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(71) **Applicant: TRIOMED INNOVATIONS CORP.** [US/US]; 40 Allen Road, South Burlington, VT 05403 (US).

(72) **Inventors: MESSIER, Pierre, Jean;** 14163 Boul, Labelle, Mirabel Quebec, J7J1M3 (CA). **OHAYON, David;** 14163 Boul. Labelle, Mirabel Quebec, J7J1M3 (CA).

(74) **Agent: DOWD, Betsy, Kingsbury;** Hoffinann & Baron, LLP, 6900 Jericho Turnpike, Syosset, NY 11791 (US).

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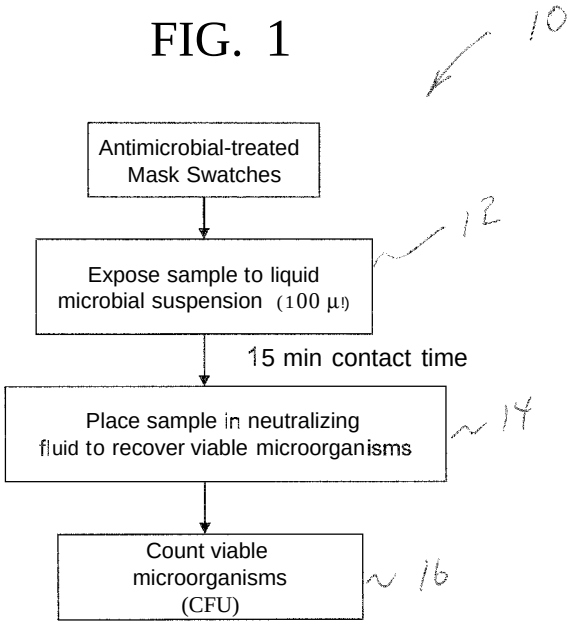
(54) **Title:** PROCESSES FOR PRODUCING ANTITOXIC FIBERS AND FABRICS



(57) **Abstract:** The invention provides a novel method for producing an antitoxic nonwoven fabric by molecularly grafting the antitoxic molecule thereto. The method comprises immersing a fibrous media comprising a material having a melt flow index of less than 150 MFI in a stable antitoxin solution comprising an antitoxin, preferably triiodide. The wet media is processed through rollers, thereby forcing the antitoxic molecule *(e.g.*, iodine) to penetrate the media. The wet media is dried, and the fabric isolated therefrom. The invention further provides products incorporating the antitoxic media formed by this molecularly grafting method, including a wound dressing, surgical drape, privacy curtain, facemask, gown, article of protective clothing, shoe covering, hair covering, air filter, medical tape, and wipe.

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PROCESSES FOR PRODUCING ANTITOXIC FIBERS AND FABRICS

CROSS-REFERENCE TO RELATED APPLICATIONS

[001] This applications claims the benefit of U.S. provisional application 61/568,982, filed on December 9, 2011, the entirety of which is incorporated herein.

FIELD OF THE INVENTION

[002] The present invention relates to antitoxic fibers and fabrics and methods for their manufacture, most particularly, to methods for molecular grafting of antitoxins to fibers and media formed therefrom. The invention also relates to products comprising the antitoxic fibers and fabrics so formed.

BACKGROUND OF THE INVENTION

[003] Various methods for producing antimicrobials for use in both nonwoven and woven fabrics are known. However, improvements in the production of antitoxins or antimicrobials and in the products that incorporate them, so that they exhibit both low toxicity and high efficacy, are still needed.

[004] A woven or nonwoven material can be loaded with antitoxin in different ways and at different points during or after processing of the material. For example, an antitoxic agent can be embedded in fibers of a nonwoven, incorporated into the interstitial spaces of a material, or glued or sprayed onto an outer layer of a fabric following production. The method of incorporation and the location of the antitoxic agent in the material may have important consequences in imparting the desired efficacy and toxicology to the material and resultant product.

[005] In the case of nonwoven materials, for example, one method involves physically entrapping the active agent within the three-dimensional structure of the nonwoven material. The active agent must have the appropriate size to be entrapped within the matrix structure of the nonwoven web. For instance, U.S. Publication No. 2006/0144403 (the '403 publication), to Messier, describes several methods of physically entrapping an active agent such as an iodine demand disinfectant resin in a three-dimensional nonwoven matrix. The '403 publication is hereby incorporated by reference in its entirety. Another method involves making use of a

meltblown system where the desired active agent is provided in a cloud at the location closest to the extrusion point of the fibers. The cloud of active agent envelops the extruded fibers exiting a spinneret. Upon cooling, the active agent becomes physically entrapped within the fibers on the collecting web.

[006] In addition to physically entrapping the active agent, certain methods of incorporating the active agent or antitoxin directly into the fiber are known. Generally, the active agent is blended with the polymer prior to extrusion so that it is present throughout the polymer. Upon solidification of the polymer, the active agent is dispersed throughout the resultant fiber. The active agent may diffuse to the surface of the nonwoven, where it exerts its toxic effect on the microorganism/toxin. For example, the '403 publication describes a method in which polymer granules are placed in a hopper along with active agent in powder form, preferably an iodine/resin disinfectant, prior to extrusion. The two components are then heated, extruded and attenuated to form fibers having the active agent incorporated therein. The resulting fibers having the active agent embedded can be air laid, vacuum laid or water laid. Nonwoven materials generated from this process can be utilized in various applications.

[007] Although methods described above produce efficacious materials, a significant loss of the antitoxic agent may be encountered during the various processing steps. In the meltblown procedure, for instance, it is found that the steps of heating and extrusion may result in sublimation or leeching of the antitoxic agent from the web. The same holds true for other downstream steps of the process. Co-owned Int'l. Pub. No. WO 201 1/103578 to Messier, *et ah,* entitled "Materials and Processes for Producing Antitoxic Fabrics" (the '578 publication), addresses these issues by providing methods of producing materials manufactured with higher concentrations of active antitoxic agent in the final product.

[008] In particular, the '578 publication discloses various methods for producing an antitoxic material by introducing iodine into a nonwoven material at various multiple stages of production. In one embodiment, a nonwoven material is formed from polymer staple fibers with an iodinated resin embedded therein, and then subjected to immersion in a liquid or gas containing triiodide or triiodine prior to being dried. The additional post-processing immersion step was found to increase the amount of active antitoxic agent that can be incorporated into a fabric.

[009] While the addition of an immersion step in the post-processing of the nonwoven was found to increase the amount of antitoxin in the product, and thus increase the measured kill performance, this additive step was also found to increase the amount of leaching and toxicity. In addition, the added immersion step increases the overall cost. Accordingly, a need still exists for a method of producing fibers and fabrics exhibiting increased antitoxin load capacity and efficacy over time combined with reduced levels of toxicity. Further reduction of the manufacturing cost resulting from wasted antitoxin in the manufacturing process is also desired.

SUMMARY

[010] The present invention provides cost-effective and efficient manufacturing processes for manufacturing fibers and fabrics formed therefrom, particularly nonwovens, containing antitoxins. The resultant fabrics exhibit advantageous properties such as increased antitoxin load capacity and efficacy combined with reduced levels of toxicity. The invention also provides products comprising the inventive antitoxic fibers and fabrics, such as: wound dressings, gowns, surgical drapes, protective clothing, shoe covers, hair covers, air filters, privacy curtains, and wipes.

[011] Although methods described above for entrapping antitoxic agents into the three-dimensional matrix or into the fibers of a nonwoven web produce efficacious materials, it is found that significant loss of the antitoxic agent may be encountered during processing. In the meltblown procedure, for instance, it is found that the steps of heating and extrusion may result in sublimation or leeching of the antitoxic agent from the web. The same holds true for other downstream steps of the process. The loss of antitoxic agent during the manufacturing process is costly. In addition, the unused antitoxins constitute undesirable hazardous waste materials that must be properly disposed of.

[012] The present invention addresses the need for efficient and cost-effective production of highly efficacious antitoxic fibers and fabrics. In particular, the novel manufacturing process of the present invention significantly increases the amount of active antitoxic agent that can be loaded or incorporated into a fiber and fibrous media formed therefrom using a molecular grafting technique employed in a single manufacturing step. The resultant fibrous media exhibit

both high efficacy and low toxicity. In addition, the manufacturing process is simplified and the amount of antitoxin lost during the process is advantageously reduced.

[013] In one aspect, a process for producing an antitoxic nonwoven fabric includes: providing a fibrous media comprising a material having a melt flow index of less than 150 MFI; forming a concentrated stable antitoxin solution comprising triiodide; fully immersing said media in said antitoxin solution to form a wet media; processing the wet media through rollers, thereby forcing the iodine to penetrate the media; and drying the wet media and isolating the fabric therefrom.

[014] The antitoxin solution may also comprise an active agent selected from the group consisting of iodine, bromine, chlorine and hydrogen peroxide.

[015] In various aspects, a wound dressing, surgical drape, privacy curtain, facemask, gown, article of protective clothing, air filter, shoe covering, hair covering, medical tape, or wipe comprises antitoxic fabrics formed according to the process described above.

[016] In a particular embodiment, an antimicrobial medical tape comprises a spunbond treated material with the antitoxin solution and, preferably, an adhesive liner.

[017] The fibrous media can be formed from a 50/50 blend of polypropylene and synthetic cellulose acetate or alginate fibers.

[018] Various aspects of a wound dressing of the present invention comprises a nonwoven formed from a 50/50 blend of polypropylene and synthetic cellulose acetate or alginate fibers having iodine molecularly grafted thereto from an immersion in a triiodide solution.

[019] In other aspects of the present invention, a facemask, for example, a surgical mask, or a privacy curtain comprises a nonwoven spunbond media characterized by an MFI of 30-40 having iodine molecularly grafted thereto from an immersion in a triiodide solution.

[020] In additional aspects, the media comprises polypropylene.

[021] In yet other aspects, the fibrous media is a nonwoven spunbond characterized by an MFI of 30-40.

[022] In further aspects, the triiodide solution has a concentration of at least 2000 ppm iodine, at least 2500, or at least 25,000 ppm iodine.

[023] In additional aspects, a surgical mask or privacy curtain comprises the antitoxic fabric formed according to the processes of the present disclosure, wherein the fibrous media comprises polypropylene, preferably a nonwoven, and wherein the antitoxin solution has a concentration of at least 2500 ppm iodine.

BRIEF DESCRIPTION OF THE DRAWINGS

[024] FIG. 1 is a representative flow diagram of a testing method for testing swatches of various antimicrobial media formed in accordance with the present disclosure.

[025] FIG. 2 is a table of results from the method of FIG. 1 for an embodiment of a facemask of the present disclosure.

[026] FIGS. 1-8 describe the results of bacterial challenges on a nonwoven layer, suitable for an outer layer of a facemask, formed by the molecular grafting method of the present disclosure.

[027] FIGS. 9-13 describe the results of bacterial challenges on a nonwoven layer, suitable for a curtain, formed in accordance with the molecular grafting method of the present disclosure.

DETAILED DESCRIPTION OF EMBODIMENTS

[028] The following sections describe exemplary embodiments of the present invention. It should be apparent to those skilled in the art that the described embodiments of the present invention provided herein are illustrative only and not limiting, having been presented by way of example only. All features disclosed in this description may be replaced by alternative features serving the same or similar purpose, unless expressly stated otherwise. Therefore, numerous other embodiments of the modifications thereof are contemplated as falling within the scope of the present invention as defined herein and equivalents thereto.

[029] Throughout the description, where items are described as having, including, or comprising one or more specific components, or where processes and methods are described as having, including, or comprising one or more specific steps, it is contemplated that, additionally, there are items of the present invention that consist essentially of, or consist of, the one or more

recited components, and that there are processes and methods according to the present invention that consist essentially of, or consist of, the one or more recited processing steps.

[030] It should be understood that the order of steps or order for performing certain actions is immaterial, as long as the invention remains operable. Moreover, two or more steps or actions may be conducted simultaneously.

[031] Scale-up and/or scale-down of systems, processes, units, and/or methods disclosed herein may be performed by those of skill in the relevant art. Processes described herein are configured for batch operation, continuous operation, or semi-continuous operation.

[032] It has been found and disclosed, for example, in co-owned U.S. Application Ser. No. 12/381,328, the contents of which are incorporated herein by reference, that when iodinated resin is embedded in a fiber, the amount of iodine released into the environment is substantially less than the amount of iodine released when the iodinated resin is in "free" powder form, and hence not associated with a fiber, as, for example, when physically adhered or glued to a substrate. Moreover, filter media comprising fibers with embedded iodinated resin leach negligible amounts of iodide and are thus not toxic. The '578 publication discloses that the amount of active antitoxic agent incorporated into a material formed from fibers embedded with iodinated resin could be further increased by adding a post-processing immersion step in the manufacturing process. However, the amount of leaching in the final product was found to increase compared to the non-woven prepared without the post-processing immersion step. Moreover, the extra immersion step increases the overall manufacturing cost.

[033] In the method of the present invention, an antitoxin is molecularly grafted to fibers and media formed therefrom in high concentrations through immersion of the media in a concentrated antitoxin solution. The resultant fabrics formed therefrom exhibit both superior efficacy and negligible toxicity. Moreover, the manufacturing process of the invention is drastically simplified and less costly compared to known methods of manufacturing antitoxic materials.

[034] In a preferred embodiment, the antitoxin solution is a triiodide solution.

[035] As used herein, triiodide refers to the triiodide ion, I_3^-, a polyatomic anion composed of three iodine atoms.

[036] The following examples, while limited to the molecular grafting of iodine from triiodide solution, are not intended to necessarily limit the application of the invention to any one antitoxin. Additional antitoxins contemplated for use in the present invention in place of, or in addition to, iodine include, but are not limited to, bromine, chlorine, fluorine and hydrogen peroxide.

[037] In alternative embodiments, the antitoxin solution for immersion of the fibrous media additionally comprises one of ethanol, 1-propanol, 2-propanol, isopropanol, cationic surfactant (e.g., benzalkonium chloride, chlorhexidine, octenidine dihydrochloride),metals , a quaternary ammonium compound (e.g., benzalkonium chloride (BAC), cetyl trimethylammonium bromide (CTMB), cetylpyridinium chloride (Cetrim, CPC), benzethonium chloride (BZT), chlorhexidine, octenidine), boric acid, brilliant green, chlorhexidine gluconate, mercurochrome, manuka honey, octenidine dihydrochloride, phenol (carbolic acid), sodium chloride, sodium hypochlorite, calcium hypochlorite, terpenes, or poly-hexa-methyl-biguanide (PHMB) or mixtures thereof. These active agents can be added alone or in combination with iodine molecule depending on the desired performance of the fabric produced.

[038] The fibers and fibrous media of the present invention can include any material, which when treated with an antitoxin in accordance with the present invention, can be defined as non-leachable; *e.g.,* a fibrous media formed therefrom exhibits no zone of inhibition, in accordance with the ASTM standard, designated E2149-01.

[039] The fibers and fibrous media prepared for immersion in the concentrated antitoxin solution in accordance with the present invention are also preferably free from hydrophobic coverings or coatings. In contrast, scrims used in some facemasks tend to be made from polypropylene fibers that are compounded with a wax that is hydrophobic in order to impart splash resistant qualities. Such media will not efficiently absorb triiodide and are not preferred for forming the antitoxic media of the present invention.

[040] It has been discovered that certain types of fibers can absorb a triiodide solution, formed as described below, and obtain a high concentration of iodine in the fiber, while exhibiting negligible or immeasurable leaching.

[041] For example, a preferred material for forming the fibrous media is polypropylene. Other materials contemplated for use in the present invention include polyethylene, polyamide, nylon, PVC, and EMAC amongst others.

[042] In one embodiment, an antitoxic nonwoven is formed by dipping the media in triiodide solution, wherein the nonwoven media comprises spunbond polypropylene fibers. The polypropylene media was found to be effective in absorbing the triiodide solution, as well as in releasing the active iodine agent in the presence of microorganisms. Products incorporating this antitoxic nonwoven, particularly, privacy curtains and facemasks, can be formed in accordance with the present invention.

[043] Another preferred fiber for use in the present invention is synthetic cellulose acetate.

[044] Yet another preferred fiber for use in the present invention is alginate.

[045] In various embodiments, a nonwoven media is formed from a blend of polypropylene and either synthetic cellulose acetate or alginate, then dipped in triiodide solution to form an antitoxic nonwoven according to the present invention. The media was found to be effective in absorbing the triiodide solution, as well as in releasing the active iodine agent in the presence of microorganisms.

[046] A particularly preferred antitoxic nonwoven media is formed from a 50/50 blend of (spunbond) polypropylene and synthetic cellulose acetate fibers dipped in triiodide solution. Another particularly preferred antitoxic nonwoven media is formed from a 50/50 blend of (spunbond) polypropylene and alginate fibers dipped in triiodide solution. Products, particularly wound dressings and antimicrobial tape, incorporating this antitoxic nonwoven are within the scope of this invention. Such wound dressings exhibit superior qualities over known dressings such as Silver-based dressings as well as other iodine dressings formed by other means.

[047] Surprisingly, it was discovered that when synthetic cellulose acetate or alginate was substituted with rayon or natural cellulose, the triiodide was absorbed in minimal amounts, and was absorbed preferentially and primarily by the polypropylene. Therefore, the blends comprising rayon or natural cellulose fibers exhibit no or negligible kill capability and are, therefore, not preferred for use with the immersion technique described herein.

[048] It was also surprisingly found that the capacity of fibers and fibrous media to absorb the antitoxin in concentrated solution, and to release the active agent upon contact with various microorganisms, differs depending on whether the media are formed from a meltblown process, or from a spunbond process.

[049] Preferred media for immersion in concentrated antitoxin solution in accordance with the inventive methods are spunbond media (that are devoid of hydrophobic coverings or coatings). Spunbond media have been found to efficiently absorb triiodide solution, to release active iodine upon contact with microorganisms, and are defined to be non-leachable as determined by the ASTM E2149-01 standard. Accordingly, when prepared according to the method of the present invention, spunbond polypropylene fibers of 30 gsm, for example, exhibit superior kill performance over a long period of time.

[050] In contrast, while meltblown fibers made from polypropylene of 30 gsm exhibited triiodide absorption when the meltblown media was immersed in triiodide solution, the fibers did not release active iodine and, therefore, did not exhibit the ability to kill or deactivate various microorganisms.

[051] It was noted by the inventors that the tested polypropylene meltblown fibers are characterized by a melt flow index (MFI) of at least 150, while the spunbond fibers of polypropylene are characterized by a low melt flow index (MFI) of about 34 MFI. While not wishing to be bound by any particular theory, it is hypothesized that the lower (of about 34 MFI) MFI fibers work well because they also have a large Molecular Weight (MW), which helps take up the iodine as well as stabilize it in the fiber. Conversely, higher MFI fibers are characterized by a low Molecular Weight that may inhibit efficient take up and stabilization of iodine, and may promote the transformation of active iodine to iodide, which is inactive and ineffective to kill micro organisms.

[052] Accordingly, in one embodiment, fibers and media formed therefrom of the present invention are characterized by a melt flow index (MFI) of at least 5 MFI. In another embodiment, the MFI is no more than 200 MFI, preferably 150 MFI. In yet another embodiment, the fibers are characterized by a melt flow index (MFI) of between about 50 and about 150 MFI. In still another embodiment, the fibers are characterized by a melt flow index (MFI) of between about 5 and about 50 MFI.

[053] The method of the present invention includes immersion of the selected fibrous media described above in a triiodide solution characterized by a high concentration of solid iodine, preferably of at least 1000 ppm. By itself, the addition of iodine to an aqueous solution can result in a maximum of only about 330 ppm. Because a higher concentration is desired to maximize the loading of the antimicrobial in the fibers, potassium iodide is preferably added and mixed with the iodine in the water or other solvent to form the triiodide solution. The potassium iodide assists in converting the diatomic iodine to triiodide ions, resulting in concentrations over the approximately 330 ppm that can be achieved by adding solid iodine alone. Concentrations of up to 5000 ppm, and higher, close to saturation levels, are achieved by mixing iodine and potassium iodide to permit the iodine concentration to rise to the desired levels.

[054] For example, a concentrated solution of about 129,600 ppm of iodine is achieved by adding potassium iodide. This solution is then diluted to the desired optimal levels.

[055] In one embodiment, a concentrated solution for immersion of the media of the present disclosure comprises between about 1500 and about 3500 ppm, or more preferably between about 2000 to about 3000 ppm, or to about 2500 ppm, for efficient molecular grafting of iodine to fibers. In various particular embodiments, curtain and face mask media are formed by immersion in a solution of between about 1500 and about 3500 ppm, or more preferably between about 2000 to about 3000 ppm, or to about 2500 ppm.

[056] In another embodiment, a concentrated solution for immersion of the media of the present disclosure comprises between about 3500 to about 6500 ppm, or more preferably between about 4500 to about 5500 ppm, or to about 5000 ppm, for efficient molecular grafting of iodine to fibers. In various particular embodiments, wound dressing media are formed by immersion in a

solution of between about 3500 to about 6500 ppm, or between about 4500 to about 5500 ppm, or to about 5000 ppm.

[057] In yet another embodiment, a concentrated solution for immersion of the media of the present disclosure comprises at least 400 ppm, preferably at least about 2500 ppm.

[058] In an additional embodiment, a concentrated solution for immersion of the media of the present disclosure comprises at least between about 1000 ppm to about 10,000, or preferably, between about 5,000 to 10,000 ppm.

[059] In still another embodiment, a concentrated solution for immersion of the media of the present disclosure comprises at least 25000 ppm for molecular grafting of iodine to fibers.

[060] In still another embodiment, a concentrated solution for immersion of the media of the present disclosure comprises between about 25000 and about 26000 ppm for molecular grafting of iodine to fibers.

[061] An example of the preparation of the concentrated triiodine solution before dilution for treating the fibrous media of the present invention is provided in Example 1 below.

[062] As one of skill in the art will appreciate, there are many different species of iodine. The inventors discovered that for the media formed in accordance with the methods of the present disclosure, at least I_2, 1_3^-, HOI, Γ, and IO_3^- are formed on the media after immersion in the concentrated antitoxin solution in a proportion where the iodine active is in a majority versus the other species. To test the media, the molecularly grafted material was cut into swatches of l"xl" and added to a 10 ml test tube of water. The sample was vortexed for 30 seconds before being analyzed on a spectrophotometer for each of the above species with a specific method for each. In this way, the amount in ppm of each species present could be determined. The inventors also observed that when air is passed through the media over a period of time, of 8 hours, for example, the media color changes from yellow to a light yellow. It was determined that the color change occurs as the species equilibrium shifts away from the I_2 towards the iodate and others, as the active iodine is released. When air is no longer flowing through the media, the yellow color comes back and the shift towards I_2 and less of the non active species like iodate and iodide.

[063] The immersion step can be performed by dipping or immersing the media in the antitoxin solution for a period of time sufficient to achieve the desired concentration. The time of immersion may be a few seconds up to a few minutes, depending on the material, the concentration of the antitoxin in solution, and the desired resultant concentration in the fabric. It will be clear to one of ordinary skill in the art that the desired concentration in the fibers can be obtained by either increasing the immersion time or the concentration of the antitoxin in solution, or with an optimal combination of these parameters, depending on manufacturing needs.

[064] FIGS. 1-8 show the testing of a material that can be used as a layer in a facemask, or other products, formed in accordance with the present invention and results therefrom for various microorganisms. A standard AATCC test method was applied to test the facemask antimicrobial properties under different conditions.

[065] A preferred embodiment of a facemask formed in accordance with the present disclosure includes at least one layer, preferably an outer layer, treated with triiodide solution in accordance with the molecular grafting methods described herein. The facemask also includes an inner layer for contacting the wearer's face. In a most preferred embodiment, the facemask also includes a middle filtration layer.

[066] FIG. 1 describes a testing method 10 of various swatches of antimicrobial media formed in accordance with the present disclosure. The antimicrobial-treated swatch is exposed to a microbial suspension for 15 minutes 12. The swatch is then placed in a neutralizing fluid to recover viable microorganisms (colonies) 14, which are then counted and recorded as colony forming units (CFU), in accordance with AATCC Test Method 100-2004 (AATCC 100 standard) 16.

[067] FIGS. 2-8 are tables of results of testing a media formed in accordance with the present disclosure, using the method 10 described in FIG. 1. FIGS. 2-8 describe the percent reduction in CFU when a sample of a non-woven spunbond material formed of polypropylene with an MFI of 30-40 MFI, treated by an immersion in a triiodide solution of 2500 ppm iodine, is contacted with various bacteria. The media can be used as an outer layer, or scrim, of a facemask, for example.

[068] The results of exposure to various microorganisms for different conditions are shown in FIG. 2-8. For example, FIG. 2 shows the percentage reduction of *P. Aeruginosa* challenge on a standard surgical mask (12.0325%) vs. freshly treated facemask swatch (99.9989%). FIG. 3 compares the result for a freshly treated facemask swatch with one which has been aged at 50°C for 47 days (2.0 yrs. at Room Temp.). As shown, the percentage reduction of *P. Aeruginosa* was 99.999613% so that there was no measurable degradation in performance. FIGS. 4-6 show similar outstanding results after 47 days for *S. aureus MRSA,* of *E.faecalis* VRE, and *K. pneumoniae* challenges, respectively.

[069] FIG. 7 describes AATCC 100 test results, using the method 10 of FIG. 1, after a simulated use test of 8 hours for both fresh samples and speed-aged for 54 days for various microorganisms. The samples were pre-conditioned using a simulated breathing machine designed to mimic the mechanics and physical conditions of human breathing, simulating airflows of 26 LPM that a mask would typically be subjected to when worn for its intended use for extended periods of time. Following the preconditioning, the bacterial challenges were administered. As demonstrated, long-term use (8 hours) under conditions of moderate physical activity has no impact on the efficacy of the antimicrobial outer scrim of the inventive facemask, even after these devices have been subjected to speed-aging at 50°C.

[070] In addition, the facemask antimicrobial-treated scrims were tested for toxicity. The values for conversion to total iodine intake are based on a sum of the iodine released from the mask, measured every fifteen minutes for a total of 8 hours, assuming a breathing rate of 1.6 m^3/hr. As shown in FIG. 8, iodine exposure levels were more than 1000 times lower than the set TLV (Threshold Limit Value) of 1.036 mg/m^3. The iodine measurement test method was based on the OSHA ID-212 standard protocol. In addition, Iodine exposure levels were found to be approximately 100 times lower than the set TUIL (Tolerable Upper Intake level) of 1100 mg per day.

[071] These tests showed that the treatment of the outer scrim of surgical masks with an iodine-based antimicrobial in accordance with the methods of the present invention provides a surgical facemask with strong antibacterial efficacy against *Pseudomonas aeruginosa, Staphylococcus aureus* MRSA, *Klebsiella pneumoniae,* and *E. faecalis* VRE after an exposure time of 15

minutes. Furthermore, stability testing performed under speed aging conditions indicated that the antimicrobial efficacy of the treated scrim material is maintained over time, even after an 8-hour usage period. Finally, the iodine immersion treatment raises no safety concerns in terms of exposure to ingested or inhaled iodine.

[072] In one embodiment, a privacy curtain, preferably a disposable curtain, suitable for replacement preferably every six months includes an antitoxic layer formed in accordance with the present disclosure. For example, the antitoxic layer can be a non-woven spunbond material formed of polypropylene with an MFI of 30-40 MFI, which was immersed in a triiodide solution of about 2500 ppm iodine.

[073] FIGS. 9-12 show the results of the same testing as done for FIGS. 3-6 for a non-woven spunbond material formed of polypropylene with an MFI of 30-40 MFI, treated by an immersion in a triiodide solution of 2500 ppm iodine, where the material can form a layer of a privacy curtain, or any other product of the present disclosure.

[074] FIG. 13 is a table of results of testing a privacy curtain formed in accordance with the present disclosure using the ASTME 2149 testing method.

[075] As described and shown in FIGS. 9-13, the treatment of hospital curtain fabrics with the molecular grafted triiodide method of the present invention resulted in strong antibacterial efficacy against *Pseudomonas aeruginosa, Staphylococcus aureus* MRSA, *Klebsiella pneumoniae,* and *E.faecalis* VRE after an exposure time of 15 minutes. Furthermore, stability testing performed under speed aging conditions indicated that the antimicrobial efficacy of the treated curtain material is maintained over time.

[076] FIG. 13 shows antimicrobial efficacy of the treated curtain as demonstrated by an alternative test method (ASTM E2149) with below detection levels of bacterial *(S. aureus, S. aureus MRSA, E. coli* and A *baumannii)* and yeast *(C. albicans)* challenges recovered within contact times as short as 5 minutes, under speed aging conditions representative of 76 days.

[077] Furthermore, testing has shown that the curtain fabrics treated with antitoxin solution in accordance with the molecular grafted triiodide method of the present invention showed no potential cytotoxic effects when tested *in vitro,* in accordance with ISO10993-5 guidelines. In

particular, an *in vitro* study was conducted to evaluate the antimicrobial curtain fabrics for potential cytotoxicity effects following the ISO 10993 guidelines. The treated curtain fabric was extracted in IX MEM (Minimum Essential Medium) at 37°C for 24 hours. The extract was then placed in contact with monolayers of fibroblasts and incubated for 48 hours. Monolayers were then examined for abnormal cell morphology and cellular degeneration. Extracts from the curtain fabric were tested and found to cause no evidence of cell lysis or toxicity (Grade 0). Accordingly, the curtain fabrics prepared in accordance with the present invention met the level of less than a Grade 2 required to meet the ISO 10993 guidelines.

[078] In a particular embodiment, an antimicrobial medical tape of the present disclosure includes a layer of a spunbond media, for example, having a MFI of less than 150 MFI, treated in accordance with the present method by immersion in a concentrated antitoxin solution comprising triiodide. Preferably, the concentration of triiodide is at least 2000 ppm. In one embodiment, the media is a 50/50 blend of (spunbond) polypropylene and alginate or synthetic cellulose acetate fibers.

[079] The medical tape preferably also includes an adhesive liner.

[080] The invention provides a novel method of making fibrous media or fabrics with antitoxic *(e.g.,* biocidal) properties. The fabrics can be either wovens or nonwovens. The antitoxic properties are imparted to the fabric by introducing an active agent, particularly an antimicrobial agent, to the fabric by immersion in a concentrated antitoxic solution. The fabrics produced in accordance with the present invention have widespread utility. For instance, they can be used as wound dressings, antimicrobial medical tape, gowns, drapes, air filters, protective clothing, shoe coverings, hair coverings, privacy curtains, facemasks, and wipes.

[081] Example 1 - preparation of triiodide solution

A. Prepare IN iodine (129,600ppm I_2):

1. In a 1L volumetric flask, add approx 250mL high purity water
2. Weigh out 175g of KI solid and add to the 1L flask containing the water
3. Swirl solution in flask to dissolve all KI.

4. Weigh out 130g of Iodine solid and add to the 1L flask containing the KI and water solution

5. Fill the volumetric flask with high purity water to the marked line

6. Add a magnetic stir bar to the flask

7. Cap the flask with a glass stopper

8. Cut a piece of parafilm and wrap it around the top of the flask/stopper to prevent any leakage

9. Take the flask and place it on a magnetic stir plate.

10. Set stir control to 7 and let the solution mix overnight to dissolve all iodine solid

11. Dilute according to needs

[082] It should be apparent to those skilled in the art that the described embodiments of the present invention provided herein are illustrative only and not limiting, having been presented by way of example only. As described herein, all features disclosed in this description may be replaced by alternative features serving the same or similar purpose, unless expressly stated otherwise. Therefore, numerous other embodiments of the modifications thereof are contemplated as falling within the scope of the present invention as defined herein and equivalents thereto.

What is Claimed is:

1. A process for producing an antitoxic fabric comprising:

   a. providing a fibrous media comprising a material having a melt flow index of less than 150 MFI ;

   b. forming a concentrated stable antitoxin solution comprising triiodide;

   c. fully immersing said media in said antitoxin solution to form a wet media;

   d. processing the wet media through rollers, thereby forcing the iodine to penetrate the media; and

   e. drying the wet media and isolating the fabric therefrom.

2. The process of claim 1, wherein the antitoxin solution further comprises an active agent selected from the group consisting of iodine, bromine, chlorine and hydrogen peroxide.

3. A wound dressing, surgical drape, privacy curtain, facemask, gown, article of protective clothing, shoe covering, hair covering, air filter, medical tape, or wipe comprising the antitoxic fabric formed according to the process of claim 1.

4. The process of claim 1, wherein the antitoxin solution comprises a concentration of at least 2000 ppm iodine.

5. The process of claim 1, wherein the antitoxin solution comprises a concentration of at least 2500 ppm iodine.

6. The process of claim 1, wherein the fibrous media is a 50/50 blend of polypropylene and synthetic cellulose acetate.

7. The process of claim 1, wherein the fibrous media is a 50/50 blend of polypropylene and alginate fibers.

8. An antitoxic layer comprising the antitoxic fabric formed according to the process of claim 1.

9. A wound dressing comprising the antitoxic fabric formed according to the process of claim 6.

10. The process of claim 8, wherein the fibrous media is a nonwoven spunbond media characterized by an MFI of 30-40.

11. The antitoxic layer of claim 8, wherein the fibrous media comprises polypropylene.

12. A surgical mask or privacy curtain comprising the antitoxic fabric formed according to the process of claim 1, wherein the fibrous media comprises polypropylene, and wherein the antitoxin solution has a concentration of at least 2500 ppm iodine.

13. The process of claim 1, wherein the antitoxic solution comprises a concentration of at least 25,000 ppm iodine.

14. A medical tape comprising the antitoxic fabric formed according to the process of claim 1, wherein the fibrous media is spunbond, the medical tape further comprising an adhesive liner.

FIG. 1

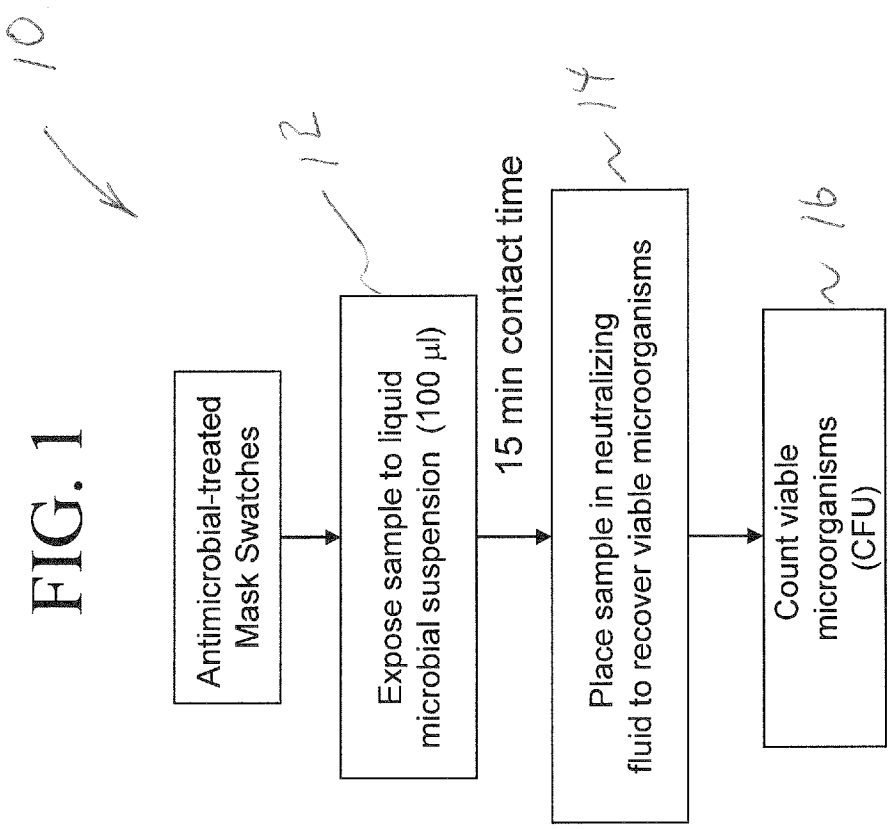
10
12
14
16
Antimicrobial-treated Mask Swatches
Expose sample to liquid microbial suspension (100 µl)
15 min contact time
Place sample in neutralizing fluid to recover viable microorganisms
Count viable microorganisms (CFU)

FIG. 2

Contact Time	% Reduction of *P. Aeruginosa* challenge on:	
	Standard Surgical Mask	TrioMed Active Surgical Mask
15 minutes	12.0325%	>99.9989%

FIG. 3

Contact Time	% Reduction of *P. aeruginosa* challenge on:	
	TrioMed Active Surgical Mask: Fresh	TrioMed Active Surgical Mask: Aged at 50°C for 47 days (2.0 yrs. at Room Temp.)
15 minutes	>99.998951%	>99.999613%

FIG. 4

Contact Time	% Reduction of *S. aureus MRSA* challenge on:	
	TrioMed Active Surgical Mask: Fresh	TrioMed Active Surgical Mask: Aged at 50°C for 47 days (2.0 yrs. at Room Temp.)
15 minutes	>99.999474%	>99.999474%

FIG. 5

Contact Time	% Reduction of *E. faecalis* VRE challenge on:	
	TrioMed Active Surgical Mask: Fresh	TrioMed Active Surgical Mask: Aged at 50°C for 47 days (2.0 yrs. at Room Temp.)
15 minutes	>99.998770%	>99.998770%

FIG. 6

Contact Time	% Reduction of *K. pneumoniae* challenge on:	
	TrioMed Active Surgical Mask: Fresh	TrioMed Active Surgical Mask: Aged at 50°C for 47 days (2.0 yrs. at Room Temp.)
15 minutes	>99.999681%	>99.999681%

FIG. 7

Bacterial Challenge (15 min contact time)	% Reduction of bacterial challenge after 8-hour simulated use on:	
	TrioMed Active Surgical Mask: Fresh	TrioMed Active Surgical Mask: Aged at 50°C for 54 days (2.3 yrs. at Room Temp.)
P. aeruginosa	99.9993%	99.9993%
S. aureus MRSA	>99.9978%	>99.9978%
E. faecalis VRE	>99.9988%	>99.9988%
K. pneumoniae	>99.9991%	>99.9991%

FIG. 8

Amount of Iodine released from the TrioMed Active Surgical Mask at a temperature of 35°C and 85% humidity level as determined using a Human Breathing Simulator at a flow rate of 26 LPM:

	Average Iodine measured in exhaled air (mg/m^3) §	**Conversion to Total Iodine Intake (micrograms)**
TrioMed Active Surgical Mask	0.0008	10.24*
Upper Exposure Limit	1.036 (TLV)	1100 (TUIL)

*Values are based on the sum of the iodine released from the TrioMed Active Surgical Mask, measured every 15 minutes for a total of 8 hours, assuming a breathing rate of 1.6 m^3/h.

§ Iodine Measurement Test Method based on the OSHA #ID-212 standard protocol.

FIG. 9

Contact Time	% Reduction of *P. aeruginosa* challenge on:	
	TrioMed Active Curtain Fabric: Fresh	TrioMed Active Curtain Fabric: Aged at 50°C for 47 days (2.0 yrs. at Room Temp.)
15 minutes	>99.998951%	>99.999613%

FIG. 10

Contact Time	% Reduction of *S. aureus MRSA* challenge on:	
	TrioMed Active Curtain Fabric: Fresh	TrioMed Active Curtain Fabric: Aged at 50°C for 47 days (2.0 yrs. at Room Temp.)
15 minutes	>99.999474%	>99.999474%

FIG. 11

Contact Time	% Reduction of *E. faecalis* VRE challenge on:	
	TrioMed Active Curtain Fabric: Fresh	TrioMed Active Curtain Fabric: Aged at 50°C for 47 days (2.0 yrs. at Room Temp.)
15 minutes	>99.998770%	>99.998770%

FIG. 12

Contact Time	% Reduction of *K. pneumoniae* challenge on:	
	TrioMed Active Curtain Fabric: Fresh	TrioMed Active Curtain Fabric: Aged at 50°C for 47 days (2.0 yrs. at Room Temp.)
15 minutes	>99.999681%	>99.999681%

FIG. 13

Contact Time	% Reduction of bacterial challenge on TrioMed Active Curtain Fabric: Aged at 50°C for 76 days (3.3 yrs. at Room Temp.)				
	S. aureus MRSA	*S. aureus*	*E. coli*	*A. baumannii*	*C. albicans*
5 minutes	99.9917%	>99.9966%	99.9977%	>99.9971%	>99.7143%
15 minutes	99.9861%	99.9993%	>99.9983%	>99.9971%	>99.7143%
60 minutes	99.9852%	>99.9966%	99.9983%	>99.9971%	>99.7143%

*Summary of ASTM2149 Test Method:

1- Place microbial challenge (10E+05 CFU/ml) in flask containing 250 ml PBS. Add 0.5 g of control or treated fabric.

2- Place in shaker incubator for specified contact time (5 min to 1 h)

3- Remove 1 ml from flask and plate using standard microbiological technique.

INTERNATIONAL SEARCH REPORT

International application No.
PCTYUS2012/068784

A. CLASSIFICATION OF SUBJECT MATTER

D06M ll/07(2006.01)i, D06M 11/50(2006.01)1, A61F 13/00(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)

D06M 11/07; A01N 25/34; A61F 13/00; A61K 9/50; A01P 1/00; D04H 1/00

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Korean utility models and applications for utility models

Japanese utility models and applications for utility models

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

eKOMPASS(KIPO internal) & Keywords: fabric, antitoxic, melt flow index, triiodide

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	WO 2011-103578 Al (SAFEL]FE/T[R]0SYN CORP.) 25 August 2011 See abstract , page 9 lines 14-23 , claims 1-6 .	1-14
A	US 7462753 B2 (MA, R. H. et al.) 9 December 2008 See abstract .	1-14
A	US 2008-0145437 Al (AMUNDSON, J. D. et al.) 19 June 2008 See abstract , paragraph [0187] .	1-14
A	US 6565866 B2 (GOTTLUND, K. L. et al.) 20 May 2003 See abstract , claim 1.	1-14
A	US 2009-0263439 Al (CASAS-SANCHEZ, J. et al.) 22 October 2009 See paragraphs [0013] , [0018] , claim 1.	1-14

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

* Special categories of cited documents:

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"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

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"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

"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
26 March 2013 (26.03.2013)	**28 March 2013 (28.03.2013)**
Name and mailing address of the ISA/KR Korean Intellectual Property Office 189 Cheongsa-ro, Seo-gu, Daejeon Metropolitan City, 302-70 1, Republic of Korea Facsimile No. 82-42-472-7140	Authorized officer JUNG, Soo Hwan Telephone No. 82-42-481-3580

Form PCT/ISA/210 (second sheet) (My 2009)

INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No.

PCT/US2012/068784

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