



US 20060278534A1

(19) **United States**(12) **Patent Application Publication**
Hwang(10) **Pub. No.: US 2006/0278534 A1**(43) **Pub. Date: Dec. 14, 2006**(54) **METHOD FOR PRODUCING NANOSILVER
ON A LARGE SCALE, METHOD FOR
MANUFACTURING
NANOSILVER-ADSORBED FIBER, AND
ANTIBACTERIAL FIBER THEREBY****Publication Classification**(51) **Int. Cl.**
C25B 1/00 (2006.01)
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Cleveland, OH 44114 (US)(21) Appl. No.: **11/348,213**(22) Filed: **Feb. 6, 2006**(30) **Foreign Application Priority Data**Jun. 13, 2005 (KR) 2005-50227
Aug. 12, 2005 (KR) 2005-74016(57) **ABSTRACT**

A method for producing nanosilver on a large scale, a method of manufacturing nanosilver-adsorbed fiber, and an antibacterial fiber manufactured thereby. The nanosilver having a size of 5 nm or less can be generated on a large scale by controlling a minutely electronic current between the two Ag electrodes in a water electrolysis system, while a voltage of 10,000~300,000 is applied to two Ag electrodes. The nanosilver-adsorbed fiber is manufactured by applying the aqueous solution containing nanosilver to the surface of the synthetic fibers; adsorbing the nanosilver onto the synthetic fibers using a process selected from the group consisting of thermal fixation, high frequency radiation, bubbling, and combinations thereof; and conducting post-finishing at 160 to 200° C.

Fig. 1

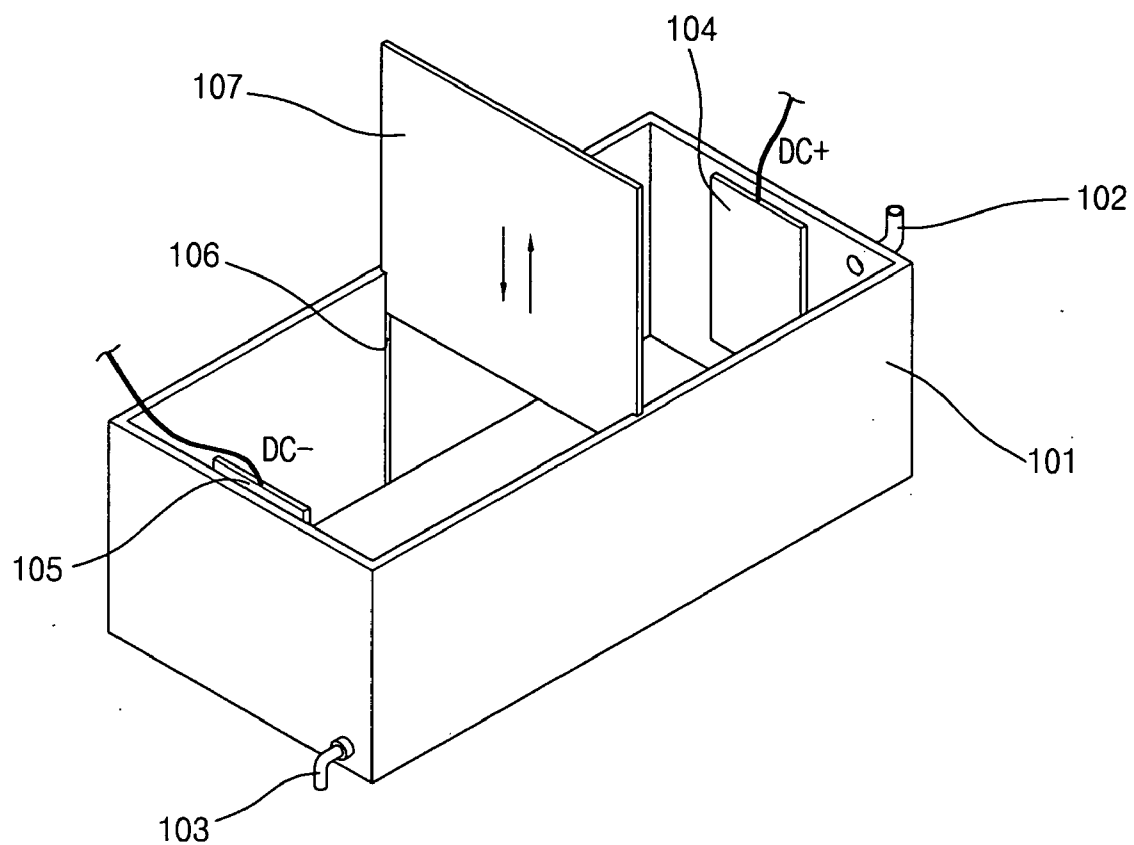


Fig. 2

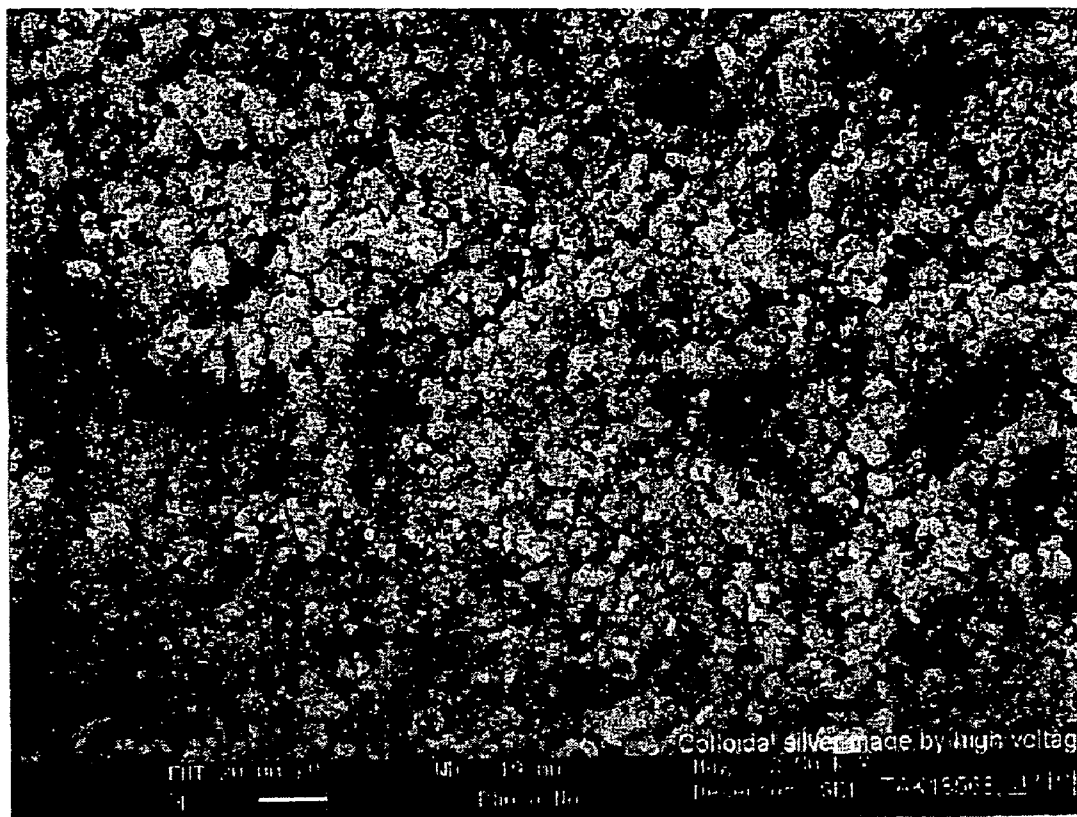
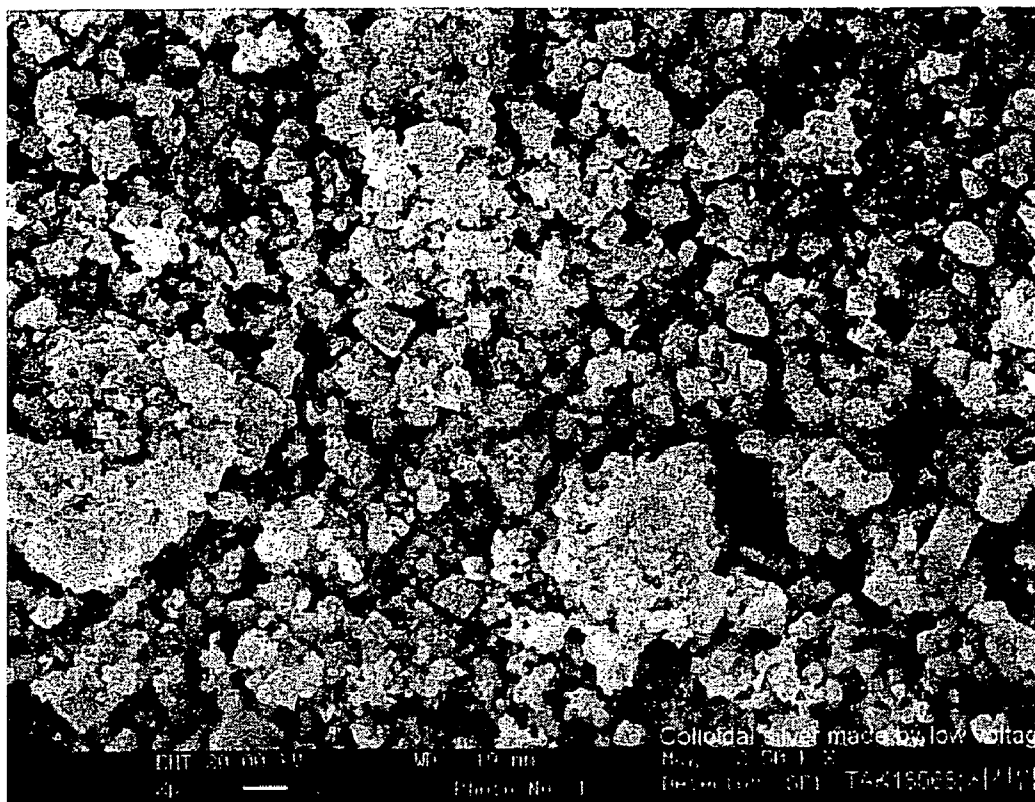


Fig. 3



**METHOD FOR PRODUCING NANOSILVER ON A
LARGE SCALE, METHOD FOR
MANUFACTURING NANOSILVER-ADSORBED
FIBER, AND ANTIBACTERIAL FIBER THEREBY**

BACKGROUND OF THE INVENTION

[0001] 1. Technical Field of the Invention

[0002] The present invention relates to a method for producing nanosilver on a large scale, a method of manufacturing nanosilver-adsorbed fiber, and antibacterial fiber manufactured thereby. More particularly, the present invention relates to the method for producing nanosilver on a large scale having a size of 5 nm or lower by allowing only a minute electric current to flow between two opposite silver electrodes in the presence of a high voltage in a water electrolysis system, a method of manufacturing nanosilver-adsorbed fiber by taking advantage of the better applicability of smaller silver particles, and an antibacterial fiber manufactured thereby.

[0003] 2. Description of the Prior Art

[0004] A variety of microbes are found in large quantities in daily living environments. Particularly, they grow and proliferate on clothes and form flora even on the skin. The microbes degrade fibers or digest nutrients in sweat or contaminants, producing bad odors or causing great damage to the health of humans.

[0005] WHO reports that microbial contamination corresponds to about 30% of the mortality in the world. Unfortunately, current scientific technologies fall short of sufficiently controlling harmful microbes.

[0006] Therefore, it is intensively researched to develop antimicrobial/germicidal agents or products that are satisfied with harmless to human bodies and have improved functionality.

[0007] As representative example of the antimicrobial agents, it is known that silver can absolutely suppress almost all single cell pathogens. Because of such antimicrobial activity, silver has been used long and widely in antimicrobial fields, for example, tableware, such as bowls, spoons, chopsticks, etc., and herbal medicines such as silver-coated pills.

[0008] As for the antibacterial function of silver, it is reportedly based on the activity of suppressing certain enzymatic reactions essential for the metabolism of pathogens, thus killing them.

[0009] Particularly in association with nano technology, silver in a nano state exhibits more powerful antibacterial and germicidal activity. Many research results report that silver in a nano state can kill as many as 650 kinds of bacteria and other microbes and show powerful suppression against fungi.

[0010] In addition, the smaller silver particles are, the more powerful antibacterial/germicidal activity, because silver becomes to the increase in surface area.

[0011] According to experimental data, silver powders show 99.9% antibacterial and germicidal efficiency over a variety of bacteria, including enterobacteria, *Staphylococcus aureus*, Salmonella, Vibrio, shigella, Pneumococcus, typhoid, and even MRSA (methicillin resistant *Staphylococcus*

aureus). That means that almost no bacteria can survive 5 minutes or longer in contact with nanosilver.

[0012] While having tenfold more potent suppression against bacteria than the conventional chloride-based agent, a nanosilver does not damage human bodies at all. Therefore it is expected to be a useful therapeutic agent against various inflammations. In addition, taking advantage of nanosilver, various functional products having antibacterial and deodorizing activities are on the market.

[0013] In the fiber and textile industries, accordingly, it is key point to produce nanosilver having such excellent effects on a large scale and to effectively intercalate the nanosilver into fibers.

[0014] In past three decades, synthetic fibers have been used in a wide range of fields of human life as complements to or substitutes for natural fibers and even as materials that are functionally superior to natural fibers. During this period, synthetic fibers for clothes have been developed towards practicality, comfort, and other functionalities. Furthermore, it has been actively researched to environment- and body-friendly synthetic fibers in recent. In a persistent effort to develop synthetic fibers that satisfy the above request, the antibacterial activity of nanosilver is applied to fibers.

[0015] Conventionally, nanosilver has been extracted using a physical method, such as liquid phase reduction, grinding, etc., or an electrolytic method. The electrolytic method is conducted under low temperature and voltage that silver having 99.9% of contents is added to distilled water, and then the silver-containing compounds are electrolyzed and carried to electrophoresis through the (+) and (-) of each molecule, results in collecting nanosilver.

[0016] On the other hand, as a representative method to intercalate nanosilver into fibers, synthetic fibers are manufactured by mixing nanosilver with raw synthetic fiber materials before the synthetic fibers are spun. However, the fibers, which are synthesized in the above mentioned method, is poor in antibacterial or germicidal activity because most silver is deeply intercalated into synthetic fibers while only a small amount of silver is exposed on the surfaces of synthetic fibers.

[0017] Alternatively, antibacterial agents, such as silver, silver oxide, nanosilver etc., are coated onto synthetic fibers. However, the antibacterial agents have poor adhesive strength with synthetic fibers; therefore the synthetic fibers are inferior to washing durability.

[0018] Leading to the present invention, intensive and thorough research, conducted by the present inventors, on antibacterial fibers and cloth resulted in the finding that nanosilver must be exposed in a larger amount on the surface of synthetic fibers, rather than be embedded within them, in order to maximize the germicidal or antibacterial effect of silver. The smaller the synthetic fibers are, the more powerful antibacterial/germicidal activities are. Also, In order to obtain the smaller nanosilver, it is designed that the mass production of nanosilver is generated by keeping up with minute electronic current between the two Ag electrodes in water electrolysis system to high voltage.

SUMMARY OF THE INVENTION

[0019] It is an aspect of the present invention to provide a method for producing nanosilver on a large scale.

[0020] It is another aspect of the present invention to provide a method for manufacturing nanosilver-adsorbed fiber that nanosilver is intensively adsorbed on surface of synthetic fibers.

[0021] It is another aspect of the present invention to provide an antibacterial fiber manufactured thereby.

[0022] In an exemplary embodiment of the present invention, a method for producing nanosilver on a large scale is provided. More practically, the nanosilver on a large scale is generated by controlling a minutely electronic current between the two Ag electrodes in water electrolysis system, while a voltage of 10,000~300,000 is applied to two Ag electrodes.

[0023] An object of the invention is to provide a nanosilver on a large scale, preferably comprising; loading water so as to immerse the two Ag electrode plates in the water electrolysis system, applying voltage of 10,000~300,000 to two Ag electrodes, moving the circuit breaker upwards or downwards relative to the voltage, and thus controlling minutely electric current between the two Ag electrodes. The water electrolysis system preferably comprises: a water reservoir provided with a water inlet valve for introducing water thereinto and a water outlet valve for draining the water therefrom; the two Ag electrode plates connected to a DC+ electric power source and a DC- electric power source respectively, the two Ag electrodes being provided on respective opposite sides of the water reservoir; a circuit breaker for dividing the water reservoir into two sections, and being provided in a middle of the water reservoir; and a groove for the circuit breaker, being formed in a middle portion of the water reservoir.

[0024] The particle size of the nanosilver is preferably from 1 to 5 nm.

[0025] Another object of the invention is to provide a method for manufacturing nanosilver-adsorbed fiber where nanosilver is intensively adsorbed on the surface of synthetic fibers. More practically, the preferred method comprises; preparing aqueous solution containing the nanosilver on a large scale; scouring and washing synthetic fibers; applying the aqueous solution containing the nanosilver to the surface of the synthetic fibers; adsorbing the nanosilver onto the synthetic fibers using a process selected from the group consisting of thermal fixation, high frequency radiation, bubbling, and combinations thereof; and conducting post-finishing at 160 to 200° C.

[0026] The latter preferred method may further comprise a dyeing step before the post-finishing.

[0027] Preferably, the thermal fixation is carried out at a temperature from 150 to 230° C.

[0028] The aqueous solution containing the nanosilver is preferably in an amount of 10 to 100 ppm of the nanosilver.

[0029] The step of applying the aqueous solution containing the nanosilver to the surface of the synthetic fibers is preferable to conducting a process selected from the group consisting of spraying, coating, and dipping.

[0030] In accordance with a further aspect of a preferred form of the present invention, an antibacterial fiber manufactured thereby, in which antibacterial fiber has the nanosilver has adsorbed thereon in an amount of 0.01 to 0.1 g per 100 g of synthetic fibers

BRIEF DESCRIPTION OF THE DRAWINGS

[0031] FIG. 1 shows a device for preparing the aqueous solution containing the nanosilver in accordance with the present invention,

[0032] FIG. 2 shows the particle distribution prepared in the presence of a high voltage in the aqueous solution containing the nanosilver according to the present invention, and

[0033] FIG. 3 shows the particle distribution prepared in the presence of a low voltage in the aqueous solution containing the nanosilver according to conventional method.

DETAILED DESCRIPTION OF THE INVENTION

[0034] Hereinafter, the present invention is described in detail.

[0035] First, a method for producing nanosilver on a large scale is described according to an exemplary embodiment of the present invention. The method for producing nanosilver on a large scale is provided, based on the electrolysis of water in which, while high voltage, namely 10,000~300,000 V, is applied to two Ag electrodes (104, 105), the nanosilver on a large scale are generated by keeping up with minute electric current between the two electrodes by controlling the height of the circuit breaker.

[0036] FIG. 1 shows a device for preparing the aqueous solution containing the nanosilver in accordance with the present invention.

[0037] More practically, the method for producing nanosilver on a large scale by using a water electrolysis system comprises:

[0038] loading water so as to immerse the two Ag electrodes;

[0039] applying voltage of 10,000~300,000 to two Ag electrodes;

[0040] moving the circuit breaker upwards or downwards with the voltage, and thus controlling minutely electric current between the two Ag electrodes.

[0041] The water electrolysis system comprises,

[0042] a water reservoir (101) provided with a water inlet valve (102) for introducing water thereinto and a water outlet (103) valve for draining the water therefrom;

[0043] the two Ag electrode plates (104, 105) connected to a DC+ electric power source and a DC- electric power source respectively, the two Ag electrode plates being provided on respective opposite sides of the water reservoir (101);

[0044] a circuit breaker (107) for dividing the water reservoir (101) into two sections, being located of a middle of the water reservoir (101); and

[0045] a groove (106) formed by the circuit breaker(107).

[0046] It will be readily understood by those skilled in the art that the water reservoir, the water inlet valve, the water outlet valve, and the circuit breaker are all electrically insulated.

[0047] Generally, it was widely acknowledged that an electric current rises in proportion to voltage, in accordance with the following Formula 1. When low voltage is applied, electric current can be controlled using circuits, diodes and the like. However when high voltage is applied, current control is generally impossible.

$$\text{Voltage(V)} = \text{Current(I)} \times \text{Resistance(R)} \quad [\text{Formula 1}]$$

[0048] Therefore, the present invention is characterized that while a high voltage, 10,000~300,000 V, is applied to electrolysis system, the electric current control is possible in high voltage. When the high voltage is applied, it is accomplished that the circuit breaker (107) installed at a middle portion of the water reservoir (101) is moved upwards or downwards to allow only minute electric current between the two electrodes. So, the nanosilver on a large scale is generated.

[0049] In more detail, when the circuit breaker (107) is absent, electric current flowing through the water reservoir with a certain voltage follows Formula 1. The other side, the circuit breaker (107) is controlled by $\frac{1}{2}$ the height of the water reservoir (101), the electric current flows on the decrease until $\frac{1}{2}$.

[0050] Thus, while high voltage, 10,000~300,000 V is applied to two Ag electrodes, the nanosilver on a large scale is generated by keeping up with minute electric current between the two electrodes by controlling the height of the circuit breaker.

[0051] The electric current control is conducted until the nanosilver size becomes 5 nm or less and preferably 1 to 5 nm. When exceeding 5 nm in size, nanosilver particles lose the property of being easily adsorbed, characteristic of nanosilver, which is generated, because their surface area is decreased. In addition, if the electric current amount is not controlled to the high voltage, silver ions are not isolated to produce nano particles, but silver plating occurs.

[0052] FIG. 2 shows the particle distribution prepared in the presence of a high voltage in the aqueous solution containing the nanosilver according to the present invention. The particle distribution is observed using scanning electron microscopy. FIG. 2 shows that the nanosilver having a size of 5 nm or less is uniformly distributed. However, FIG. 3 shows the particle distribution prepared in the presence of a low voltage in the aqueous solution containing the nanosilver according to conventional method. FIG. 3 shows that the particles are non-uniformly distributed, with particle aggregations found therein.

[0053] In addition, the nanosilver prepared according to the exemplary embodiment of the present invention is in a nanosilver solution state so that the nanosilver particles are uniformly distributed, and the nanosilver can be uniformly and readily coated or adsorbed on synthetic fibers.

[0054] Another exemplary embodiment of the present invention is related to a method manufacturing nanosilver-adsorbed fiber. The method is comprised of; preparing aqueous solution containing the nanosilver synthesized on a large scale; scouring and washing synthetic fibers; applying the aqueous solution containing the nanosilver to the surface of the synthetic fibers; adsorbing the nanosilver onto the synthetic fibers using a process selected from the group consisting of thermal fixation, high frequency radiation, bubbling, and combinations thereof; and conducting post-finishing at 160 to 200° C.

[0055] The method may further comprise a dyeing step before the post-finishing.

[0056] The nanosilver-adsorbed fiber may be carried out on general cloth types, for example, leather, natural fibers, and synthetic fibers, and preferably with synthetic fibers.

[0057] The term "synthetic fiber" as used herein means generic fiber made from raw chemical materials, such as polyester, nylon, acryl, etc. Preferably, synthetic fiber has smooth surface such that the nanosilver can be easily adsorbed thereon, in contrast with natural fibers consisting of warp and weft. In the case of natural fibers, nanosilver is deeply intercalated into natural fibers, thus antimicrobial activity is poor. The application of the aqueous solution containing the nanosilver to the surface of synthetic fibers may be carried out using a spraying method, a coating method, or a dipping method followed by coating using a knife or a roll knife.

[0058] The antibacterial fiber is preferably adsorbed with nanosilver in an amount of 0.01 to 0.1 g per 100 g of synthetic fibers. According to the exemplary embodiment of the present invention, large amounts of nanosilver can be adsorbed on the synthetic fibers in comparison to conventional methods. When the amount of the nanosilver adsorbed on the synthetic fiber is less than 0.01 g, the synthetic fibers have insufficient antibacterial activity. On the other hand, if the amount of the nanosilver adsorbed on the synthetic fibers is more than 0.1 g, the cost for excessively increases relative to the improvement of antibacterial effects.

[0059] The adsorption of the nanosilver onto the surface of synthetic fibers may be achieved using various processes. An example among preferred processes is a thermal fixation process at 150 to 230° C. The thermal fixation process makes the cloth flexible. Here, when the temperature during the process is below 150° C., the surface of raw fiber becomes too flexible. On the other hand, when the temperature during the process is higher than 230° C., the surface of raw fiber becomes too stiff. Thermal fixation process is needed to be carried out under about 2 atm.

[0060] Another process for the adsorption of the nanosilver onto the surface of the synthetic fibers uses high-frequency radiation. The high-frequency radiation process uses an ultrasonic wave frequency that exceeds the upper limit of the range of audio frequencies (16 to 16000 Hz). Generally, ultrasonic waves may be generated by applying an ultrasonic signal produced in an electric circuit to an ultrasonic oscillator. The irradiation of ultrasonic waves onto the nanosilver solution produces innumerable fine voids. The innumerable fine voids are helpful in adsorbing the nanosilver onto the surface of synthetic fibers.

[0061] Another process for the adsorption of the nanosilver onto the surface of the synthetic fibers may be accomplished through bubbling. In this process, nanosilver particles, ionized by electrolysis, are oscillated leftwards and rightwards, upwards and downwards, or backwards and forwards by the bubbling. The oscillation of the nanosilver particles activates mobility, which is accelerated in the presence of a voltage, so that the nanosilver particles are uniformly distributed over the synthetic fibers. To carry out the above process using the bubbling, the target synthetic fibers are immersed in a separate inner vessel placed inside the water reservoir which has a plurality of openings through which bubbles are generated at a lower portion of the water reservoir.

[0062] Next, post-finishing is conducted, in which the synthetic fibers having nanosilver adsorbed thereon are ironed at 160 to 200° C.

[0063] In addition, a dyeing process may be further conducted before the post-finishing. In the dyeing process, the nanosilver-adsorbed fiber may be dyed at about 130° C. for 3 to 5 hours with a mixture of a dye and a dispersant.

[0064] An antibacterial fiber prepared according to the exemplary embodiment of the present invention includes nanosilver adsorbed thereon in an amount from 0.01 to 0.1 g per 100 g of the synthetic fibers. The synthetic fibers have excellent antibacterial activity because the nanosilver particles are intensively adsorbed on the surface thereon.

[0065] The antibacterial fiber is semi-permanently maintained washing durability, since the antibacterial fiber was manufactured by easily adsorptive properties of the nanosilver itself. Test results for washing durability of the antibacterial fiber made of nanosilver-adsorbed fiber reveals that the nanosilver remained thereon even after 50 washes.

[0066] In addition, the nanosilver is intensively adsorbed onto the surface of the synthetic fibers. Therefore, the antibacterial fiber according to the exemplary embodiment of the present invention is excellent to antibacterial activity, and simultaneously can prevent poor perspiration functionality and the generation of static electricity.

[0067] A better understanding of the present invention may be obtained in light of the following examples, which are set forth to illustrate, but are not to be construed to limit the present invention.

EXAMPLE 1

[0068] Step 1: Preparation of Aqueous Solution Containing the Nanosilver

[0069] As shown in FIG. 1, the device for preparing aqueous solution containing the nanosilver comprises a water reservoir (101), provided with a water inlet valve (102) for introducing water thereinto and a water outlet (103) valve for draining the water therefrom; the two Ag electrodes (104, 105) connected to a DC+ electric power source and a DC- electric power source respectively, the two Ag electrodes being provided on respective opposite sides of the water reservoir (101); a circuit breaker (107) for dividing the water reservoir (101) into two sections, being located at a middle of the water reservoir (101); and a groove (106) being formed owing to the circuit breaker (107).

[0070] In this device, water was loaded on the water reservoir (101) with the level of immersing the two Ag electrodes (104, 105). Next, 30,000 V was applied across the two Ag electrodes in the water electrolysis system. The mass production of nanosilver was generated by keeping up with minute electric current between the two electrodes by controlling the height of the circuit breaker (107) to the water reservoir (101).

[0071] The nanosilver thus prepared was proven to have a size of 5 nm or less, with uniform particle distribution as measured using a scanning electron microscope (Model LEICA-STEROSCAN440) in FITI Testing & Research Institute of Korea (FIG. 2).

[0072] Step 2: Preparation of Antibacterial Fiber

[0073] Synthetic fibers were washed with water and scoured at a maximum temperature of 125° C. so that the synthetic fibers were made clean and neat.

[0074] Thereafter, the synthetic fibers were immersed in aqueous solution containing the nanosilver. Here, the temperature of the aqueous solution containing the nanosilver for the adsorption process was maintained at 230° C. under a pressure of about 2 atm, so that the nanosilver was thermally fixed on the surface of the synthetic fibers. In order to ensure the thermal fixation of the nanosilver onto the synthetic fibers, ultrasonication was conducted and bubbles were generated to accelerate the mobility of the nanosilver particles.

[0075] Afterwards, a dye and a dispersant were mixed in acetic acid and the synthetic fibers were dyed at 130° C. for 3 to 5 hours, followed by post-finishing in which the cloth was pressed at 200° C.

EXAMPLE 2

[0076] An antibacterial fiber was manufactured in a same manner to that of Example 1, with the exception that the circuit breaker (107) was controlled to cause the size of nanosilver to be 5 nm or less in the presence of 300,000 volts (DC+, DC-) in Step 1 of Example 1.

COMPARATIVE EXAMPLE 1

[0077] The same procedure as in Example 1 was performed, with the exception that 220 volts (DC+, DC-) was applied in Step 1.

[0078] As seen in FIG. 3, the nanosilver prepared in Comparative Example 1 was observed to aggregate together, with a non-uniform particle distribution under a SEM (model: LEICA-STEROSCAN440) in FITI Testing & Research Institute of Korea.

[0079] While this invention has been described in connection with certain exemplary embodiments and examples, it is to be understood that the present invention is not limited to the disclosed embodiments and examples, but, on the contrary, is intended to cover various modifications included within the spirit and scope of the appended claims and equivalents thereof.

[0080] As described hereinbefore, the present invention provides a method for producing nanosilver on a large scale by applying a high voltage in water electrolysis, and an antibacterial fiber having intensively nanosilver adsorbed thereon. Furthermore, the nanosilver adsorbed cloth is free of the problems possessed by general synthetic fibers, that is, poor perspiration functionality and high static electricity generation, and as well, shows potent suppression against a broad range of bacteria and microbes.

[0081] Although the preferred embodiments of the present invention have been disclosed for illustrative purposes, those skilled in the art will appreciate that various modifications, additions and substitutions are possible, without departing from the scope and spirit of the invention as disclosed in the accompanying claims.

What is claimed is:

1. A method for producing nanosilver on a large scale by using a water electrolysis system, comprising:

providing two Ag electrode plates;

applying a voltage of 10,000~300,000 to said two Ag electrode plates to produce a minutely electronic current between said two Ag electrode plates, said electrode plates being equipped in the water electrolysis system; and

controlling said minutely electronic current between the two Ag electrodes in the water electrolysis system.

2. The method according to claim 1, wherein the method for producing nanosilver on a large scale by using a water electrolysis system comprising a water reservoir having two opposite sides and a middle and provided with a water inlet valve for introducing water into said water reservoir and a water outlet valve for draining the water from said water reservoir; a DC+ electric power source and a DC- electric power source, said two Ag electrode plates being connected to said DC+ electric power source and said DC- electric source respectively, the two Ag electrode plates being provided on respective opposite sides of the water reservoir; a circuit breaker for dividing the water reservoir into two sections, being provided in the middle of the water reservoir;

and a groove formed by said circuit breaker, wherein said method comprises:

loading water to immerse said two Ag electrode plates in the water electrolysis system;

applying a voltage of 10,000~300,000 to said two Ag electrode plates; and

moving said circuit breaker upwards or downwards with the voltage to control the minutely electric current between said two Ag electrode plates.

3. The method according to claim 1, wherein the particle size of the nanosilver is 1 to 5 nm.

4. A method of manufacturing nanosilver-adsorbed fiber comprising:

preparing an aqueous solution containing the nanosilver produced according to the method of claim 1;

scouring and washing synthetic fibers, said synthetic fibers having a surface;

applying the aqueous solution containing the nanosilver to the surface of said synthetic fibers;

adsorbing the nanosilver onto said synthetic fibers using a process selected from the group consisting of thermal fixation, high frequency radiation, bubbling, and combinations thereof; and

conducting post-finishing at 160 to 200° C.

5. The method according to claim 4, further comprising a dyeing process before the step of conducting post-finishing at 160 to 200° C.

6. The method according to claim 4, wherein the thermal fixation process is conducted at a temperature from 150 to 230° C.

7. The method according to claim 4, wherein the aqueous solution containing the nanosilver is in an amount of 10 to 100 ppm of the nanosilver.

8. The method according to claim 4, wherein the step of applying the aqueous solution containing the nanosilver to the surface of said synthetic fibers is carried out by a process selected from the group consisting of spraying, coating and dipping.

9. An antibacterial fiber having nanosilver adsorbed thereon in an amount of 0.01 to 0.1 grams per 100 grams of synthetic fibers, and being produced according to the method of claim 4.

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