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(54) **DYED FABRIC HAVING ULTRAVIOLET  
RADIATION PROTECTION AND  
ANTIMICROBIAL PROTECTION**

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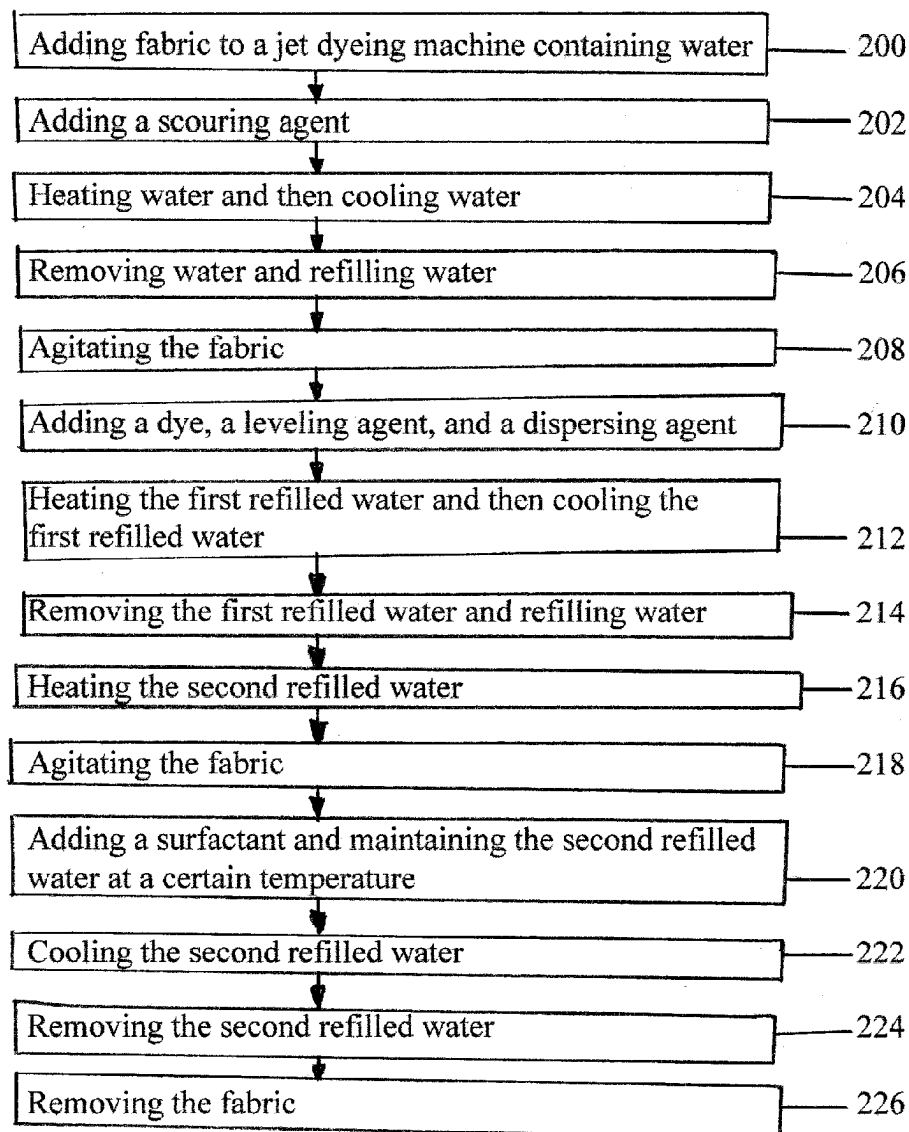
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(57) **ABSTRACT**

A fabric having ultraviolet radiation protection and antimicrobial protection has a quantity of ZnO incorporated into the fabric and a quantity of a dye incorporated into the fabric. A synthetic fabric having ultraviolet radiation protection and antimicrobial protection has a quantity of ZnO incorporated into the synthetic fabric and a quantity of a dye incorporated into the synthetic fabric.



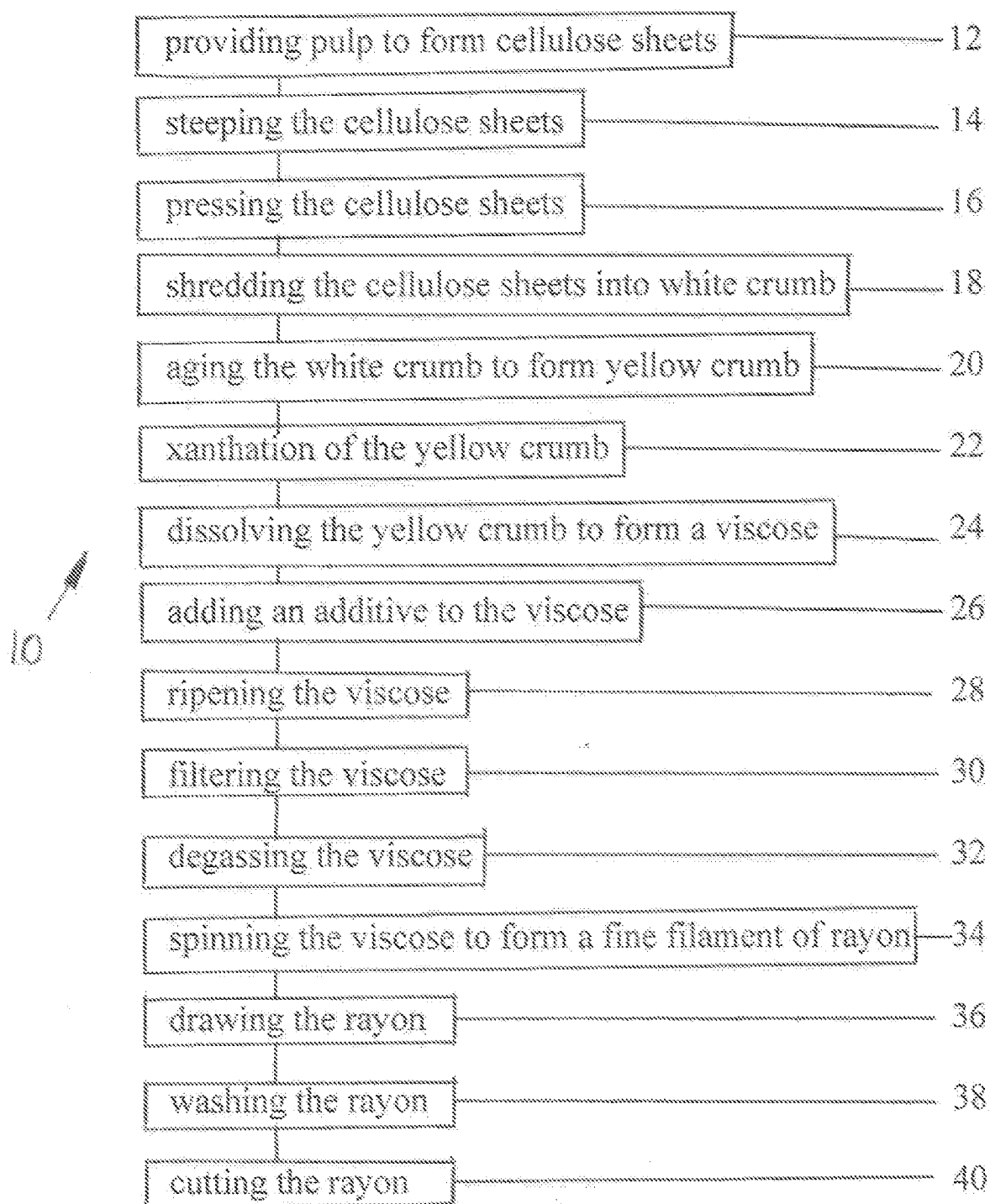


FIG. 1

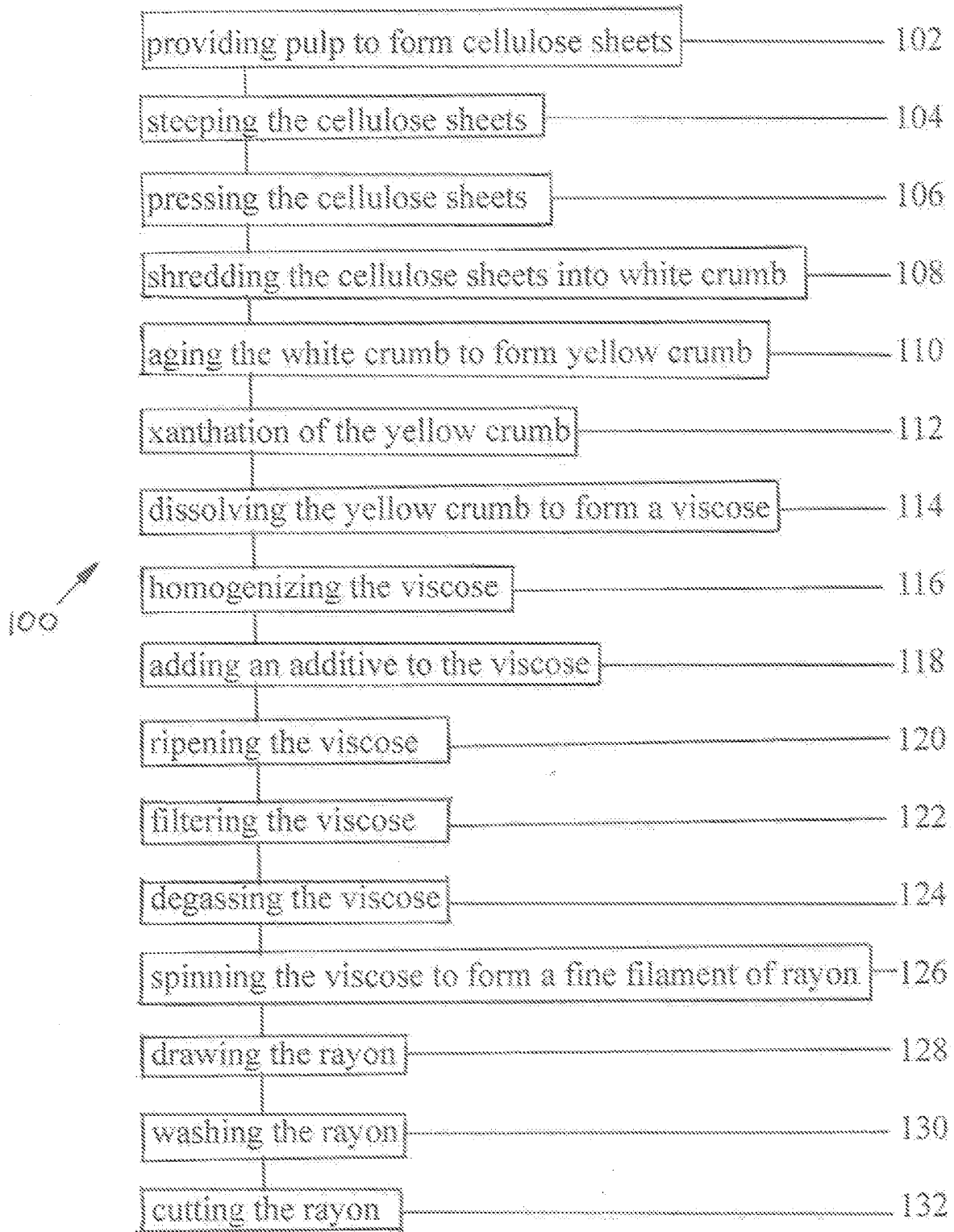


FIG. 2

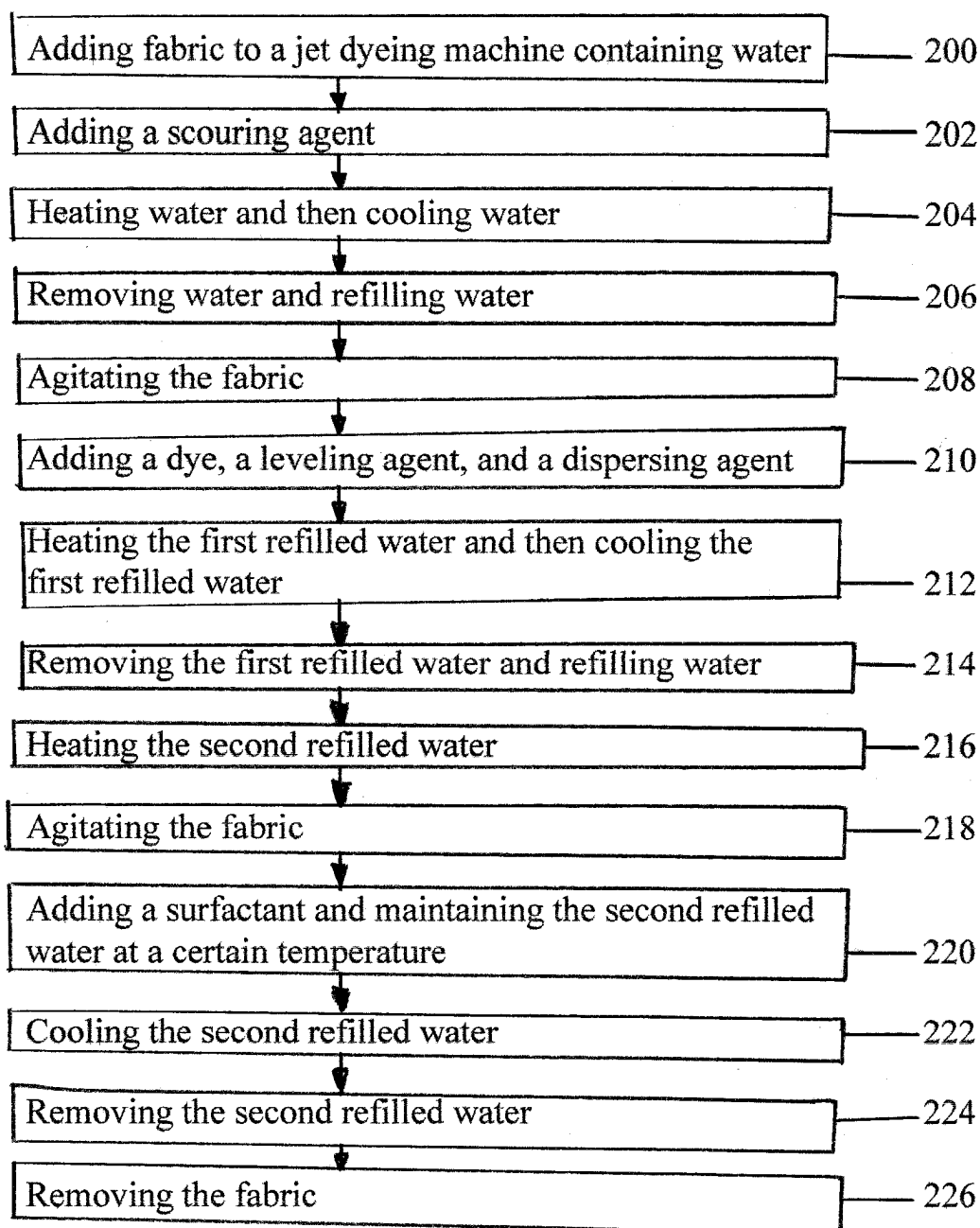


FIG. 3

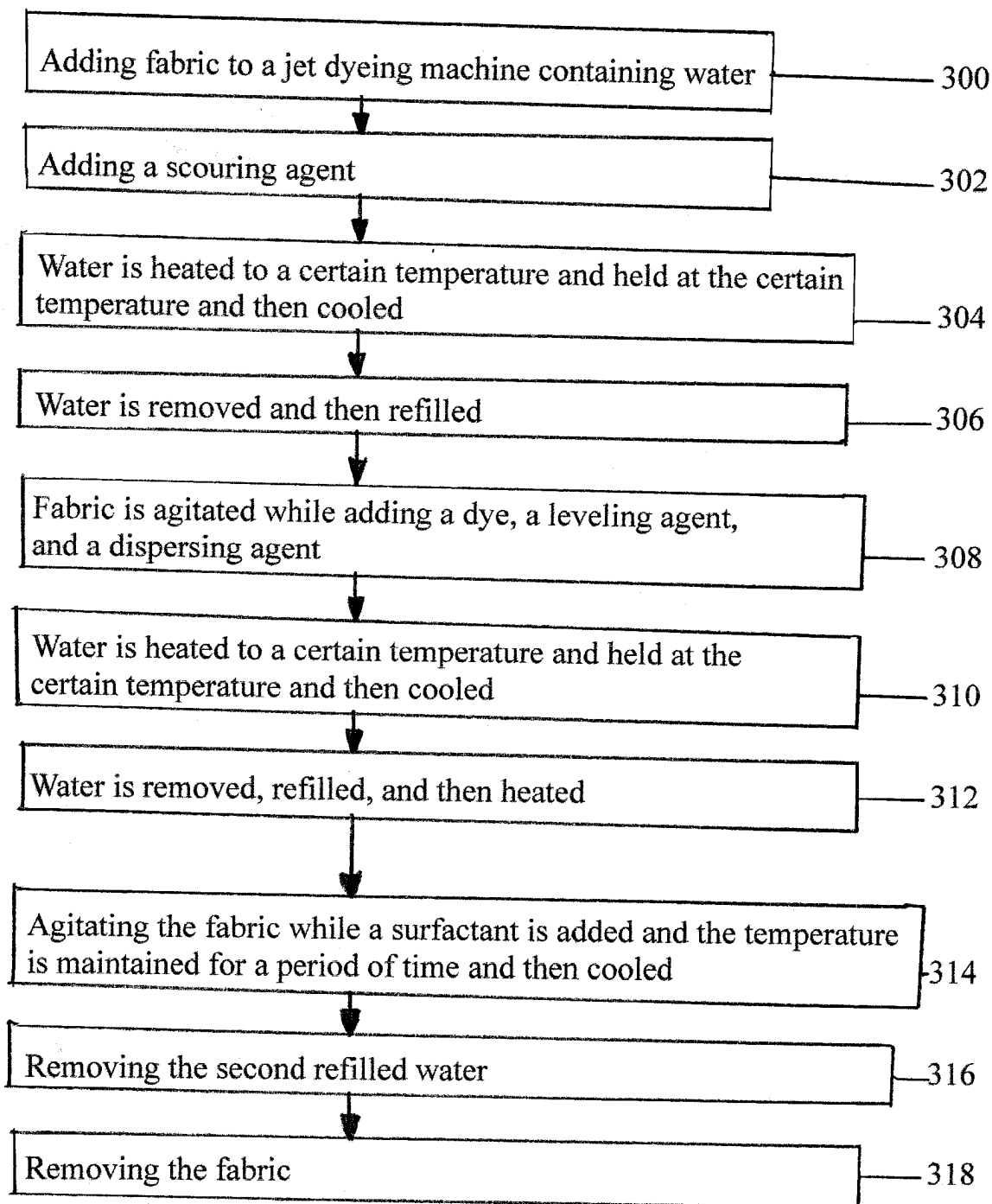


FIG. 4

## DYED FABRIC HAVING ULTRAVIOLET RADIATION PROTECTION AND ANTIMICROBIAL PROTECTION

### BACKGROUND

[0001] This disclosure relates to a dyed fabric having ultraviolet (UV) radiation protection incorporated therein, and more specifically, to a dyed synthetic fabric having incorporated therein an additive for providing UV protection and antimicrobial protection to the fabric and the fabric for use in manufacturing a textile or a garment.

[0002] Ecological friendly fabrics or Eco-friendly fabrics are gaining in popularity and use in clothing. An Eco-friendly fabric may be a natural fiber such as cotton, hemp, or bamboo which has been grown in soil that has not been treated with pesticides for a number of years. Some examples of other Eco-friendly fabrics are organic cotton, sisal, a combination of hemp and recycled rayon, a combination of hemp and cotton, broadcloth, denim, linen, and a combination of bamboo and recycled rayon. Natural fibers, which may be derived from plants or animals, such as wool, angora, silk, alpaca, cashmere, and silk are also examples of Eco-friendly fabrics. Synthetic fabrics, which may be made from synthetic sustainable products, such as nylon, rayon, olefin, spandex, and tencel are also examples of Eco-friendly fabrics.

[0003] To assist an individual in determining whether a garment has protection against ultraviolet radiation, a rating system has been developed. This rating system is known in the industry as the UPF (Ultraviolet Protection Factor) rating system. Clothing having a rating of UPF 50 are able to block out 98% of the sun's ultraviolet radiation. Further, by way of example, a garment having a rating of UPF 15-24 will only block out 93.3% to 95.9% of ultraviolet radiation. Exposure to the sun's harmful ultraviolet radiation (known as UVA/UVB rays) can damage the skin, can cause sunburn, and can lead to skin cancer over prolonged exposure.

[0004] There are a number of factors that affect the level of ultraviolet radiation protection provided by a fabric and the UPF rating. Some factors are the weave of the fabric, the color of the fabric, the weight of the fabric, the fiber composition of the fabric, the stretch of the fabric, moisture content of the fabric. If the fabric has a tight weave or a high thread count then the fabric will have a higher UPF rating. However, even though the fabric has a higher UPF rating, the fabric may be less comfortable because a tighter weave or higher thread count means that the fabric is heavy or uncomfortable to wear. Another factor that affects protection is the addition of chemicals such as UV absorbers or UV diffusers during the manufacturing process. As can be appreciated, some of the features that make a garment comfortable to wear also make the garment less protective. A challenge for a clothing manufacturer is to provide clothing having both protection from the sun and being comfortable to wear.

[0005] Athletic clothing or active wear clothing is typically manufactured from synthetic material such as polyester or nylon. Polyester may be formed into a filament yarn that is used to weave a fabric or garment. To form polyester, dimethyl terephthalate is placed in a container and first reacted with ethylene glycol in the presence of a catalyst at a temperature of 302-410° F. The resulting chemical, a monomer alcohol, is combined with terephthalic acid and raised to a temperature of 472° F. Newly-formed polyester, which is clear and molten, is extruded through a slot

provided in the container to form long ribbons. the long molten ribbons are allowed to cool until they become brittle. The ribbons are cooled and then cut into tiny polymer chips. These tiny polymer chips are then melted at 500-518° F. to form a syrup-like melt or liquid. This melt is put into a metal container called a spinneret and forced through its tiny holes to produce special fibers. The emerging fibers are brought together to form a single strand. This strand is wound on a bobbin for further processing or to be woven into yarn.

[0006] Various methods and additives have been proposed for incorporating UV protection into synthetic materials. However, once the synthetic materials are incorporated with UV protection one problem has been dyeing fabric manufactured from the synthetic materials. In particular, dye may fade from the synthetic fabric during use and over time. If dye is incorporated into active wear clothing then it is desirable to prevent the color from fading from the clothing.

[0007] Therefore, it would be desirable to provide a dyed fabric, such as a synthetic fabric, in which the dye does not fade over time. Moreover, there is a need for a process for incorporating a dye into UV protected fabric that may be used to manufacture an article of clothing in which the dye does not fade during use. Furthermore, it would be advantageous to be able to manufacture an UV protected garment, fabric, or textile that accepts dye that will last over time and after multiple uses.

### BRIEF SUMMARY

[0008] In one form of the present disclosure, a fabric having ultraviolet radiation protection and antimicrobial protection comprises ZnO (zinc oxide) incorporated into the fabric and a dye incorporated into the fabric.

[0009] In another form of the present disclosure, a method for dyeing a fabric having incorporated therein ultraviolet radiation protection and antimicrobial protection comprises the steps of adding the fabric to a jet dyeing machine containing water, adding a scouring agent to the jet dyeing machine, heating the water and cooling the water, removing the water from the jet dyeing machine and refilling with water for a first time, agitating the fabric in the jet dyeing machine, adding a dye, a leveling agent, and a dispersing agent to the jet dyeing machine, heating the first refilled water and cooling the first refilled water, removing the first refilled water from the jet dyeing machine and refilling with water for a second time, heating the second refilled water to a certain temperature, agitating the fabric in the jet dyeing machine, adding a surfactant to the jet dyeing machine, maintaining the second refilled water at the certain temperature and cooling the second refilled water, removing the second refilled water from the jet dyeing machine, and removing the fabric from the jet dyeing machine.

[0010] In yet another form of the present disclosure, a fabric having ultraviolet radiation protection and antimicrobial protection comprises a quantity of ZnO incorporated into the fabric and a quantity of a dye incorporated into the fabric.

[0011] In another form of the present disclosure, a method for dyeing a fabric having incorporated therein ultraviolet radiation protection and antimicrobial protection comprises the steps of adding the fabric to a jet dyeing machine containing water, agitating the fabric, adding a scouring agent to the jet dyeing machine, heating the water, maintaining the water at a certain temperature for a period of time, and cooling the water after the period of time, remov-

ing the water from the jet dyeing machine and refilling with water for a first time, agitating the fabric in the jet dyeing machine, adding a dye, a leveling agent, and a dispersing agent to the jet dyeing machine, heating the first refilled water, maintaining the first refilled water at a certain temperature for a period of time, and cooling the first refilled water after the period of time, removing the first refilled water from the jet dyeing machine and refilling with water for a second time, heating the second refilled water, agitating the fabric in the jet dyeing machine, adding a surfactant to the jet dyeing machine, maintaining the second refilled water at a certain temperature for a period of time, and cooling the second refilled water after the period of time, removing the second refilled water from the jet dyeing machine, and removing the fabric from the jet dyeing machine.

**[0012]** In still another form of the present disclosure, a synthetic fabric having ultraviolet radiation protection and antimicrobial protection comprises a quantity of ZnO incorporated into the synthetic fabric and a quantity of a dye incorporated into the synthetic fabric.

**[0013]** In yet another form of the present disclosure, a method for dyeing a synthetic fabric having incorporated therein ultraviolet radiation protection and antimicrobial protection comprises the steps of adding the synthetic fabric to a jet dyeing machine containing water, adding a scouring agent to the jet dyeing machine, heating the water to a certain temperature at a certain rate, maintaining the water at the certain temperature for a period of time, and cooling the water after the period of time to a certain temperature at a certain rate, removing the water from the jet dyeing machine and refilling with water for a first time, agitating the synthetic fabric in the jet dyeing machine, adding a dye, a leveling agent, and a dispersing agent to the jet dyeing machine while agitating the synthetic fabric, heating the first refilled water to a certain temperature at a certain rate, maintaining the first refilled water at the certain temperature for a period of time, and cooling the first refilled water after the period of time to a certain temperature at a certain rate, removing the first refilled water from the jet dyeing machine and refilling with water for a second time, heating the second refilled water to a certain temperature at a certain rate, agitating the synthetic fabric in the jet dyeing machine, adding a surfactant to the jet dyeing machine while agitating the synthetic fabric, maintaining the second refilled water at the certain temperature for a period of time, and cooling the second refilled water after the period of time to a certain temperature at a certain rate, removing the second refilled water from the jet dyeing machine, and removing the synthetic fabric from the jet dyeing machine.

**[0014]** The present disclosure provides a dyed fabric having incorporated therein ultraviolet radiation protection and antimicrobial protection to be used to produce or manufacture a dyed fabric which is lightweight and can be worn in any temperature.

**[0015]** The present disclosure provides a dyed fabric having incorporated therein ultraviolet radiation protection and antimicrobial protection for providing enhanced protection from both UVA and UVB radiation.

**[0016]** The present disclosure provides a dyed fabric having incorporated therein ultraviolet radiation protection and antimicrobial protection for providing a dyed fabric in which the dye will last for an extended period of time.

**[0017]** The present disclosure also provides a dyed fabric having incorporated therein ultraviolet radiation protection and antimicrobial protection which retains ultraviolet radiation protection and antimicrobial protection after use or after cleaning.

**[0018]** The present disclosure also provides a dyed fabric having incorporated therein ultraviolet radiation protection and antimicrobial protection which retains dye after use or after cleaning.

**[0019]** The present disclosure provides a dyed fabric having incorporated therein ultraviolet radiation protection and antimicrobial protection to be used to produce or manufacture a fabric which is comfortable to wear.

**[0020]** The present disclosure provides a dyed fabric having incorporated therein ultraviolet radiation protection and antimicrobial protection which can be manufactured without increasing the cost of the fabric.

**[0021]** The present disclosure provides a dyed fabric having incorporated therein ultraviolet radiation protection and antimicrobial protection that is incorporated into active wear clothing or athletic clothing.

**[0022]** The present disclosure is also directed to a method for dyeing a fabric having incorporated therein ultraviolet radiation protection and antimicrobial protection for providing a dyed fabric in which the dye will last for an extended period of time.

**[0023]** The present disclosure also provides a method for dyeing a fabric having incorporated therein ultraviolet radiation protection and antimicrobial protection to be used to produce or manufacture a dyed fabric which is lightweight and can be worn in any temperature.

**[0024]** These and other advantages of the present disclosure will become apparent after considering the following detailed specification in conjunction with the accompanying drawings, wherein:

#### BRIEF DESCRIPTION OF THE DRAWINGS

**[0025]** FIG. 1 is a flowchart diagram of a method for incorporating ultraviolet radiation protection and antimicrobial protection into rayon;

**[0026]** FIG. 2 is a flowchart diagram of another method for incorporating ultraviolet radiation protection and antimicrobial protection into rayon;

**[0027]** FIG. 3 is a flowchart diagram of a method for dyeing a fabric having incorporated therein UV radiation protection and antimicrobial protection;

**[0028]** FIG. 4 is another flowchart diagram of a method for dyeing a fabric having incorporated therein UV radiation protection and antimicrobial protection;

#### DETAILED DESCRIPTION OF A PREFERRED EMBODIMENT

**[0029]** Various methods or processes are disclosed herein for the immobilization of UV-blocking nanoparticles on Eco-friendly fabric to incorporate UV protection in fabric. Once the UV-blocking nanoparticles are attached, the Eco-friendly fabric will be able to protect a wearer of the fabric from UV radiation. One method comprises direct immobilization from in situ formation of the particles. A second method comprises carboxylation or phosphorylation of the fabric followed by binding of the UV-blocking nanoparticles to the modified fabric. A third method comprises modifying UV-blocking nanoparticles with a self-assembled monolayer

(SAM) or polymer layer containing an active chemical group capable of binding to the fabric and deposited on the fabric from solution. Once these fabrics are prepared incorporating UV protection and antimicrobial protection, by one or more of the following methods, various dyeing methods are described further herein to dye these treated fabrics.

**[0030]** ZnO nanoparticles are generally formed by the precipitation of a zinc salt (acetate, sulfate, nitrate, chloride) using either aqueous hydroxide or an amine. The following examples disclose direct immobilization from in situ formation of the ZnO nanoparticles.

#### Example 1 Solution Sol-Gel Process, Hydroxide Base

**[0031]** 4.39 g. zinc acetate (20 mmol) is dissolved in 100 mL deionized or distilled water. A textile is added to this solution and 100 mL 0.4M NaOH is added while mixing. The suspension is mixed for 2 hours to form a suspension of zinc oxide nanoparticles in contact with the fabric. The textile is removed from the nanoparticle suspension and laundered in a household washing machine. As can be appreciated, a fabric may be treated to have ultraviolet radiation protection incorporated in the fabric by the steps of dissolving zinc acetate or other zinc salt in a liquid to form a solution containing Zn (II) ions, adding a fabric to the solution, mixing the solution and the fabric, and adding a base to the solution when the solution and the fabric are being mixed to form a suspension of zinc oxide nanoparticles in contact with the fabric.

#### Example 2 Solution Sol-Gel Process, Amine Base

**[0032]** 4.39 g. zinc acetate (20 mmol) is dissolved in 100 mL deionized water. A textile is added to this solution while mixing and 40 mmol amine is added while mixing. Amines used may include ethanolamine, ethylenediamine, (tris) hydroxymethylaminomethane, or others. The textile is removed from the nanoparticle suspension and laundered in a household washing machine.

#### Example 3 Mechanochemical Process

**[0033]** 5.75 g. zinc sulfate heptahydrate (20 mmol) and 0.88 g (15 mmol) sodium chloride are powdered finely and blended, then placed with a textile in a ball mill or similar mechanical mixer. 1.6 g (40 mmol) sodium hydroxide is powdered and added to the mixer. After twenty minutes, the textile is removed and rinsed thoroughly with water.

**[0034]** The following examples disclose carboxylation or phosphorylation of the fabric followed by binding of the UV-blocking nanoparticles to the modified fabric.

#### Example 4 Modification of Textile with Phosphonic Acid Groups

**[0035]** For this process it will be necessary to modify a textile with phosphonic acid groups. This can be accomplished in a number of ways, but it is desirable to use materials that are non-toxic and/or renewably sourced chemicals. Phosphorylated cellulose should form covalent linkages with ZnO and TiO<sub>2</sub> nanoparticles. The interaction between phosphonates and oxide surfaces are used for modification of the oxide surfaces. In essence, the procedure consists of condensing the cellulose textile with a bis (phosphonic acid), phosphonate, or phosphate species, either organic or inorganic. Urea may be added to forestall discoloration of the textile. Phosphorylation takes place driven by the elimination of water. The resulting phosphorylated textile will directly bind both zinc oxide and titanium oxide nanoparticles. It will be necessary to restrict the degree of phosphorylation of the textile to prevent great alteration in the properties of the textile by controlling a reaction time. This process does not require in situ synthesis of the zinc oxide nanoparticles. Commercially available zinc oxide nanoparticles may be used.

**[0036]** A sample of cotton textile is wetted with a 10% v/v solution of phosphoric acid or bis-phosphonic acid containing 10-30% w/v urea. The textile is pressed to remove excess solution and baked in an oven at 85-100° C. for 5 minutes to dry, then at 170° C. for 2-4 minutes to cure unreacted groups. The textile is removed from the oven and washed with water. The textile is then used without further modification in subsequent deposition steps.

#### Example 5 Modification of a Textile by Partial TEMPO-H<sub>2</sub>O<sub>2</sub> Oxidation

**[0037]** A sample of cotton textile (ca. 1 g) is added to a solution composed of 90 mL water with 10 mg (0.065 mmol) TEMPO and 0.22 g (2 mmol) sodium bromide. Hydrogen peroxide 3% is added (0.9 mL, 1 mmol) and the reaction stirred at RT for 10 minutes to 2 hours. The material is washed with water, dried, and used without further modification in the following ZnO deposition step.

#### Example 6 Immobilization of Nanoparticles on a Phosphorylated or Carboxylated Cellulose Surface

**[0038]** Ca. 1 mg/mL nanoparticles are suspended in water, ethyl alcohol, or other solvent. The phosphorylated or carboxylated cellulose textile is added to the suspension and the suspension is gently mixed over a reaction period of 1 to 12 hours. The textile is removed from the suspension and subjected to tumble drying or another drying procedure to force surface condensation and cure remaining groups.

**[0039]** The following example discloses modifying UV-blocking nanoparticles with a self-assembled monolayer (SAM) or polymer layer containing an active chemical group capable of binding to the fabric and deposited on the fabric from solution.

#### Example 7 Grafting to Attachment of Cellulose to Nanoparticles Through Reactive Groups

**[0040]** In this method, ZnO particles are synthesized separately by any of the means discussed in Examples 1-3 or the ZnO particles may be purchased commercially. The ZnO particles are suspended in water or a weak non-nucleophilic aqueous buffer and an organosilane or phosphonate with one of the given combinations of reactive groups, as shown in Table 1, is added. Multidentate ligand or polymeric silanes may also be added to this mixture to facilitate the formation of a durable reactive layer and an oxide, alkoxide, or salt of another metal such as Ti or Si may be added first to form a surface layer of another oxide in the ZnO particles. After a reaction time of 1 to 12 hours, the particles are collected by centrifugation and washed with water. The particles are then resuspended in water or buffer and added to the textile. The conditions for binding of the particles to the textile vary depending on the headgroup, as shown in Table 1, but may involve direct application of the particles to the textile similarly to the process disclosed in Example 6, raising the



pH of the suspension containing the textile, or heating the textile either in or after removal from the suspension. This process has the advantage of yielding extremely fine control over the nature of the linkage between particle and textile. This process has a further advantage in that the treated textile will be durable due to the robustness of self-assembled siloxane layers on oxide.

the desired functional group. The multidentate silane yields a more densely crosslinked siloxane surface than monodentate silanes alone, forming a more stable layer on ZnO.

**[0043]** Although the above examples and methods are applicable to the manufacturing process in which ultraviolet radiation protection is incorporated into the fabric, textile, or garment when initially produced, the following discloses

TABLE 1

Molecule name (if commercially available)	Linker	Headgroup	Commercially available?
3-glycidioxypropyl-triethoxysilane	Triethoxysilane	Glycidyl ether	Yes
2-(3,4-cyclohexyloxy)ethyltriethoxysilane	Triethoxysilane	Cyclohexyl oxide	Yes
Hydroxymethyl-triethoxysilane	Triethoxysilane	Hydroxymethyl	Yes
Isocyanatopropyl trimethoxysilane	Trimethoxysilane	Isocyanate	Yes
Bis(triethoxysilyl) ethane	Triethoxysilane (2)	N/A	Yes
6-azidosulfonylhexyl triethoxysilane	Triethoxysilane	Azidosulfonyl	Yes
	Triethoxysilane	Vinylsulfone	No
	Triethoxysilane	Aryl azide	No
	Phosphonate	Glycidyl ether	No
	Phosphonate	Cyclohexyl oxide	No
	Phosphonate	Azidosulfonyl	No
	Phosphonate	Vinylsulfone	No
	Phosphonate	Aryl azide	No
Bis(triethoxysilyl) propylamine	Triethoxysilane (2)	Secondary amine	Yes
APTES/EGDE	Triethoxysilane	Amine/Ethylene glycol diglycidyl ether	Yes, 2 components

**[0041]** The terms “fabric” or “textile” are intended to include fibers, filaments, yarn, melt, textiles, material, woven and non-woven fabric, knits, and finished products such as garments. The methods described herein may be used in treating fibers, filaments, yarn, textiles, and fabrics. For example, fibers may be initially treated by use of one or more of the disclosed methods and the fibers may be manufactured into a fabric or a textile. Once manufactured into a fabric, the fabric may be treated by use of one or more of the disclosed methods. In this manner, individual fibers and the entire fabric are treated to incorporate UV protection. As can be appreciated, the treated fabric may be used to manufacture a garment such as, by way of example only, shirts, pants, gloves, hats, coats, jackets, shoes, socks, uniforms, athletic clothing, and swimwear. It is also possible and contemplated that the treated fabric may be used to construct non-apparel items such as blankets, sheets, sleeping bags, backpacks, and tents.

**[0042]** Further, it is also possible to further modify ZnO particles with a thin layer of other oxides in a “core-shell” type procedure by adding a reactive precursor to a suspension of the ZnO oxides. Oxides that can be deposited in this manner include SiO<sub>2</sub> from tetraethoxysilane (TEOS) or sodium silicate, and Al<sub>2</sub>O<sub>3</sub> and TiO<sub>2</sub> either from the appropriate alkoxides, aluminate/titanate compounds, or other hydrolyzable aluminum or titanium compounds. A second oxide shell of this type may enhance the formation and stability of both directly applied ZnO-textile conjugates and those formed by modification of nanoparticles with an organic monolayer. ZnO can also be modified by the addition of a multidentate silane along with a silane containing

various methods of incorporating ultraviolet radiation protection directly to clothing being laundered. By use of the following methods, a garment after purchase may be made a protected garment by an end user.

**[0044]** In general, the methods may comprise the self-assembly of certain polyanionic materials onto a ZnO surface to create a linker which will bind the particles to a cellulose (cotton) surface. Several acidic or oxyanion functional groups are capable of self-assembly onto ZnO. These functional groups include siloxane, silanol, carboxylic acid, carboxylate, phosphonic acid, phosphonate, boronic acid or other groups capable of binding to oxide layers. Boronic acid is capable of forming very strong interactions with carbohydrates, including the glycosidically linked glucose units making up cellulose. One method or approach is to prepare a polymer bearing boronic acid groups and use that polymer to bind ZnO to cotton.

**[0045]** All synthetic material such as polyester and nylon that is used in the manufacture of athletic clothing or active wear clothing may be rendered UV-absorbing using a ZnO preparation. These types of fabrics may resist treatment using the methods as outlined with respect to Examples 8, 9, and 10. One solution to this problem is to prepare ZnO particles coated with functional groups capable of being grafted directly to polyester or nylon materials. This may be accomplished by using benzophenone photografting chemistry. The following examples and methods are applicable to the manufacturing process in which ultraviolet radiation protection is incorporated into the artificial or synthetic composition, polymer, fabric, textile, or garment when initially produced.

**[0046]** The following methods provide for the direct grafting of ZnO particles to nonpolar, non-natural polymers such as nylon and polyester. Nylon and polyester have little in the way of chemical functionality, containing only aliphatic and aromatic C—H bonds and amide or ester linkages between monomers. The method is capable of directly functionalizing C—H bonds. The following method describes preparing ZnO particles coated with functional groups capable of being grafted directly to polyester or nylon materials by using the photografting reaction of benzophenone.

#### Example 11 Grafting ZnO onto Artificial or Synthetic Fibers

**[0047]** In this method, an artificial fabric composed of polyester, nylon, or other polymer lacking hydroxyl functional group is modified by use of a preparation of a zinc oxide particle modified with a layer of reactive groups capable of C—H activation. Examples of the reactive functional group capable of C—H activation are benzophenone, sulfonylazides, aryl azides, or diazonium salts. The prepared particles are coated onto the fabric and a reaction is initiated using UV light, heat, or both. By way of example only, a mercury-vapor UV lamp may be used and the time for exposure may be one hour. Unbound particles are washed off the fabric. This second step, a curing step, bonds the prepared particles to the fabric. This method adds a second UV-absorbing chromophore which cross-links and becomes further bonded to the polymer surface of the fabric upon exposure to UV light. In this method, zinc oxide particles can be composed of pure zinc oxide or zinc oxide coated with aluminum, titanium, or silicon oxides in a core-shell configuration. The result is an artificial fabric with photografted zinc oxide particles.

**[0048]** By way of example, the zinc oxide particles were prepared in the following manner. Five grams of zinc oxide nanoparticles were used and suspended in a solution of 98% ethyl alcohol. Two grams of benzophenone silane linker were suspended in this solution and the pH of the solution was adjusted to 12. After twelve hours, the zinc oxide particles were recovered by centrifugation and dried overnight at 50-60° C. in an oven.

**[0049]** It is also possible to prepare a phosphoether of 4-hydroxybenzophenone and use this self-assembling molecule to functionalize ZnO particles. The resulting particles, having a monolayer of nonpolar molecules, will be substantially nonpolar and will adhere to nonpolar polyester and nylon. In order to bond the particles to the polymer surface an UV light may be used to initiate a reaction. Again, the process has the advantage of adding a second UV absorbing chromophore which cross-links and becomes further bonded to the polymer surface upon exposure to UV light.

**[0050]** The following describes an additive for incorporating UV protection into a polymer prior to the polymer being placed into a spinneret or prior to the polymer being formed into fibers. Nylon and polyester have little in the way of chemical functionality, containing only aliphatic and aromatic C—H bonds and amide or ester linkages between monomers. The additive is capable of directly functionalizing C—H bonds.

#### Example 12 Additive

**[0051]** An artificial fabric composed of polyester, nylon, or other polymer lacking hydroxyl functional group is

modified by use of an additive of a quantity of zinc oxide particles modified with a layer of a reactive group that forms a bond with a synthetic polymer having C—H bonds. Examples of the reactive functional group capable of C—H activation are benzophenone, sulfonylazides, aryl azides, diazonium salts, isocyanate, oxime, and azo. The prepared particles may be added to the synthetic polymer prior to the synthetic polymer being placed into a spinneret. Further, it is also contemplated that the additive may be packaged with the synthetic polymer and the packaged additive and synthetic polymer may be placed into the spinneret. The modified zinc oxide particles can also be coated with aluminum, titanium, or silicon oxides in a core-shell configuration.

**[0052]** By way of example, the zinc oxide particles were prepared in the following manner. A quantity of zinc oxide particles was suspended in a solution of 98% ethyl alcohol, a quantity of benzophenone silane linker was suspended in the solution of zinc oxide particles and 98% ethyl alcohol, the pH of the solution of zinc oxide particles, 98% ethyl alcohol, and benzophenone silane linker was adjusted to 12, the pH adjusted solution of zinc oxide particles, 98% ethyl alcohol, and benzophenone silane linker was placed into a centrifuge, the zinc oxide particles prepared by centrifugation was recovered after a period of time, and the recovered prepared zinc oxide particles were dried. By further way of example only, five grams of zinc oxide nanoparticles were used and suspended in a solution of 98% ethyl alcohol. Two grams of benzophenone silane linker were suspended in this solution and the pH of the solution was adjusted to 12. After twelve hours, the zinc oxide particles were recovered by centrifugation and dried overnight or for eight hours at 50-60° C. in an oven.

**[0053]** By way of example only and in not a limiting sense, it is also possible to prepare a phosphoether of 4-hydroxybenzophenone and use this self-assembling molecule to functionalize ZnO particles. The resulting particles, having a monolayer of nonpolar molecules, will be substantially nonpolar and will adhere to nonpolar polyester or nylon. The resulting or modified zinc oxide particles can also be coated with aluminum, titanium, or silicon oxides in a core-shell configuration. Further, it is to be understood that many other benzophenone derivatives are suitable for use to prepare a self-assembling molecule to functionalize ZnO particles.

**[0054]** Synthetic material such as rayon that is used in the manufacture of athletic clothing or active wear clothing may be rendered UV-absorbing and antimicrobial using a ZnO preparation. This type of fabric may resist treatment using the methods as outlined with respect to Examples 8, 9, and 10. One solution to this problem is to prepare ZnO particles coated with functional groups capable of being grafted directly to rayon material. This may be accomplished by using benzophenone photografting chemistry. The following examples and methods are applicable to the manufacturing process in which ultraviolet radiation protection and antimicrobial protection are incorporated into the rayon polymer, fabric, textile, or garment when initially produced.

**[0055]** The following methods provide for the direct grafting of ZnO particles to rayon. The following method describes preparing ZnO particles coated with functional groups capable of being grafted directly to rayon material by using the photografting reaction of benzophenone.

**[0056]** Most commercial rayon production utilizes the viscose process. In particular, reference is made to FIG. 1 in

connection with the following description of a flowchart diagram for a method for incorporating ultraviolet radiation protection and antimicrobial protection into rayon **10**. This process or method may comprise the following steps. Initially, in a first step **12**, purified cellulose is provided from specially processed wood pulp to form cellulose sheets. The cellulose sheets are saturated with a solution of caustic soda or sodium hydroxide. The solution is allowed to steep for enough time so that the caustic solution penetrates the cellulose to convert some of it into soda cellulose, the sodium salt of cellulose. This is known as the steeping step and is illustrated in a step **14**. This is necessary to facilitate controlled oxidation of the cellulose chains and the ensuing reaction to form cellulose xanthate. The soda cellulose is squeezed mechanically to remove any excess caustic soda solution. This is known as the pressing step and is shown in a step **16**. The soda cellulose is mechanically shredded to increase surface area and to make the cellulose easier for further processing. This is known as the shredding step and is depicted in a step **18**. This shredded cellulose is sometimes referred to as "white crumb". White crumb is then allowed to stay in contact with ambient air so that an oxidation process occurs. The high alkalinity of the white crumb partially oxidizes the cellulose to degrade the cellulose to lower molecular weights. Degradation of the cellulose must be carefully controlled in order to produce chain lengths short enough to provide manageable viscosities in the spinning solution. However, the chain lengths must be long enough to provide good physical properties to the fiber product. This is known as the aging step, as is shown in a step **20**. Once the white crumb is properly aged the white crumb is placed in a churn or other mixing vessel. Once in the churn the white crumb is treated with gaseous carbon disulfide. The soda cellulose reacts with the carbon disulfide to form xanthate ester groups. The carbon disulfide also reacts with the alkaline medium to form inorganic impurities which give the cellulose mixture a yellow color and this material is called "yellow crumb". The yellow crumb is a block copolymer of cellulose and cellulose xanthate because accessibility to the carbon disulfide is restricted in the crystalline regions of the soda cellulose. As illustrated in a next step **22**, this is known as the xanthation step. In a next step **24**, known as the dissolving step, the yellow crumb is dissolved in aqueous caustic solution. In the dissolving step **24**, an additive, as disclosed herein, is added or introduced in a step **26**. For example, the yellow crumb may be provided to a dissolving tank and the additive may also be provided to the dissolving tank. The amount of additive added to the dissolving tank may be 1-2% based on the weight of the dissolved cellulose. The large xanthate substituents on the cellulose force the chains apart, reducing the interchain hydrogen bonds and allowing water molecules to solvate and separate the chains. This leads to a solution of insoluble cellulose. The yellow crumb is not completely soluble at this stage due to the blocks of un-xanthated cellulose in the crystalline regions. The cellulose xanthate solution or suspension has a very high viscosity. The viscose is allowed to stand for a period of time to ripen. This is known as the ripening step and is shown as a step **28**. In a next step **30**, a filtering step, the viscose is filtered to remove undissolved materials that might disrupt the spinning process or cause defects in the rayon filament. The very next step in the process is known as a degassing step **32**. In the degassing step **32** bubbles of air trapped in the viscose are

removed. After the degassing step **32** is a step known as the spinning or wet spinning step **34**. In the spinning step the viscose is forced through a spinneret. The spinneret has a number of small holes and each hole produces a fine filament of viscose. The result of the spinning step is the formation of fine filaments of rayon having ultraviolet radiation protection and antimicrobial protection incorporated therein. In a next step, known as the drawing step **36**, the rayon filaments are stretched while the cellulose chains are still relatively mobile. The rayon filaments are washed to remove any salts or other water soluble impurities. This is the washing step of the process and is shown as a step **38**. Finally, the rayon may be passed through a rotary cutter to provide a fiber which can be processed in much the same way as cotton. This is the cutting step, which is illustrated as a step **40**. As can be appreciated, when the quantity of rayon is treated or incorporated with the additive, as discussed herein, the rayon has the properties of ultraviolet radiation protection and antimicrobial protection.

[0057] Referring now to FIG. 2, another embodiment of a method for incorporating ultraviolet radiation protection and antimicrobial protection into rayon **100** is shown. The method **100** comprises the following steps. Initially, in a first step **102**, purified cellulose is provided from specially processed wood pulp to form cellulose sheets. The cellulose sheets are saturated with a solution of caustic soda or sodium hydroxide. The solution is allowed to steep for enough time so that the caustic solution penetrates the cellulose to convert some of it into soda cellulose, the sodium salt of cellulose. This is known as the steeping step and is illustrated in a step **104**. This is necessary to facilitate controlled oxidation of the cellulose chains and the ensuing reaction to form cellulose xanthate. The soda cellulose is squeezed mechanically to remove any excess caustic soda solution. This is known as the pressing step and is shown in a step **106**. The soda cellulose is mechanically shredded to increase surface area and to make the cellulose easier for further processing. This is known as the shredding step and is depicted in a step **108**. This shredded cellulose is sometimes referred to as "white crumb". White crumb is then allowed to stay in contact with ambient air so that an oxidation process occurs. The high alkalinity of the white crumb partially oxidizes the cellulose to degrade the cellulose to lower molecular weights. Degradation of the cellulose must be carefully controlled in order to produce chain lengths short enough to provide manageable viscosities in the spinning solution. However, the chain lengths must be long enough to provide good physical properties to the fiber product. This is known as the aging step, as is shown in a step **110**. Once the white crumb is properly aged the white crumb is placed in a churn or other mixing vessel. Once in the churn the white crumb is treated with gaseous carbon disulfide. The soda cellulose reacts with the carbon disulfide to form xanthate ester groups. The carbon disulfide also reacts with the alkaline medium to form inorganic impurities which give the cellulose mixture a yellow color and this material is called "yellow crumb". The yellow crumb is a block copolymer of cellulose and cellulose xanthate because accessibility to the carbon disulfide is restricted in the crystalline regions of the soda cellulose. As illustrated in a next step **112**, this is known as the xanthation step. In a next step **114**, known as the dissolving step, the yellow crumb is dissolved in aqueous caustic solution. For example, the yellow crumb may be provided to a dissolving tank and the

additive may also be provided to the dissolving tank. The large xanthate substituents on the cellulose force the chains apart, reducing the interchain hydrogen bonds and allowing water molecules to solvate and separate the chains. This leads to a solution of insoluble cellulose. The yellow crumb is not completely soluble at this stage due to the blocks of un-xanthated cellulose in the crystalline regions. The cellulose xanthate solution or suspension has a very high viscosity. The solution is then provided to a homogenizer, such as a tank, at this point in the process, which is a step **116**. Once the solution is provided to the homogenizer an additive may be introduced into the tank and this is an adding additive step **118**. The amount of additive added to the tank may be 1-2% based on the weight of the dissolved cellulose. The viscose is allowed to stand for a period of time to ripen. This is known as the ripening step and is shown as a step **120**. In a next step **122**, a filtering step, the viscose is filtered to remove undissolved materials that might disrupt the spinning process or cause defects in the rayon filament. Further, it is important to note that the size of the particles of the additive need to be small enough to be able to pass through a filter utilized in the filtering step **122**. The very next step in the process is known as a degassing step **124**. In the degassing step **124** bubbles of air trapped in the viscose are removed. After the degassing step **124** is a step known as the spinning or wet spinning step **126**. In the spinning step the viscose is forced through a spinneret. The spinneret has a number of small holes and each hole produces a fine filament of viscose. The result of the spinning step **126** is the formation of fine filaments of rayon having ultraviolet radiation protection and antimicrobial protection incorporated therein. In a next step, known as the drawing step **128**, the rayon filaments are stretched while the cellulose chains are still relatively mobile. The rayon filaments are washed to remove any salts or other water soluble impurities. This is the washing step of the process and is shown as a step **130**. Finally, the rayon may be passed through a rotary cutter to provide a fiber which can be processed in much the same way as cotton. This is the cutting step, which is illustrated as a step **132**. As can be appreciated, when the quantity of rayon is treated or incorporated with the additive, as discussed herein, the rayon has the properties of ultraviolet radiation protection and antimicrobial protection. Further, although not shown in FIG. 1, it is also possible to incorporate the step **116** wherein the viscose is provided to a homogenizer.

**[0058]** As can be appreciated, various other steps in the above described methods may be included. By way of example only, some other steps may include providing a slurry tank, providing a slurry press, providing an aging drum, providing a hopper, providing a heat exchanger, providing a ripening tank, providing vacuum, providing a deaerator, providing a spinning tank, providing a stretching mechanism or machine, providing steam, providing a drier and opener, and providing a bale press.

**[0059]** The following describes an additive for incorporating UV protection and antimicrobial protection into rayon as described in the methods shown in FIGS. 1 and 2. Rayon has little in the way of chemical functionality, containing only aliphatic and aromatic C—H bonds and amide or ester linkages between monomers. The additive is capable of directly functionalizing C—H bonds.

#### Example 13 Additive

**[0060]** An artificial fabric composed of rayon is modified by use of an additive of a quantity of zinc oxide particles modified with a layer of a reactive group that forms a bond with rayon having C—H bonds. Examples of the reactive functional group capable of C—H activation are benzophenone, sulfonylazides, aryl azides, diazonium salts, isocyanate, oxime, and azo. The prepared particles may be added during the process of manufacturing rayon so that the particles are added prior to the rayon being placed into a spinneret or prior to a wet spinning step. Further, it is also contemplated that the additive may be packaged with rayon and the packaged additive and rayon may be placed into the spinneret. The modified zinc oxide particles can also be coated with aluminum, titanium, or silicon oxides in a core-shell configuration.

**[0061]** By way of example, the zinc oxide particles were prepared in the following manner. A quantity of zinc oxide particles was suspended in a solution of 98% ethyl alcohol, a quantity of benzophenone silane linker was suspended in the solution of zinc oxide particles and 98% ethyl alcohol, the pH of the solution of zinc oxide particles, 98% ethyl alcohol, and benzophenone silane linker was adjusted to 12, the pH adjusted solution of zinc oxide particles, 98% ethyl alcohol, and benzophenone silane linker was placed into a centrifuge, the zinc oxide particles prepared by centrifugation was recovered after a period of time, and the recovered prepared zinc oxide particles were dried. By further way of example only, five grams of zinc oxide nanoparticles were used and suspended in a solution of 98% ethyl alcohol. Two grams of benzophenone silane linker were suspended in this solution and the pH of the solution was adjusted to 12. After twelve hours, the zinc oxide particles were recovered by centrifugation and dried overnight or for eight hours at 50-60° C. in an oven. It is also possible and contemplated that the additive may comprise ZnO nanoparticles that are uncoated. It is further possible that the additive may comprise nanoparticles or particles that are uncoated and made by any of the methods described herein.

**[0062]** By way of example only and in not a limiting sense, it is also possible to prepare a phosphoether of 4-hydroxybenzophenone and use this self-assembling molecule to functionalize ZnO particles. The resulting particles, having a monolayer of nonpolar molecules, will be substantially nonpolar and will adhere to rayon. The resulting or modified zinc oxide particles can also be coated with aluminum, titanium, or silicon oxides in a core-shell configuration. Further, it is to be understood that many other benzophenone derivatives are suitable for use to prepare a self-assembling molecule to functionalize ZnO particles.

**[0063]** It is also possible to control the luster, sheen, or shininess of a fiber formed from an artificial material by varying an additive of zinc oxide particles percentage or concentration added to the artificial material prior to the artificial material being formed into a fiber. Zinc oxide particles may be used as an additive for matting, dulling, or gloss reduction of synthetic fibers. In particular, an artificial material is modified by use of an additive of a quantity of zinc oxide particles modified with a layer of a reactive group that forms a bond with artificial material having C—H bonds. Examples of the reactive functional group capable of C—H activation are benzophenone, sulfonylazides, aryl azides, diazonium salts, isocyanate, oxime, and azo. The prepared particles may be added to the artificial material

prior to the artificial material being placed into a spinneret or prior to a wet spinning step. Further, it is also contemplated that the additive may be packaged with artificial material and the packaged additive and artificial material may be removed from the packaging or container and placed into the spinneret. The modified zinc oxide particles can also be coated with aluminum, titanium, or silicon oxides in a core-shell configuration. In order to control the luster of the formed fiber, the percentage or concentration of zinc oxide particles may be added to artificial material in the following manner. To have a fiber that is bright, the percentage or concentration of zinc oxide particles will be added in the range of 0.05% to 0.10%. To have a fiber that is semi dull, the percentage or concentration of zinc oxide particles will be added in the range of 0.2% to 0.3%. Also, to have a fiber that is dull, the percentage or concentration of zinc oxide particles will be added at 0.5%. The zinc oxide particles cause the surface of the formed fiber to be rougher, reducing the sheen while at the same time being opaque which also reduces the transparency of the fiber.

**[0064]** By way of example, the zinc oxide particles used for dulling were prepared in the following manner. A quantity of zinc oxide particles was suspended in a solution of 98% ethyl alcohol, a quantity of benzophenone silane linker was suspended in the solution of zinc oxide particles and 98% ethyl alcohol, the pH of the solution of zinc oxide particles, 98% ethyl alcohol, and benzophenone silane linker was adjusted to 12, the pH adjusted solution of zinc oxide particles, 98% ethyl alcohol, and benzophenone silane linker was placed into a centrifuge, the zinc oxide particles prepared by centrifugation was recovered after a period of time, and the recovered prepared zinc oxide particles were dried. By further way of example only, five grams of zinc oxide nanoparticles were used and suspended in a solution of 98% ethyl alcohol. Two grams of benzophenone silane linker were suspended in this solution and the pH of the solution was adjusted to 12. After twelve hours, the zinc oxide particles were recovered by centrifugation and dried overnight or for eight hours at 50-60° C. in an oven.

**[0065]** By way of example only and in not a limiting sense, it is also possible to prepare a phosphoether of 4-hydroxybenzophenone and use this self-assembling molecule to functionalize ZnO particles. The resulting particles, having a monolayer of nonpolar molecules, will be substantially nonpolar and will adhere to rayon. The resulting or modified zinc oxide particles can also be coated with aluminum, titanium, or silicon oxides in a core-shell configuration. Further, it is to be understood that many other benzophenone derivatives are suitable for use to prepare a self-assembling molecule to functionalize ZnO particles.

**[0066]** Once fabric is manufactured by one or more of the above disclosed methods or by use of one or more of the above disclosed additives, dyeing the fabric may be accomplished by one or more of the following methods. Referring now to FIG. 3, a flowchart diagram for a method for dyeing a fabric having incorporated therein UV radiation protection and antimicrobial protection is illustrated. In a step **200**, fabric that has been manufactured to have incorporated therein UV radiation protection and antimicrobial protection is added to a jet dyeing machine containing water. In a step **202**, a scouring agent is added to the jet dyeing machine. The water is then heated and then cooled in a step **204**. The water is then removed from the jet dyeing machine and is refilled with water for a first time, as is shown in a step **206**. In a step

**208**, the fabric is agitated in the jet dyeing machine. A dye, a leveling agent, and a dispersing agent are then added to the jet dyeing machine in a step **210** while the jet dyeing machine is being agitated. The first refilled water is heated and then cooled in a step **212**. In a step **214**, the first refilled water is removed from the jet dyeing machine and then water is refilled into the jet dyeing machine for a second time. The second refilled water is then heated to a certain temperature, as is shown in a step **216**. The fabric is then agitated in the jet dyeing machine in a step **218**. A surfactant is then added to the jet dyeing machine as depicted in a step **220** while the jet dyeing machine is being agitated and the second refilled water is maintained at the certain temperature. In a step **222**, the second refilled water is then cooled. The second refilled water is removed from the jet dyeing machine. This is shown in a step **224**. Finally, in a step **226**, the fabric is removed from the jet dyeing machine and the fabric is dyed to a desired color. The dyed fabric may be used to manufacture a textile or a garment such as, by way of example only, shirts, pants, gloves, hats, coats, jackets, shoes, socks, uniforms, athletic clothing, and swimwear. It is also possible and contemplated that the treated fabric may be used to construct non-apparel items such as blankets, sheets, sleeping bags, backpacks, and tents. The fabric has ultra-violet radiation protection and antimicrobial protection and is comprised of ZnO incorporated into the fabric and the dye incorporated into the fabric.

**[0067]** Referring now to FIG. 4, another flowchart diagram for a method for dyeing a fabric having incorporated therein UV radiation protection and antimicrobial protection is shown. In particular, the method is, by way of example only, based on dyeing a 1 kg piece of synthetic fabric that has been manufactured to have incorporated therein UV radiation protection and antimicrobial protection. In a step **300**, the 1 kg piece of fabric is added to 10 liters of water in a jet dyeing machine and the fabric is agitated. In a step **302**, 10 grams (1.0 g/L) of a scouring agent, such as Sunmorr B-90, is added to the dyeing machine. In a step **304**, the water is heated to 60° C. at a rate of 2.5° C. per minute and held at 60° C. for 10 minutes and then cooled at a rate of 2.5° C. per minute until the temperature of the water reaches 35° C. In a step **306**, the water within the jet dyeing machine is removed and the jet dyeing machine is then refilled with 10 liters of water. In a step **308**, while the fabric is agitated, a dye such as 10 grams (1% on weight of fabric) of Dianix® Blue E-R 150%, a leveling agent such as 5 grams (0.5 g/L) of Leveling Sunsolt 1200K, and a dispersing agent such as 30 grams (3.0 g/L) of Carrier Sunsolt LM-7 are added to the jet dyeing machine. In a step **310**, the water in the jet dyeing machine is heated to 130° C. at a rate of 1.6° C. per minute and held at 130° C. for 30 minutes and the water is then cooled at a rate of 2.5° C. per minute until the temperature reaches 80° C. In a step **312**, the water is removed from the jet dyeing machine and then refilled with 10 liters of water and then heated to 60° C. at a rate of 2.5° C. per minute. In a step **314**, the fabric is agitated while a surfactant such as 30 grams (3.0 g/L) of BH-320 KS is added to the jet dyeing machine and the temperature of the water is maintained at 60° C. for 10 minutes and then cooled to 35° C. at a rate of 2.5° C. per minute. In a step **316**, the water is removed from the jet dyeing machine and the fabric is removed from the jet dyeing machine. As has been discussed, the dyed fabric may be used to manufacture a textile or a garment. Further, the fabric is a synthetic fabric having ultraviolet radiation pro-

tection and antimicrobial protection which comprises a quantity of ZnO incorporated into the synthetic fabric and a quantity of a dye incorporated into the synthetic fabric.

**[0068]** From all that has been said, it will be clear that there has thus been shown and described herein a method for dyeing fabric having incorporated therein UV radiation protection and antimicrobial protection which fulfills the various advantages sought therefore. It will become apparent to those skilled in the art, however, that many changes, modifications, variations, and other uses and applications of the subject method for dyeing fabric having incorporated therein UV radiation protection and antimicrobial protection are possible and contemplated. All changes, modifications, variations, and other uses and applications which do not depart from the spirit and scope of the disclosure are deemed to be covered by the disclosure, which is limited only by the claims which follow.

**1:** A fabric having ultraviolet radiation protection and antimicrobial protection comprising:

ZnO incorporated into the fabric;  
a quantity of a low foaming scouring agent incorporated into the synthetic fabric;  
a dye incorporated into the fabric;  
a leveling agent incorporated into the fabric; and  
a dispersing agent incorporated into the fabric.

**2:** The fabric of claim **1** wherein the fabric comprises a 1 kg piece of fabric.

**3:** The fabric of claim **1** wherein the leveling agent incorporated into the fabric comprises five grams.

**4:** The fabric of claim **1** wherein the dispersing agent incorporated into the fabric comprises thirty grams.

**5:** The fabric of claim **1** wherein the dye incorporated into the fabric comprises ten grams.

**6:** The fabric of claim **1** wherein the fabric comprises a 1 kg piece of fabric and the dye incorporated into the fabric comprises ten grams.

**7:** The fabric of claim **1** wherein the fabric comprises a 1 kg piece of fabric and the leveling agent incorporated into the fabric comprises five grams.

**8:** The fabric of claim **1** wherein the fabric comprises a 1 kg piece of fabric and the dispersing agent incorporated into the fabric comprises thirty grams.

**9:** The fabric of claim **1** wherein the leveling agent incorporated into the fabric comprises five grams and the dye incorporated into the fabric comprises ten grams.

**10:** The fabric of claim **1** wherein the dispersing agent incorporated into the fabric comprises thirty grams and the dye incorporated into the fabric comprises ten grams.

**11:** A fabric having ultraviolet radiation protection and antimicrobial protection comprising:

a quantity of ZnO incorporated into the fabric;  
a quantity of a very low foaming scouring agent incorporated into the synthetic fabric;  
a quantity of a dye incorporated into the fabric;  
a quantity of a leveling agent incorporated into the fabric;  
a quantity of a dispersing agent incorporated into the fabric; and  
a quantity of a surfactant incorporated into the fabric.

**12:** The fabric of claim **11** wherein the fabric is a 1 kg piece of fabric.

**13:** The fabric of claim **11** wherein the quantity of the dye is ten grams.

**14:** The fabric of claim **11** wherein the quantity of the leveling agent is five grams.

**15:** The fabric of claim **11** wherein the quantity of the dispersing agent is thirty grams.

**16:** The fabric of claim **11** wherein the quantity of the surfactant is thirty grams.

**17:** The fabric of claim **11** wherein the quantity of the dye is ten grams and the quantity of the leveling agent is five grams.

**18-19:** (canceled)

**20:** The synthetic fabric of claim **11** wherein the quantity of the scouring agent is ten grams.

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