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(54) FLAMEPROOF RAYON FIBER, PROCESS FOR PRODUCTION THEREOF, AND FLAMEPROOF FIBER STRUCTURE

(57) A flameproof rayon fiber having excellent flameproofness as well as excellent flame retardance, a method for manufacturing the same, and a flameproof fiber structure are provided. The flameproof rayon fiber according to the present invention includes components of silicon and sodium. Glass remains when the fiber is burned at 800°C, the glass component has a property of softening at 800°C, and when subjected to an X-ray fluorescence analysis, the rayon fiber has a silicon content in the range of 5 to 30% by mass and a sodium content in the range of 0.1 to 3% by mass. The flameproof rayon fiber according to the present invention can be manufactured by preparing an undiluted viscose solution; adding a solution containing a silicate compound containing an alkali metal to the undiluted viscose solution so as to make an alkali metal-containing silicate compound-added viscose solution; performing spinning by extruding the alkali metal-containing silicate compound-added viscose solution through a spinneret into a spinbath containing sulfuric acid, thus producing a fiber to be treated containing the silicate compound; and treating, in a scouring or aftertreatment process, the fiber to be treated with a solution having a pH in the range of 4 to 11 and a buffer action and containing sodium. A flameproof fiber structure of the present invention contains at least 30% by mass of the flameproof rayon fiber.

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Description

Technical Field

[0001] The present invention relates to a flameproof rayon fiber, a method for manufacturing the flameproof rayon fiber, and a flameproof fiber structure.

Background Art

[0002] Conventionally, for cellulose fibers having flame retardance and manufacturing methods thereof, a number of studies have been conducted to improve flame retardance by facilitating carbonization. Recently, several techniques utilizing the following have been proposed for flameproof cellulose fibers. By combining cellulose and glass with the use of a viscose rayon spinning technique, the glass remains even if the cellulose is decomposed, so that burning stops. For example, Patent document 1 proposes production of a composite fiber by mixing viscose and sodium silicate and spinning the mixture in a bath containing sulfuric acid. Patent document 2 proposes a cellulose fiber including aluminum, which is obtained by mixing sodium silicate with viscose and using sodium aluminate in the scouring process. Patent document 3 proposes a cellulose fiber including Mg, which is obtained by mixing sodium silicate with viscose and bring the fiber into contact with an alkali solution containing Mg in the scouring or aftertreatment process.

[0003] However, the cellulose fiber disclosed in Patent document 1 merely is a complex of cellulose and silicic acid. Thus, when the fiber is exposed to high temperatures, a glass skeleton is formed but decomposition of cellulose cannot be inhibited. Therefore, it is necessary to improve the flame retardance further. Further, while the cellulose fiber disclosed in Patent document 2 contains aluminum, it has been suggested that aluminum could be neurotoxic. Thus, a further improvement in safety is necessary. Further, when the cellulose fiber disclosed in Patent document 3 is exposed to high temperatures, a glass skeleton is formed, so that not only does the fiber exhibit a flameproof ability but also it has washing durability. However, in the case of the flameproof rayon of Patent document 3, efforts to perform Mg treatment may become complicated.

Prior art document

Patent document

[0004]

Patent document 1: British Patent No. 1,064,271
Patent document 2: Japanese Patent No. 3179104
Patent document 3: Japanese Patent No.4094052

Disclosure of Invention

Problem to be Solved by the Invention

[0005] With the foregoing in mind, the present invention provides a flameproof rayon fiber having excellent flame-proofness as well as excellent flame retardance, a method for manufacturing the flameproof rayon fiber and a flameproof fiber structure.

Means for Solving Problem

[0006] The flameproof rayon fiber according to the present invention is a rayon fiber that includes components of silicon and sodium. Glass remains in the rayon fiber when the rayon fiber is burned at 800°C. The glass has the property of softening at 800°C, and when subjected to an X-ray fluorescence analysis, the rayon fiber has a silicon content in the range of 5 to 30% by mass and a sodium content in a range of 0.1 to 3% by mass.

[0007] The method for manufacturing the flameproof rayon fiber according to the present invention includes preparing an undiluted viscose solution; adding a solution containing a silicic compound containing an alkali metal to the undiluted viscose solution so as to make an alkali metal-containing silicic compound-added viscose solution; performing spinning by extruding the silicic compound-added viscose solution through a spinneret into a spinbath containing a sulfuric acid, thus producing a fiber to be treated containing the silicic compound; and treating, in a scouring or aftertreatment process, the fiber to be treated with a solution having a pH in the range of 4 to 11 and a buffer action and containing sodium.

[0008] The flameproof fiber structure of the present invention contains at least 30% by mass of the flameproof rayon fiber.

Effects of the Invention

[0009] The flameproof rayon fiber according to the present invention exhibits excellent flameproof ability and self-extinguishability (flame retardance) because the rayon fiber contains components of silicon and sodium. Further, the Flameproof rayon fiber according to the present invention is not halogenic. Thus, even if the fiber is burned, gas that emerges due to the burning does not contain toxic substances such as cyan and halogen compounds. Moreover, since the principal component of the flameproof rayon fiber according to the present invention is rayon, the fiber is degradable in soil.

Brief description of the drawings

[0010]

[FIG. 1] FIG. 1 is a micrograph showing a flameproof rayon fiber of one example of the present invention being ashed at 800°C.
[FIG. 2] FIG. 2 is a micrograph showing a flameproof rayon fiber of another example of the present invention being ashed at 800°C.
[FIG. 3] FIG. 3 is a micrograph showing a flameproof rayon fiber of a comparative example being ashed at 800°C.
[FIG. 4] FIG. 4 is a micrograph showing a flameproof rayon fiber of another comparative example being ashed at 800°C.

Description of the Invention

[0011] In the present invention, flameproofness refers to a flame barrier property that can be achieved as a result of a glass skeleton remaining. More specifically, even when in contact with a flame, an afterflame time is short and a damaged area is small. Such an ability is useful for providing a property in which, for example, even when a cigarette smoked in bed falls onto a sheet, the sheet only becomes charred and the fire does not spread. Further, flame retardance refers to a property of having self-extinguishability and a fiber itself is resistant to burning. More specifically, it is such a property that even when fire is set to an opened fiber staple, the fire self extinguishes without causing a flash.

[0012] The flameproof rayon fiber according to the present invention contains components of silicon and sodium. The rayon fiber according to the present invention softens at temperatures lower than 1000°C, for example, at a temperature of about 800°C as the temperature of a burning cigarette, and has biodegradability. Since components other than the rayon component form compounds containing silicon and sodium (mainly, sodium silicate), the rayon fiber has a reduced load on the environment.

[0013] The rayon fiber is a fiber obtained by xanthating cellulose, followed by dilution and dissolution in dilute alkali so as to prepare viscose, and then coagulating and regenerating this viscose. The rayon fiber is not limited particularly by its material such as cellulose or manufacturing method.

[0014] Since the flameproof rayon fiber contains silicon and sodium, it is assumed that the flameproof rayon fiber forms a soda glass structure when it is burned and its softening point drops. Consequently, the glass softens quickly in high temperatures such as about 800°C, inhibiting decomposition of cellulose. Normally, when cellulose is burned, the burning continues because the gas resulting from the decomposition due to the heat is combustible. However, because the flameproof rayon fiber forms a soda glass structure when it is burned, decomposition of cellulose is inhibited to suppress the burning and the fire self extinguishes.

[0015] When subjected to an X-ray fluorescence analysis, the flameproof rayon fiber has a silicon content of 5 to 30% by mass, preferably in the range of 8 to 23% by mass, and more preferably in the range of 13 to 19% by mass. By setting the silicon content of the rayon fiber according to the present invention to the mentioned range, it is possible to maintain the strength and texture of the rayon fiber.

[0016] When subjected to an X-ray fluorescence analysis, the flameproof rayon fiber has a sodium content of 0.1 to 3% by mass, preferably in the range of 0.15 to 1.5% by mass, and more preferably in the range of 0.2 to 1.0% by mass. By setting the sodium content in the flameproof rayon fiber according to the present invention to the mentioned range, it is possible to achieve a flameproof rayon fiber having more favorable flameproofness and self-extinguishability.

[0017] Further, when subjected to an X-ray fluorescence analysis, the ratio of the silicon content to the sodium content (mass ratio of silicon/sodium) in the flameproof rayon fiber is preferably 10 or more and less than 90. The mass ratio of silicon/sodium is a parameter that indicates the susceptibility of the fiber to softening. The smaller the mass ratio of silicon/sodium, the likelier it is for the flameproof rayon fiber to soften when being burned due to the formation of soda glass within the fiber, and thus, the self-extinguishability (flame retardance) improves. The mass ratio of silicon/sodium is more preferably 15 to 70. When the mass ratio of silicon/sodium is less than 90, the chance of sodium silicate ($xNa_2 \cdot ySiO_2 \cdot zH_2O$; where x is 1 to 5, y ≥ x, and z is 1 to 3) being formed is relatively high, so that favorable flame retardance can be achieved. Further, when the mass ratio of silicon/sodium is 10 or more, the flameproof rayon fiber softens while leaving a glass skeleton, so that favorable flameproofness and flame retardance can be achieved.

[0018] Sodium may be present in the flameproof rayon fiber such that at least part thereof is contained in the rayon fiber and the remaining part is adhered to the surface of the rayon fiber. Whether sodium is present in the rayon fiber (inside the fiber) or not can be determined by washing the fiber with water. The silicon and the sodium compound are not limited particularly by which state they are in. They may be mixed uniformly in the fiber or may be present in a compatible or incompatible state. As long as the sodium is partially present in the form of a sodium compound such as sodium silicate, the remainder may be contained in the form of sodium salt such as sodium oxide and sodium hydroxide.

[0019] The flameproof rayon fiber has an ash content preferably in the range of 10 to 50% by mass, more preferably in the range of 15 to 40% by mass, and particularly preferably in the range of 25 to 38% by mass. Here, the ash content refers to an inorganic material left as a remainder after an organic material is incinerated at high temperatures. When the ash content is less than 10% by mass, the flameproofness of the flameproof rayon fiber tends to drop. In contrast, when the ash content exceeds 50% by mass, the strength of the flameproof rayon fiber tends to drop or the texture thereof tends to be impaired. Further, when the ash content exceeds 40% by mass, it tends to be difficult to achieve the same texture as conventional rayon fibers that do not use a flame retardant. Therefore, by setting the ash content of the flameproof rayon fiber according to the present invention to the mentioned range, it is possible to achieve a flameproof rayon fiber having favorable flameproofness and favorable texture. In the present invention, the ash content of the flameproof rayon fiber is measured in conformity with JIS L 1015 8.20 and is a value expressed as percent by mass of the mass of a component remaining after burning a flameproof rayon fiber at 850°C with respect to an absolute dry mass of the flameproof rayon fiber. The same holds true for the following.

[0020] The flameproof rayon fiber has an LOI value of preferably 31 or more, and more preferably 32 or more by twisted fiber string measurement (E-1) in conformity with JIS L 1091 E (oxygen index test). Further, the flameproof rayon fiber has an LOI value of preferably 23 or more, and more preferably 24 or more by nonwoven fabric measurement (E-2) in conformity with JIS L 1091 E (oxygen index test). The LOI values of the rayon fiber according to the present invention satisfy the mentioned ranges, respectively. Thus, the flameproof rayon fiber is preferable because it has flame retardance as well as flameproofness.

[0021] The flameproof rayon fiber has an L value (whiteness) of preferably 40 to 90, more preferably 44 to 86, and particularly preferably 48 to 70. The L value is a whiteness indicator with a scale of 0 (black) to 100 (white). As the value is larger and positive, the color becomes whiter. Although the L value of 100 means that the color is white, the whiteness of typical rayon fibers is about 92 to 95. Due to a change in the hue of cellulose at the time of heating, the color does not become pure white. Therefore, it tends to be difficult to produce rayon fibers having an L value of more than 90. As for rayon fibers having an L value of less than 40, their hue tends to deteriorate when they are processed in the form of product, so that the product value tends to drop.

[0022] The flameproof rayon fiber is not particularly limited by its fineness and generally has a fineness in the range of 1 to 17 dtex, and preferably in the range of 1.7 to 10 dtex. When the fineness is less than 1 dtex, the strength of the rayon fiber tends to drop. When the fineness exceeds 17 dtex, the thickness of the fiber becomes excessively large, so that the fiber tends to be coarse. Also, the flameproof rayon fiber is not particularly limited by its length, either, and can be used as a filament or a staple. The fiber length can be set freely, and the fiber with a length of 5 to 20 mm can be used as a paper screen, a wallpaper or the like and that with a length of 20 to 200 mm can be used for a nonwoven fabric or a spun yarn. A filament tow can be used without cutting after the scouring.

[0023] The cross-section of the flameproof rayon fiber is not particularly limited by its shape but can be selected suitably according to the intended use. For example, a circular shape, a deformed circular shape, a hollow shape, an oblate shape, etc. can be selected.

[0024] The flameproof rayon fiber according to the present invention has useful physical properties that rayon as regenerated cellulose generally has, such as biodegradability, water absorptivity, hygroscopicity, antistatic property and thermal stability. Since rayon as the principal component of the flameproof rayon fiber according to the present invention has biodegradability, it can be decomposed within one to three months when buried in the soil. Further, the components other than rayon are compounds principally containing silicic acid and sodium (mainly, sodium silicate). Therefore, the flameproof rayon fiber according to the present invention has a reduced load on the environment.

[0025] The flameproof rayon fiber according to the present invention can be obtained as follows. First, a silicic compound containing an alkali metal, for example, sodium silicate ($Na_2O \cdot nSiO_2 \cdot xH_2O$; where n is 1 to 3 and x is 10 to 20) is added to an undiluted viscose solution to prepare an alkali metal-containing silicic compound-added viscose solution (hereinafter, simply referred to as the viscose solution). Then, spinning is carried out by extruding the viscose solution through a spinneret into a spinbath containing a sulfuric acid (H_2SO_4) to produce a fiber to be treated containing the silicic compound. During the spinning process, the silicic compound containing an alkali metal, for example, sodium silicate ($Na_{2O} \cdot nSiO_2 \cdot xH_2O$; where n is 1 to 3 and x is 10 to 20) in the viscose solution reacts with the sulfuric acid (H_2SO_4) and turns into silicon dioxide (SiO_2; in the form of polymer). Subsequently, in a scouring or aftertreatment process, the obtained fiber is treated with a solution having a pH in the range of 4 to 11 and a buffer action and containing sodium, thus obtaining the flameproof rayon fiber according to the present invention. As a result of this treatment, silicon and sodium react with each other and form a compound. It is estimated that the compound containing silicon and sodium has the following structure in the rayon fiber. In the rayon fiber, silicic acid forms a layered structure and sodium in the form of sodium oxide is present between the layers of the unit structure. The silicic acid and the sodium oxide are bonded to each other due to sharing some part of oxygen, so that a gel of silicic acid and sodium is produced to form sodium silicate ($xNa_2O \cdot ySiO_2 \cdot zH_2O$; where x is 1 to 5, $y \geq x$, and z is 1 to 3). On the other hand, conventional flameproof rayon fiber manufacturing is carried out in the same manner as the manufacturing method according to the present invention until the step where sodium silicate reacts with sulfuric acid and turns into silicon dioxide. However, since large sulfur content will remain if nothing is done, the sulfur content has to be removed through a scouring process. Since sodium remaining in the fiber also is removed from the system during the scouring process, sodium and silicic acid are not bonded to each other in the fiber.

[0026] The spinbath can be a general acid spinbath containing sulfuric acid, for example, a Muller bath containing H_2SO_4, $ZnSO_4$ and Na_2SO_4 in the ranges of 110 to 170 g/liter, 10 to 30 g/liter and 150 to 350 g/liter, respectively. Further, the temperature of the spinbath generally is 45 to 65°C.

[0027] The undiluted viscose solution may have a general composition. For example, it is possible to use an undiluted viscose solution containing cellulose, NaOH and CS_2 in the ranges of 5 to 15% by mass, 5 to 10% by mass and 1 to 5% by mass, respectively.

[0028] The silicic compound containing an alkali metal preferably is in the range of 10 to 100% by mass and more preferably in the range of 25 to 70% by mass on the basis of silicon dioxide (SiO_2) with respect to the mass of cellulose contained in the undiluted viscose solution. Since the silicic compound containing an alkali metal in the viscose solution is considered to react with the sulfuric acid (H_2SO_4) and turn into silicon dioxide (SiO_2; in the form of polymer), the values are expressed on the basis of silicon dioxide (SiO_2). The silicon dioxide contained in the mentioned range makes it possible to maintain the strength and texture of the fiber, so that, when treated with the sodium-containing solution, a rayon fiber having favorable flameproofness can be manufactured.

[0029] The silicate compound containing an alkali metal can be, for example, sodium silicate. The process of adding the silicate compound containing an alkali metal such as sodium silicate may be carried out by mixing an aqueous solution of the silicate compound containing an alkali metal in a general undiluted viscose solution.

[0030] The ratio of the sodium silicate to be added is preferably in the range of 10 to 100% by mass, more preferably in the range of 15 to 80% by mass and particularly preferably in the range of 30 to 70% by mass on the basis of SiO_2 with respect to cellulose in the undiluted viscose solution. By setting the amount of sodium silicate to the mentioned range, it is possible to adjust the amount of silicon dioxide contained in the fiber to be treated to the amount suitable for the flameproof rayon fiber according to the present invention. For the sodium silicate, sodium silicate No. 3 (JIS K 1408) can be used, for example.

[0031] Also, in the scouring or aftertreatment process, the fiber to be treated containing a silicon component that has been obtained in the spinning process is treated with the solution having a pH in the range of 4 to 11 and a buffer action and containing sodium, thereby allowing the silicon and the sodium to react with each other, so that a compound containing silicon and sodium is formed. The compound containing silicon and sodium is believed to form sodium silicate. For example, there are a treatment of bringing the fiber to be treated into contact with the sodium-containing solution having a buffer action in place of sulfuric acid after bleaching during the scouring process; a treatment of bringing the fiber to be treated into contact with the sodium-containing solution having a buffer action after scouring during the scouring process; a treatment of mixing an oil solution component with the sodium-containing solution having a buffer action and bringing the fiber to be treated into contact with the mixed solution in an oil solution treatment in the scouring process; and a treatment of steeping the fiber to be treated in the sodium-containing solution having a buffer action (as an aftertreatment process) after scouring and drying the fiber to be treated. At this time, the bath ratio may be selected suitably in accordance with the sodium-containing solution having a buffer action to be used, and the mass of the fiber to be treated: the mass of the solution is in the range of 1 : 10 to 1:1000, for example. Further, it is generally possible to carry out favorable treatment when the bath temperature is in the range of 0 to 100°C and the steeping time is about 30 sec, and preferably in the range of 20 to 300 sec.

[0032] The sodium-containing solution having a buffer action may have a pH in the range of 4 to 11, preferably in the range of 6 to 10, more preferably in the range of 7 to 8.6, and particularly preferably in the range of 7.3 to 8.6. When the pH is less than 4, sodium does not penetrate the fiber, so that self-extinguishability cannot be achieved. On the other hand, when the pH exceeds 11, the silicic acid content in the fiber leaches out, resulting in a low ash content after the treatment. Consequently, it becomes difficult to achieve the flameproofness.

[0033] Further, for bringing sodium into reaction with silicic acid in the fiber in an efficient manner, the sodium-containing solution having a buffer action is preferably an aqueous solution having a pH in the range of 4 to 11. In the present invention, the "sodium-containing solution having a buffer action" refers to a solution having a buffer action, in other words, a buffer solution containing sodium and having a pH in the range of 4 to 11 and the solution may be in any form. For example, it is possible to use a buffer solution containing water-soluble sodium salt having no buffer action and sodium salt having a buffer action, a buffer solution containing sodium salt having a buffer action, and a buffer solution containing water-soluble sodium salt having no buffer action and an agent having a buffer action such as weak acid or weak base. In particular, a buffer solution containing sodium salt having a buffer action is preferable in terms of taking sodium into the fiber in an efficient manner. As the water-soluble sodium salt having no buffer action, sodium chloride, sodium sulfate, sodium nitrate or the like can be used. Examples of sodium salts having a buffer action include sodium hydrogen carbonate (baking soda), sodium carbonate, disodium hydrogen phosphate, sodium dihydrogen phosphate, and sodium citrate.

In particular, a buffer solution of alkaline sodium carbonate or sodium hydrogen carbonate is more preferable in terms of applications where inclusion of phosphorus components is undesirable or in terms of pH. Further, these sodium salts can be used in combination of two or more.

[0034] The sodium salt content of the sodium-containing solution having a buffer action is preferably defined by the concentration of sodium ions in terms of controlling the solution. The concentration of sodium ions in the sodium-containing solution having a buffer action is preferably in the range of 500 to 10,000 mg/L, and more preferably in the range of 1,000 to 8,000 mg/L.

[0035] As a result of the inclusion of the flameproof rayon fiber, a flameproof fiber structure such as woven fabric, knit fabric and nonwoven fabric can be obtained. The content of the flameproof rayon fiber in the flameproof fiber structure is preferably 30% by mass, and more preferably 60 to 80% by mass. When the content of the flameproof rayon fiber is 30% by mass or more, it is possible to obtain a flameproof fiber structure having excellent flameproofness and flame retardance. Other fibers used in the flameproof fiber structure are not particularly limited, and examples of the other fibers include binder fibers such as low-melting polyester fibers, flame retardant acrylic (modacrylic) fibers such as "Kanekaron" (trade name, manufactured by Kaneka Corporation) and nonflammable fibers such as aramid (aromatic polyamide) fibers.

[0036] As described above, the flameproof rayon fiber according to the present invention is a rayon fiber having favorable flameproofness and flame retardance. Further, the rayon fiber has an excellent texture, resistance to dry-cleaning and biodegradability. The flameproof rayon fiber according to the present invention is processed into woven fabric, knit fabric, nonwoven fabric, etc. and useful for the purposes such as disaster prevention items, kitchen fan filters, sheets, pillow cases, bedding mats, bedding covers, fire protection screens, interior goods (carpets, chair coverings, curtains, wall paper bases, wall materials, etc.), vehicle interior materials (mats, lining fabric, etc.), etc., for example.

Examples

[0037] Hereinafter, the present invention will be described more specifically by way of Examples. It should be noted that the present invention is not limited to the following Examples.

(Example 1)

(1) Manufacture of viscose solution

[0038] An undiluted viscose solution containing 8.5% by mass of cellulose, 5.7% by mass of sodium hydroxide and 2.6% by mass of carbon disulfide was produced. Then, a mixture solution of No. 3 sodium silicate, sodium hydroxide and water was added to the produced undiluted viscose solution such that cellulose and sodium hydroxide accounted for 7.2% by mass and 7.4% by mass of the composition of the viscose solution, respectively, thus making a sodium silicate-added viscose solution. The ratio of added sodium silicate was 50% by mass on the basis of SiO_2 with respect to the mass of cellulose.

(2) Spinning

[0039] The sodium silicate-added viscose solution was spun at a spinning speed of 50 m/min and at a stretch ratio of 50% by two bath stretch spinning, thus obtaining fibers having a fineness of about 3.3 dtex. The composition of a first bath (spinbath) was such that a sulfuric acid accounted for 115 g/liter, zinc sulfate accounted for 15 g/liter and sodium sulfate accounted for 350 g/liter, and the temperature of the first bath was 48°C and the temperature of a second bath (hot water bath) was set to 85°C. The sodium silicate-added viscose solution was extruded through a spinneret, thus producing a silicon-containing rayon filament tow (fibers to be treated).

(3) Scouring

[0040] The filament tow cut into a fiber length of 51 mm with a cutter was used in the scouring process. The scouring process included a hot water treatment, bleaching, souring and water-washing in this order. Excess moisture was removed by compression rollers, followed by drying for seven hours in a constant-temperature dryer at 60°C. The resultant fibers to be treated had physical properties such as a fineness of 3.3 dtex, a dry strength of 1.4 cN/dtex, a wet strength of 0.8 cN/dtex, a dry elongation of 25% and a wet elongation of 20%.

(4) Aftertreatment

[0041] As a sodium-containing solution having a buffer action (hereinafter referred to as a sodium-based buffer solution), an aqueous solution (bath temperature: 50°C, pH: 7.76) containing 0.38% by mass of sodium sulfate and 0.05% by mass of sodium hydrogen carbonate was used. In this aqueous solution, the dried fibers to be treated were steeped for 30 sec. At this time, the bath ratio was set such that the mass of the fibers to be treated: the mass of the aqueous solution was 1 : 20. Next, the fibers to be treated were washed with water and then dewatered centrifugally. Finally, the fibers were dried in a constant-temperature dryer at 105°C for 30 minutes, thus obtaining flameproof rayon fibers b of Example 1 (in the following, referred to as the fibers b).

(Example 2)

[0042] Flameproof rayon fibers c (hereinafter referred to as the fibers c) of Example 2 were manufactured in the same manner as Example 1 except that an aqueous solution (bath temperature: 50°C, pH: 7.79) containing 0.34% by mass of sodium sulfate and 0.1% by mass of sodium hydrogen carbonate was used as a sodium-based buffer solution in the aftertreatment.

(Example 3)

[0043] Flameproof rayon fibers d (hereinafter referred to as the fibers d) of Example 3 were manufactured in the same manner as Example 1 except that an aqueous solution (bath temperature: 50°C, pH: 7.93) containing 0.17% by mass of sodium sulfate and 0.3% by mass of sodium hydrogen carbonate was used as a sodium-based buffer solution in the aftertreatment.

(Example 4)

[0044] Flameproof rayon fibers e (hereinafter referred to as the fibers e) of Example 4 were manufactured in the same manner as Example 1 except that an aqueous solution (bath temperature: 50°C, pH: 7.31) containing 0.41% by mass of sodium sulfate and 0.01% by mass of sodium hydrogen carbonate was used as a sodium-based buffer solution in the aftertreatment.

(Example 5)

[0045] Flameproof rayon fibers f (hereinafter referred to as the fibers f) of Example 5 were manufactured in the same manner as Example 1 except that an aqueous solution (bath temperature: 50°C, pH: 8.40) containing 0.1% by mass of sodium hydrogen carbonate was used as a sodium-based buffer solution in the aftertreatment.

(Example 6)

[0046] Flameproof rayon fibers g (hereinafter referred to as the fibers g) of Example 6 were manufactured in the same manner as Example 1 except that an aqueous solution (bath temperature: 50°C, pH: 8.42) containing 0.5% by mass of sodium hydrogen carbonate was used as a sodium-based buffer solution in the aftertreatment.

(Example 7)

[0047] Flameproof rayon fibers h (hereinafter referred to as the fibers h) of Example 7 were manufactured in the same manner as Example 1 except that an aqueous solution (bath temperature: 50°C, pH: 8.43) containing 1.0% by mass of sodium hydrogen carbonate was used as a sodium-based buffer solution in the aftertreatment.

(Example 8)

[0048] Flameproof rayon fibers i (hereinafter referred to as the fibers i) of Example 8 were manufactured in the same manner as Example 1 except that an aqueous solution (bath temperature: 50°C, pH: 4.69) containing 0.5% by mass of sodium dihydrogen phosphate was used as a sodium-based buffer solution in the aftertreatment.

(Example 9)

[0049] Flameproof rayon fibers j (hereinafter referred to as the fibers j) of Example 9 were manufactured in the same manner as Example 1 except that an aqueous solution (bath temperature: 50°C, pH: 4.53) containing 1.0% by mass of sodium dihydrogen phosphate was used as a sodium-based buffer solution in the aftertreatment.

(Example 10)

[0050] Flameproof rayon fibers k (hereinafter referred to as the fibers k) of Example 10 were manufactured in the same manner as Example 1 except that an aqueous solution (bath temperature: 50°C, pH: 4.24) containing 3.0% by mass of sodium dihydrogen phosphate was used as a sodium-based buffer solution in the aftertreatment.

(Example 11)

[0051] Flameproof rayon fibers l (hereinafter referred to as the fibers 1) of Example 11 were manufactured in the same manner as Example 1 except that an aqueous solution (bath temperature: 50°C, pH: 10.86) containing 0.5% by mass of sodium carbonate was used as a sodium-based buffer solution in the aftertreatment.

(Example 12)

[0052] Flameproof rayon fibers m (hereinafter referred to as the fibers m) of Example 12 were manufactured in the same manner as Example 1 except that an aqueous solution (bath temperature: 50°C, pH: 8.70) containing 0.5% by mass of disodium hydrogen phosphate was used as a sodium-based buffer solution in the aftertreatment.

(Example 13)

[0053] Flameproof rayon fibers n (hereinafter referred to as the fibers n) of Example 13 were manufactured in the same manner as Example 1 except that an aqueous solution (bath temperature: 50°C, pH: 8.76) containing 1.0% by mass of disodium hydrogen phosphate was used as a sodium-based buffer solution in the aftertreatment.

(Example 14)

[0054] Flameproof rayon fibers o (hereinafter referred to as the fibers o) of Example 14 were manufactured in the same manner as Example 1 except that an aqueous solution (bath temperature: 50°C, pH: 8.79) containing 3.0% by mass of disodium hydrogen phosphate was used as a sodium-based buffer solution in the aftertreatment.

(Example 15)

[0055] Flameproof rayon fibers p (hereinafter referred to as the fibers p) of Example 15 were manufactured in the same manner as Example 1 except that an aqueous solution (bath temperature: 50°C, pH: 8.3) containing 0.2M of disodium hydrogen phosphate and 0.2M of sodium dihydrogen phosphate at a ratio (volume ratio) of 94.7: 5.3 was used as a sodium-based buffer solution in the aftertreatment.

(Example 16)

[0056] Flameproof rayon fibers q (hereinafter referred to as the fibers q) of Example 16 were manufactured in the same manner as Example 1 except that an aqueous solution (bath temperature: 50°C, pH: 8.0) containing 0.2M of disodium hydrogen phosphate and 0.2M of sodium dihydrogen phosphate at a ratio (volume ratio) of 91.5: 8.5 was used as a sodium-based buffer solution in the aftertreatment.

(Example 17)

[0057] Flameproof rayon fibers r (hereinafter referred to as the fibers r) of Example 17 were manufactured in the same manner as Example 1 except that an aqueous solution (bath temperature: 50°C, pH: 7.6) containing 0.2M of disodium hydrogen phosphate and 0.2M of sodium dihydrogen phosphate at a ratio (volume ratio) of 81: 19 was used as a sodium-based buffer solution in the aftertreatment.

(Example 18)

[0058] Flameproof rayon fibers s (hereinafter referred to as the fibers s) of Example 18 were manufactured in the same manner as Example 1 except that an aqueous solution (bath temperature: 50°C, pH: 7.2) containing 0.2M of disodium hydrogen phosphate and 0.2M of sodium dihydrogen phosphate at a ratio (volume ratio) of 61: 39 was used as a sodium-based buffer solution in the aftertreatment.

(Example 19)

[0059] Flameproof rayon fibers t (hereinafter referred to as the fibers t) of Example 19 were manufactured in the same manner as Example 1 except that an aqueous solution (bath temperature: 50°C, pH: 6.8) containing 0.2M of disodium hydrogen phosphate and 0.2M of sodium dihydrogen phosphate at a ratio (volume ratio) of 37.5: 62.5 was used as a sodium-based buffer solution in the aftertreatment.

(Example 20)

[0060] Flameproof rayon fibers u (hereinafter referred to as the fibers u) of Example 20 were manufactured in the same manner as Example 1 except that an aqueous solution (bath temperature: 50°C, pH: 6.3) containing 0.2M of disodium hydrogen phosphate and 0.2M of sodium dihydrogen phosphate at a ratio (volume ratio) of 18.5: 81.5 was used as a sodium-based buffer solution in the aftertreatment.

(Comparative Example 1)

[0061] Flameproof rayon fibers a (hereinafter referred to as the fibers a) of Comparative Example 1 were manufactured in the same manner as Example 1 except that the fibers to be treated were not subjected to aftertreatment with an aqueous solution containing sodium.

(Comparative Example 2)

[0062] Commercially available rayon fibers (trade name: "HELON", manufactured by Shandong Helon Co., LTD, hereinafter simply referred to as HELON) were used as Comparative Example 2.

(Comparative Example 3)

[0063] Flameproof rayon fibers v (hereinafter referred to as the fibers v) of Comparative Example 3 were manufactured in the same manner as Example 1 except that an aqueous solution (bath temperature: 50°C, pH: 7.8) containing 3% by mass of sodium sulfate was used in the aftertreatment.

[0064] For the flameproof fibers of Examples 1 to 20 and Comparative Examples 1 to 3, a performance test was conducted as follows. Tables 1 and 2 provide the results of the performance test.

(Performance Test)

(1) Ash content

[0065] The ash content was measured in conformity with JIS L 1015 8.20. Specifically, the mass of a component remaining after burning each of the fibers having a mass of 1 g for two hours in an electric furnace at 850°C was measured so as to determine the ash content of each of the fibers. Incidentally, the ash content is expressed as a percent by mass of the mass of the residual component after burning with respect to the mass obtained by subtracting a water content from the mass of the fibers. Further, after the fibers were washed with water, their ash contents were determined in the same manner. The water-washing was carried out as follows.

[Water-washing]

[0066] To the water-washing, a water absorption test method (weaved basket method) as one of the purity test methods for absorbent cotton defined by the Japanese Pharmacopoeia was applied. Specifically, 2 g of fibers were weighed and they were put into a container. As the container, a cylindrical basket processed with an enameled wire and having a height of 8 cm and a diameter of about 5 cmϕ was used. After putting the fibers into the container uniformly, the container was steeped in ion-exchanged water at 25°C for three minutes. Subsequently, the fibers were taken out from the container and dewatered centrifugally, followed by drying in a drier. The dried fibers were used as a water-washed sample.

(2) Flame Retardance

[0067] The fibers were spread flat into a plate shape, subjected directly to a flame of a disposable lighter (the flame length: 2.5 cm) that was located 2 cm below them, and observed. The flame was applied perpendicularly to the fiber mass. It is noted that each evaluation sample (fiber mass) was produced by opening 1 to 2 g of cut fibers into a web using a carding machine and rendering this web in the form of fiber mass. Further, the fibers were water-washed in the manner described above, subjected to a flame in the same manner and observed. On the basis of the observation results, the flame retardance was evaluated on a scale from A to D as follows.

A: when a flame was brought close to a fiber mass, only the part to which the flame was applied burned and the remaining part did not burn.
B: when a flame was brought close to a fiber mass, fire traveled somewhat on the surface of the fiber mass but went out when the flame was moved away.
C: when a flame was brought close to a fiber mass, fire traveled on the surface of the fiber mass and the fire remained even when the flame was moved away.
D: when the flame was brought close to a fiber mass, fire spread

In the above, general rayon fibers were evaluated as D as a result of observing them. They were manufactured by a general manufacturing method where sodium silicate was not added to viscose and aftertreatment with an aqueous solution containing sodium was not performed.

(Whiteness)

[0068] The whiteness (L value) was measured in conformity with JIS L 10 15 8.17 C (by Hunter) as follows. 20 g of fibers opened with a carding machine were placed in a constant-temperature ventilation drier (trade name: "FC-612", manufactured by Advantec Toyo Kaisha, Ltd.) set at a temperature of 190°C for heat treatment for five minutes, thus producing a sample. A whiteness meter "ZE-2000" manufactured by Nippon Denshoku Industries Co., LTD. was used to measure the whiteness. 20 g of the produced sample was put into a container included with the whiteness meter and the orientation of the sample was changed to measure the color four times (L, a, b). The average of the values (L values) obtained from the four measurements was adopted as the fiber whiteness.

(Measurement of LOI)

[0069] In conformity with JIS L 1091 E (oxygen index test) and with the use of an oxygen index flammability tester (ON-1 type) manufactured by Toyo Rika Kogyo Co., LTD, a twisted fiber string (E-1) or nonwoven fabric (E-2) as a test piece was attached to a holder to measure the LOI value. The test piece was produced as follows.

[0070] E-1 test piece (twisted fiber string): 1 g of a sample staple was opened and adjusted to have a fiber length of 20 to 30 cm. Then, the fibers were fixed on one end and twists were applied to the fibers from the other end. Specifically, twists were applied to the fibers while pulling the fibers, and the application of twists was stopped immediately before the emergence of bumps. Subsequently, the twisted fibers were folded into two parts at the center, thus producing a twisted fiber string having a length of about 110 mm and a width of about 6 mm.

[0071] E-2 test piece (nonwoven fabric): 30% by mass of low-melting polyester fibers (trade name: "4080", manufactured by Unitika LTD., fineness: 4.4 dtex, fiber length: 51 mm) and 70% by mass of the flameproof rayon fibers were mixed with each other, the mixture was rendered in the form of card webs using a carding machine, and the card web was placed on another such that the total mass per unit area reached 300 g/m^2. Subsequently, the card webs were placed on a punching plate, a nylon mesh was placed on top of the card webs, and a weight was placed on top of the nylon mesh such that a load of 20 g/cm^2 was applied thereto. Then, they were placed in a constant-temperature ventilation drier (trade name: "FC-612", manufactured by Advantec Toyo Kaisha, Ltd.) set at a temperature of 180°C. After being set aside for 10 minutes in the drier, they were taken out and thus obtaining a nonwoven fabric having a length of 150 mm and a width of 60 mm.

(Ashing)

[0072] The fibers were set aside in an electric furnace set to a default temperature of 800°C and ashing of the fibers was observed with a microscope (trade name: "ECLIPSE E600", manufactured by Nikon Corporation, magnification: 320x) to check the presence or absence of softening and the presence or absence of bubbles.

[0073]

[TABLE 1]

			Aqueous solution (bath) used in aftertreatment				After aftertreatment		After water-washing			Ashing (800°C)
			Composition	Concentration of each composition (% by mass)	pH	Concentration of Na^+ (mg/L)	Ash (% by mass)	Flame retardance evaluation	Ash content (% by mass)	Flame retardance evaluation	Whitness (L value)	
Comp. EX.	1	fibers a	(untreated)	-	-	-	29.5	C	29.2	C	88.05	not softened no bubbles
	2	HELON	-	-	-	-	31.2	B to C	-	-	74.18	not softened no bubbles
	3	fibers v	Na_2SO_4	1.0	7.9	3239	31.0	C	-	C	-	not softened no bubbles
Ex.	1	fibers b	Na_2SO_4/ $NaHCO_3$	0.38/0.05	7.76	1368	29.2	A	29.2	A to B	68.07	partially softened bubbles partially present
	2	fibers c	Na_2SO_4/ $NaHCO_3$	0.34/0.10	7.79	1375	29.5	A	29.5	A to B	67.85	softened small surface asperities bubbles present
	3	fibers d	Na_2SO_4/ $NaHCO_3$	0.17/0.3	7.93	1372	29.6	A	29.1	A to B	54.38	partially softened bubbles present

(continued)

			Aqueous solution (bath) used in aftertreatment				After aftertreatment		After water-washing			Ashing (800°C)
			Composition	Concentration of each composition (% by mass)	pH	Concentration of Na^+ (mg/L)	Ash (% by mass)	Flame retardance evaluation	Ash content (% by mass)	Flame retardance evaluation	Whitness (L value)	
	4	fibers e	Na_2SO_4/ $NaHCO_3$	0.41/0.01	7.31	1356	30.0	B	29.5	B	78.69	softened surface asperities present bubbles partially present
	5	fibers f	$NaHCO_3$	0.1	8.40	274	29.4	B	28.8	B	76.08	softened surface asperities present bubbles present
	6	fibers g	$NaHCO_3$	0.5	8.42	1369	30.9	A	28.7	A to B	58.06	partially softened no bubbles
	7	fibers h	$NaHCO_3$	1.0	8.43	2737	38.2	A	33.0	A	48.30	softened large surface asperities bubbles present
	8	fiber i	NaH_2PO_4	0.5	4.69	958	30.0	B	-	-	83.74	partially softened bubbles present

(continued)

			Aqueous solution (bath) used in aftertreatment				After aftertreatment		After water-washing			Ashing (800°C)
			Composition	Concentration of each composition (% by mass)	pH	Concentration of Na^+ (mg/L)	Ash (% by mass)	Flame retardance evaluation	Ash content (% by mass)	Flame retardance evaluation	Whitness (L value)	
	9	fibers j	NaH_2PO_4	1.0	4.53	1916	29.4	B	-	-	81.39	softened small surface asperities bubbles present
	10	fibers k	NaH_2PO_4	3.0	4.24	5748	30.7	B	-	-	85.35	partially softened bubbles partially present
	11	fibers l	Na_2CO_3	0.5	10.86	2170	40.3	A	-	-	39.70	partially softened bubbles present
	12	fibers m	Na_2HPO_4	0.5	8.70	1620	29.4	A	-	-	55.33	partially softened no bubbles
	13	fibers n	Na_2HPO_4	1.0	8.76	3239	29.9	A	-	-	60.26	softened small surface asperities minute bubbles present

(continued)

			Aqueous solution (bath) used in aftertreatment				After aftertreatment		After water-washing			Ashing (800°C)
			Composition	Concentration of each composition (% by mass)	pH	Concentration of Na^+ (mg/L)	Ash (% by mass)	Flame retardance evaluation	Ash content (% by mass)	Flame retardance evaluation	Whitness (L value)	
	14	fibers o	Na_2HPO_4	3.0	8.79	9717	35.6	A	-	-	47.41	partially softened no bubbles
	15	fibers p	Na_2HPO_4/ NaH_2PO_4	2.69/0.13	8.30	4476	31.3	A	-	-	-	partially softener bubbles present
	16	fibers q	Na_2HPO_4/ NaH_2PO_4	2.60/0.20	8.00	4402	32.2	A	-	-	-	partially softened bubbles present
	17	fibers r	Na_2HPO_4/ NaH_2PO_4	2.30/0.46	7.60	4161	30.9	A	-	-	-	partially softened bubbles present
	18	fibers s	Na_2HPO_4/ NaH_2PO_4	1.73/0.94	7.20	3702	31.1	A	-	-	-	partially present bubbles present
	19	fibers t	Na_2HPO_4/ NaH_2PO_4	1.07/1.50	6.80	3161	30.5	A to B	-	-	-	partially softened no bubbles
	20	fibers u	Na_2HPO_4/ NaH_2PO_4	0.53/1.96	6.30	2725	31.0	B	-	-	-	partially softened no bubbles

[0074]

[TABLE 2]

		LOI value	
		Twisted fiber string (E-1 method)	Nonwoven fabric (E-2 method)
Ex.	fibers b	32.0	24.2
	fibers g	32.9	25.4
	fibers l	38.2	30.2
	fibers m	33.3	24.8
Comp. Ex.	fibers a	29.8	18.0
	HELON	30.3	18.8

[0075] As can be seen from Tables 1 and 2, the flameproof rayon fibers of Examples had an LOI value of 31 or more when measured in the form of a twisted fiber string (E-1 method) and an LOI value of 24 or more when measured in the form of a nonwoven fabric (E-2 method). Thus, they had excellent flame retardance.

[0076] FIGS. 1 to 4 are micrographs respectively showing the flameproof rayon fibers of Examples 2 and 5 and Comparative Examples 1 and 2 being ashed at 800°C. As can be seen from FIGS. 1 to 4 and Table 1, the fibers of Comparative Examples were not softened at 800°C and no bubbles were observed. On the other hand, the fibers of Examples softened and bubbles were observed. That is, the fibers of Examples formed a soda glass structure when being burned, so that their softening point dropped. Thus, the glass softened quickly in high temperatures and inhibited the decomposition of cellulose.

[0077] Further, among the fibers b to u (Examples), the fibers e tended to have somewhat low flame retardance because the component having a buffer action of the aqueous solution used in the aftertreatment was in small amount. Further, the fibers f tended to have somewhat low flame retardance because the concentration of sodium ions in the aqueous solution used in the aftertreatment was small. The fibers i, j and k tended to have somewhat low flame retardance because the aqueous solutions used in the aftertreatment all had a low pH. On the other hand, the fiber v (Comparative Example) did not have flame retardance because the aqueous solution used in the aftertreatment contained sodium but did not contain an agent having a buffer action, in other words, the aqueous solution was not a buffer solution containing sodium. It is considered that this was due to the absence of sodium in the fibers.

[0078] For the fibers b to h (Examples) and the fibers a and v (Comparative Examples), their flame retardance after water-washing was measured. For the fibers b to h, there was substantially no decrease in the ash content after water-washing and substantially no change in the flame retardance. Thus, it is considered that sodium was present in the flameproof rayon fibers of Examples.

[0079] Further, for the fibers b to o (Examples) and the fibers a and HELON (Comparative Examples), the whiteness of their samples after heat treatment at 190°C was measured. The fibers i, j and k had high whiteness, in other words, they maintained whiteness but tended to have somewhat low flame retardance. The fibers 1 had low whiteness, in other words, they were tinted but had high flame retardance. The fibers 1 were at a level that might result in reduction in product value depending on the applications. The remaining fibers of Examples all maintained high flame retardance and product value.

(Component Analysis)

[0080] The components of the fibers a and HELON (Comparative Example) and the fibers b, f and g and the water-washed fibers g were determined by an X-ray fluorescence analysis as follows. Table 3 provides the results.

[X-ray Fluorescence Analysis]

[0081] The X-ray fluorescence analysis was performed using an X-ray fluorescence spectrometer "LAB CENTER XRF-1700," manufactured by Shimadzu Corporation, by a theoretical calculation by an FP method. The outline of this measurement device and the measurement conditions are as follows.

(i) Outline of the measurement device

[0082]

Range of elements to be measured: $_4$Be to $_{92}$U
X-ray tube: 4 kw thin window, Rh target
Spectral element: LiF, PET, Ge, TAP, SX
Primary X-ray filter: four-kind automatic exchange (Al, Ti, Ni, Zr)
Field stop: five-kind automatic exchange (diameters of 1, 3, 10, 20, 30 mmϕ)
Detector: scintillation counter (heavy element), proportional counter (light element)

(ii) Measurement conditions

Tube voltage-tube current: 40 kw-95 mA

[0083] For the measurement, cut fibers of the fibers a and HELON (Comparative Examples) and the fibers b, f, g and the water-washed fibers g (Examples) were used. The measurement was made such that the irradiation surface was adjusted to be 10 mmϕ in diameter and several millimeters in thickness and irradiated with light reaching from above and passing therethrough.

[0084]

[TABLE 3]

		Element content (% by mass)				Mass ratio of Si/Na
		O	C	Si	Na	
Blank (theoretical value)		57	43	< 0.1	< 0.1	-
Ex.	fibers b	43	39	17.298	0.25	69.2
	fibers f	43	41	16.085	0.125	128.7
	fibers g	44	40	16.134	0.262	61.6
	fibers (after water-washing	44	40	15.553	0.139	111.9
Comp. Ex.	fibers a	46	37	17	< 0.1	-
	HELON	45	37	17.721	0.197	90

[0085] From the results provided in Table 3, it can be considered that the fibers b, f and g and the water-washed fibers g contained components of silicon and sodium and the components formed sodium silicate. The fibers g were water-washed to check the resistance of the flame retardance to water-washing. As can be seen from the results of the fibers g after water-washing provided in Table 3, the fibers maintained a sodium content of 0.1% by mass or more even after being washed with water. Further, as can be seen from the results of the fibers g after water-washing, at least sodium was partially present in the fibers. Further, in view of the mass ratio of Si/Na of the fibers b, f, g and the fibers g after water-washing (Examples) and HELON (Comparative Example), it is confirmed that particularly favorable flame retardance can be achieved when the mass ratio is less than 90.

(Flameproof Nonwoven Fabric)

[0086] With the use of a sample staple of each of the fibers b, g, 1 and m (Examples) and the fibers a and HELON, flameproof nonwoven fabrics were produced. 30% by mass of low-melting polyester fibers (trade name: "4080", manufactured by Unitika LTD., fineness: 4.4 dtex, fiber length: 51 mm) and 70% by mass of a sample staple were mixed with each other, the mixture was rendered in the form of card webs using a carding machine, and the card webs were placed on top of another such that the total mass per unit area reached 300 g/m^2. Subsequently, the card webs were placed on a punching plate, a nylon mesh was placed on top of the card webs, and a weight was placed on top of the nylon mesh such that a load of 20 g/cm^2 was applied thereto. Then, they were placed in a constant-temperature ventilation drier (trade name: "FC-612", manufactured by Advantec Toyo Kaisha, Ltd.) set at a temperature of 180°C. They were set aside in the drier for 10 minutes to let the low-molten polyester fibers melt to bond the fibers to each other. The nonwoven fabric was taken out from the drier, and thus obtaining the flameproof nonwoven fabric. The flameproof nonwoven fabrics respectively containing the fibers b, g, l and m of Examples exhibited favorable flame retardance.

Industrial Applicability

[0087] As described above, the present invention can provide a flameproof rayon fiber having favorable flameproofness for preventing fire as well as flame retardance (self-extinguishability), and a method for manufacturing the flameproof rayon fiber. Also, the rayon fiber, which is the principal component of the present invention, has biodegradability, while other components mainly are compounds containing silicon and sodium, so that a flameproof rayon fiber with a reduced load to the environment can be provided. In particular, the flameproof rayon fiber according to the present invention can be used as a material replacing glass fibers, asbestos, aramid fibers, etc., which have been used conventionally in flameproof products. The flameproof rayon fiber according to the present invention is processed into woven fabric, knit fabric, nonwoven fabric, etc. and useful for the purposes such as disaster prevention items, kitchen fan filters, sheets, pillow cases, bedding mats, bedding covers, fire protection screens, interior goods (carpets, chair coverings, curtains, wall paper bases, wall materials, etc.), vehicle interior materials (mats, lining fabric, etc.), etc., for example.

Claims

1. A flameproof rayon fiber having flameproofness, comprising:

components of silicon and sodium;
wherein glass remains in the rayon fiber when the rayon fiber is burned at 800°C,
the glass has a property of softening at 800°C, and
when subjected to an X-ray fluorescence analysis, the rayon fiber has a silicon content in a range of 5 to 30% by mass and a sodium content in a range of 0.1 to 3% by mass.

2. The flameproof rayon fiber according to claim 1, wherein the rayon fiber has an LOI value of 31 or more by twisted fiber string measurement (E-1) or an LOI value of 24 or more by nonwoven fabric measurement (E-2) in conformity with JIS L 1091 E (oxygen index test).

3. The flameproof rayon fiber according to claim 1 or 2, wherein the rayon fiber has an ash content in a range of 10 to 50% by mass.

4. The flameproof rayon fiber according to any one of claims 1 to 3, wherein the ratio of the silicon content to the sodium content (mass ratio of silicon/sodium) in the rayon fiber is in a range of 10 or more and less than 90.

5. A method for manufacturing a flameproof rayon fiber, comprising:

preparing an undiluted viscose solution;
adding a solution containing a silicate compound containing an alkali metal to the undiluted viscose solution so as to form an alkali metal-containing silicate compound-added viscose solution;
performing spinning by extruding the silicate compound-added viscose solution through a spinneret into a spinbath containing a sulfuric acid, thus producing a fiber to be treated containing the silicate compound; and
treating, in a scouring or aftertreatment process, the fiber to be treated with a solution having a pH in a range of 4 to 11 and a buffer action and containing sodium.

6. A flameproof fiber structure comprising at least 30% by mass of the flameproof rayon fiber according to any one of claims 1 to 4.

FIG. 1

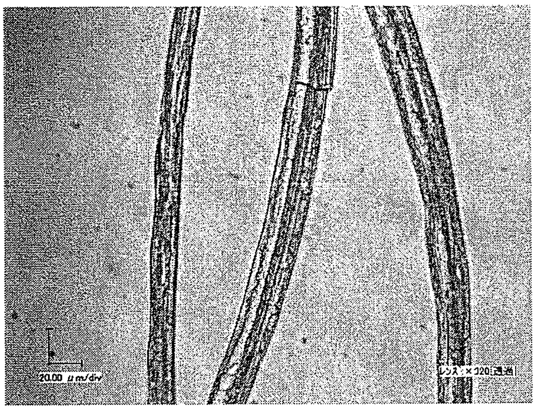


FIG. 2

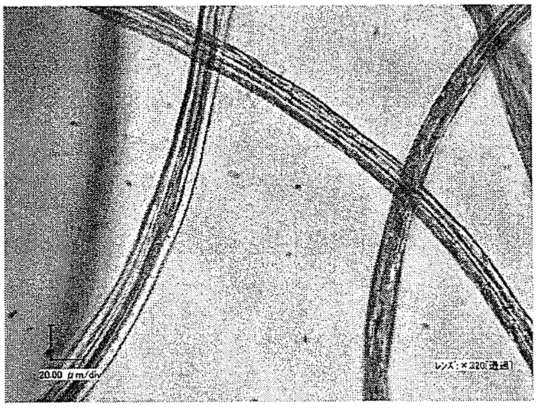


FIG. 3

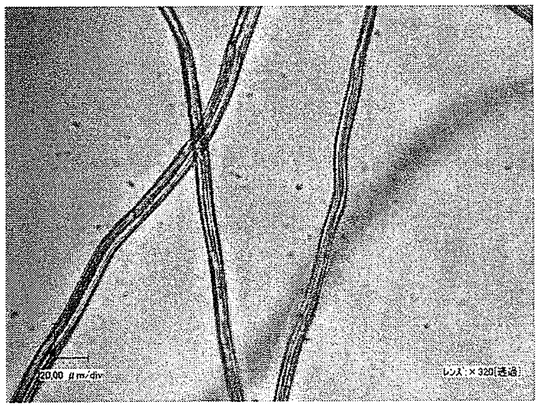
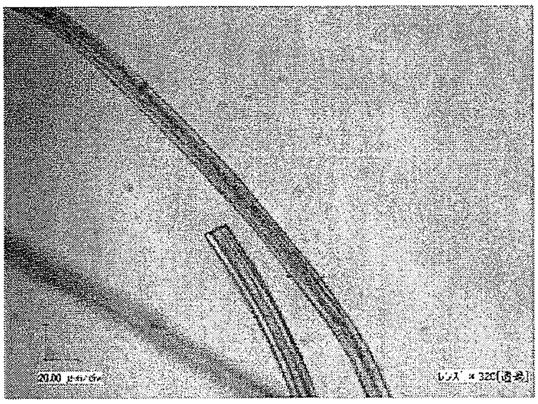


FIG. 4



INTERNATIONAL SEARCH REPORT

International application No.
PCT/JP2009/071771

A. CLASSIFICATION OF SUBJECT MATTER
D01F2/10(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)
D01F2/00-2/30, D01F11/00-11/16, D06M10/00-11/84, D06M16/00, D06M19/00-23/18

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	1996-2010
Kokai Jitsuyo Shinan Koho	1971-2010	Toroku Jitsuyo Shinan Koho	1994-2010

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

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	JP 4094052 B2 (Daiwabo Co., Ltd.), 04 June 2008 (04.06.2008), claims & EP 1918431 A1 claims & US 2009/0030131 A1 & WO 2007/023777 A1 & CN 101248225 A	1-6

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

*	Special categories of cited documents:	"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
"A"	document defining the general state of the art which is not considered to be of particular relevance	"X"	document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
"E"	earlier application or patent but published on or after the international filing date	"Y"	document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art
"L"	document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)	"&"	document member of the same patent family
"O"	document referring to an oral disclosure, use, exhibition or other means		
"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 15 February, 2010 (15.02.10)	Date of mailing of the international search report 23 February, 2010 (23.02.10)
Name and mailing address of the ISA/ Japanese Patent Office Facsimile No.	Authorized officer Telephone No.

Form PCT/ISA/210 (second sheet) (April 2007)

INTERNATIONAL SEARCH REPORT

International application No.
PCT/JP2009/071771

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 2008-501867 A (Sateri International Co., Ltd.), 24 January 2008 (24.01.2008), claims & WO 2005/118922 A1 claims & US 2008/0093767 A1 & EP 1753900 A1 & DE 602005011000 D & FI 20045204 A & CN 1965113 A & AT 414190 T & ES 2317236 T	1-6
A	JP 3179104 B2 (Kemira Fibres Oy), 25 June 2001 (25.06.2001), claims & WO 1993/013249 A1 claims & JP 7-506629 A & US 5417752 A & EP 619848 A1 & DE 69228197 C & DE 69228197 T & FI 916187 A & AT 175732 E & ES 2128412 T & AT 175732 T & AU 3161293 A	1-6

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- JP 3179104 B **[0004]**
- JP 4094052 B **[0004]**