber cross section was observed and measured in the following analytical method. The preparation (ion milling) of the fiber cross section (cross section parallel to the fiber axis direction) was performed using a cross section polisher (CP) ("SM-09020 CP" manufactured by JEOL Ltd.) under the processing conditions of an acceleration voltage of 6 kV. The morphological observation was carried out with a field emission scanning electron microscope (FE-SEM) ("ULTRA plus" manufactured by Carl Zeiss Co., Ltd.) at an acceleration voltage of 2 kV. Since a composition image of a sample depends on the average atomic number, the image becomes bright in a portion of the sample that includes heavy elements and becomes dark in a portion of the sample that includes light elements. In this example, the sample (polyester-based fiber) included the polyethylene terephthalate, the brominated epoxy flame retardant, and the antimony compound. Therefore, the order in which the bright composition image appeared was (1) antimony compound, (2) brominated epoxy flame retardant, and (3) polyethylene terephthalate. Namely, the image of the antimony compound is the brightest. Using the image analysis software ("winROOF" available from Mitani Corporation), the number of antimony compounds per 360 μm^2 and the diagonal widths of the respective antimony compounds were measured to calculate the average value and the standard deviation of the diagonal widths of the antimony compounds per 360 μm^2, which were set respectively as the average diagonal width and the standard deviation of the diagonal width of the antimony compound.

(Flame retardance)

The assessment was carried out by the following four criteria based on the LOI value and occurrence of dripping as a result of the combustion test.

A: Dripping does not occur, and LOI value is 25 or more.

B: Dripping does not occur, and LOI value is 23 or more and less than 25.

C: Dripping occurs, and LOI value is 23 or more.

D: LOI value is less than 23 regardless of dripping.

<Measurement of LOI value>

The LOI value was measured in conformity with JIS L 1091 E (oxygen index test). Specifically, ends of a filament (16 cm in length and 0.25 g in weight) were tied lightly by a double-side tape, pinched by a twisting device and twisted. After twisting sufficiently, the filament was folded in two at a middle thereof and twisted. The ends of the twisted filament were fixed by a Cellophane (registered trademark) tape such that a total length would be 7 cm. The obtained sample was pre-dried at 105°C for 60 minutes, and further was dried in a desiccator for 30 minutes or more. The dried sample was adjusted to a certain oxygen concentration. After 40 seconds, the top of the sample was ignited by an igniter with the igniter flame restricted to 8 to 12 mm. The igniter was apart from the sample after the ignition. The oxygen concentration after flaming with a length of 5 cm or more or flaming continuously for 3 minutes or more was measured, and the test was repeated three times under the same conditions, thereby obtaining the limiting oxygen index LOI.

<Combustion test>

The filament was cut to have a length of 150 mm, 0.7 g of which was tied in a bundle, and one end thereof was pinched by a clamp so as to be fixed to a stand in order to have an effective length of 120 mm and was hung vertically. A flame of 20 mm length was contact with the fixed filament for 3 seconds such that the filament would be burned for the purpose of observing the dripping.

(Touch – smoothness)

Function evaluation was performed by the following four criteria by comparing with human hair.

A: The texture is extremely smooth equivalent to that of human hair, and roughness is not perceived.

B: The texture is smooth similar to that of human hair, and roughness is not perceived substantially.

C: Roughness is perceived.

D: Roughness is perceived considerably.

(Stickiness)

Function evaluation was performed by the following four criteria by comparing with human hair.

A: The level is equivalent to that of human hair, and stickiness is not perceived.

B: The level is substantially equivalent to that of human hair, and stickiness is not perceived substantially.

C: Stickiness is perceived.

D: Stickiness is perceived considerably.

(Color development)

A tow filament with a length of 30 cm and a total fineness of 100,000 dtex was evaluated by visual observation in the sunlight by the following four criteria by comparing with human hair in terms of appearance.

A: A vivid tone approximate to that of human hair is exhibited.

B: The fiber is slightly whitish and vividness in color is slightly inferior to human hair in terms of appearance.

C: The fiber is whitish and vividness in color is inferior to human hair in terms of appearance.

D: The fiber is considerably whitish and vividness in color is evidently inferior to human hair in terms of appearance.

(Spinning stability)

After spinning continuously a polyester-based fiber (a multi-filament having a filament number of 120) for 6 hours, the spinning stability was determined by the following four criteria on the basis of the average filament breakage number for 1 hour.

A: Filament breakage number is 0.

B: Filament breakage number is 1.

C: Filament breakage number is 2 to 4.

D: Filament breakage number is 5 or more.

[Table 1]

		Ex. 1	Ex. 2	Ex. 3	Ex. 4	Ex. 5	Ex. 6	Ex. 7	Com. Ex. 1	Com. Ex. 2	Com. Ex. 3	Com. Ex. 4
Blend rate	PET	100	100	100	100	100	100	100	100	100	100	100
	Flame retardant	20	20	20	20	20	20	20	20	20	20	20
	Flame retardant auxiliary I	2	2	2	2							
	Flame retardant auxiliary II					2						
	Flame retardant auxiliary III						2					
	Flame retardant auxiliary IV							2				
	Flame retardant auxiliary V								2			
	Flame retardant auxiliary VI									2		
	Flame retardant auxiliary VII										2	
pH of antimony compound		9	9	9	9	9.5	7	5	2.5	5	9	9
Melt kneading condition	Discharge amount Q (kg/hour)	50	50	20	100	50	50	50	50	50	50	100
	Screw rotation number R (rpm)	150	250	250	75	150	150	150	150	150	150	50
	Q/R	0.33	0.20	0.08	1.33	0.33	0.33	0.33	0.33	0.33	0.33	2.00
Properties	Average diagonal width of Sb compound	0.19	0.17	0.12	0.34	0.11	0.03	0.03	0.03	0.16	0 66	0.72
	Standard deviation of diagonal width of Sb compound	0 21	0.15	0.09	0.32	0.05	0.01	0.01	0.01	0.14	0.75	0.84
	Flame retardance	A	A	A	A	A	A	A	A	A	A	A
	Touch (smoothness)	A	A	A	B	A	A	A	C	D	C	C
	Stickiness	A	A	B	A	A	B	B	B	A	A	A
	Color development	B	B	B	C	B	A	A	C	D	C	C
	Spinning stability	A	B	C	A	B	A	A	C	D	D	D

Ex.: Example, Com.Ex: Comparative example

The result in the above Table 1 illustrates that the polyester-based fibers in Examples 1-7 where compounds containing pentavalent antimony and having a pH of 3 to 10 are used as the antimony compounds and where the average diagonal widths of the antimony compounds in the fiber cross sections are 0.5 μm or less had a smooth touch similar to that of human hair with substantially no roughness. Furthermore, the polyester-based fibers in Examples 1-3 and 5-7 where the standard deviations of the diagonal widths of the antimony compounds in the fiber cross sections are 0.3 or less exhibited also excellent color development. In particular, the polyester-based fibers in Examples 6 and 7 where the average diagonal widths of the antimony compounds in the fiber cross sections are less than 0.1 μm exhibited remarkable color development. And, the polyester-based fibers in Examples 1, 2 and 5 where the average diagonal widths of the antimony compounds in the fiber cross sections are in the range of 0.1 to 0.5 μm and the standard deviations of the diagonal widths are in the range of 0.1 to 0.3 had an excellent smooth and dry touch without roughness and stickiness.

On the other hand, regarding the polyester-based fiber of Comparative example 1 using a compound containing pentavalent antimony and having a pH of less than 3 and the polyester-based fiber of Comparative example 2 using antimony trioxide, the brominated epoxy flame retardant aggregated due to the antimony compound having high acidity, which caused roughness and thus the touch was unfavorable, and the color development was also unfavorable. Similarly, regarding the polyester-based fibers in Comparative examples 3 and 4 where the average diagonal widths of the antimony compounds in the fiber cross sections exceed 0.5 μm, roughness occurred and the touch was degraded, and the color development was also unfavorable.

From the comparison between Examples 1-4 and Comparative example 4, it was confirmed that when the ratio Q/R of the discharge amount Q (kg/hour) to the screw rotation number R (rpm) of the extruder during the melt kneading is too large, the average diagonal width of the antimony compound in the fiber cross section exceeds 0.5 μm.

Explanation of letters and numerals

100: Fiber cross section (a cross section in the direction parallel to the fiber axis direction)

10: Antimony compound

The invention may be embodied in other forms without departing from the spirit or essential characteristics thereof. The embodiments disclosed in this application are to be considered in all respects as illustrative and not limiting. The scope of the invention is indicated by the appended claims rather than by the foregoing description, and all changes which come within the meaning and range of equivalency of the claims are intended to be embraced therein.

14 AOUT 2014

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WHAT IS CLAIMED IS:

1. A polyester-based fiber for artificial hair, comprising a polyester resin, a brominated epoxy flame retardant and an antimony compound,

the polyester resin is polyalkylene terephthalate and/or a copolymerized polyester containing polyalkylene terephthalate as a main component,

the antimony compound is a compound containing pentavalent antimony and having a pH of 3 to 10, and

the antimony compound has an average diagonal width of 0.5 μm or less in the fiber cross section parallel to the fiber axis direction.

2. The polyester-based fiber for artificial hair according to claim 1, wherein the average diagonal width of the antimony compound is 0.1 to 0.5 μm, and the standard deviation of diagonal width is 0.1 to 0.3 in the fiber cross section parallel to the fiber axis direction.

3. The polyester-based fiber for artificial hair according to claim 1, wherein the average diagonal width of the antimony compound is less than 0.1 μm in the fiber cross section parallel to the fiber axis direction.

4. The polyester-based fiber for artificial hair according to any one of claims 1 to 3, wherein the antimony compound is antimony pentoxide having a pH of 3 to 10.

5. A method for producing the polyester-based fiber for artificial hair according to any one of claims 1 to 4,

the method comprises melt kneading a polyester resin composition comprising a polyester resin, a brominated epoxy flame retardant and an antimony compound by an extruder,

during the melt kneading, a ratio Q/R of a discharge amount Q (kg/hour) and a screw rotation number R (rpm) of the extruder is 1.8 or less.

6. A fiber bundle for hair, comprising the polyester-based fiber for artificial hair according to any one of claims 1 to 4, and at least one fiber selected from the group consisting of human hair, animal hair, a polyvinyl chloride-based fiber, a modacrylic fiber, a polyamide-based fiber, a polyolefin-based fiber, a regenerated protein fiber and another polyester-based fiber.

7. A hair ornament product comprising the polyester-based fiber for artificial hair according to any one of claims 1 to 4.

8. The hair ornament product according to claim 7, further comprising at least one fiber selected from the group consisting of human hair, animal hair, a polyvinyl chloride-based fiber, a modacrylic fiber, a polyamide-based fiber, a polyolefin-based fiber, a regenerated protein fiber and another polyester-based fiber.

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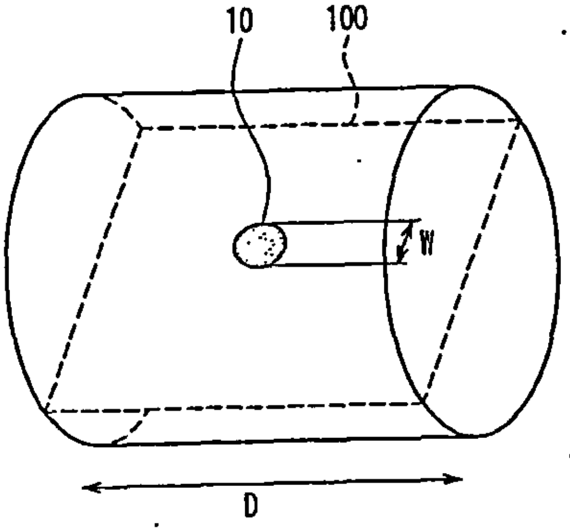

FIG. 1

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Planche de l'abrégé

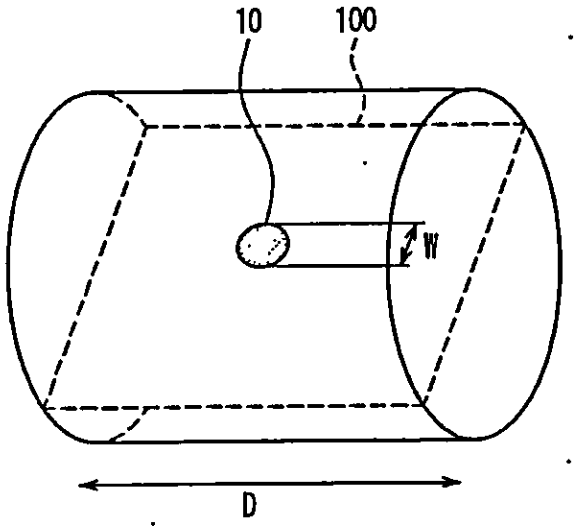

FIG. 1