TEXTILES TREATED WITH HYPERBRANCHED POLYETHYLENEIMINE DERIVATIVES FOR ODOR CONTROL PROPERTIES

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Publication Classification

Publication Classification

Publication Classification

Publication Classification

(51) Int. Cl.
D06M 15/21 (2006.01)

(52) U.S. Cl. 252/8.61; 427/372.2

ABSTRACT

The present disclosure is directed to synthetic textiles treated with h-PEI derivatives, such derivatives possessing the general structure shown below:

\[(R)_{x} - h\text{-PEI} - (A)_{y}\]

where \(R\) is a non-hyperbranched hydrocarbon group, said hydrocarbon group having at least one linear portion, said linear portion having between 5 and 30 carbon atoms; where \(x\) is a number from 1 to 10,000; where h-PEI is a hyperbranched polyethylenimine; where \(A\) is an organic compound having from 1 to 4 carbon atoms; where \(y\) is a number from 0 to 500; and wherein \(R\) is present in an amount of between about 0.1% and about 80% by weight of the h-PEI derivative. The chemical treatment imparts durable odor control, softness, wrinkle resistance, and moisture wicking to substrates treated therewith.
TEXTILES TREATED WITH HYPERBRANCHED POLYETHYLENEIMINE DERIVATIVES FOR ODOR CONTROL PROPERTIES

TECHNICAL FIELD

[0001] The present disclosure is directed to the field of hyperbranched polyethyleneimine derivatives useful for treating textile substrates. More specifically, this disclosure relates to treatments used to provide wash-durable odor control in textiles, particularly in synthetic or synthetic-containing textile substrates. The chemical treatment also reduces wrinkling and imparts softness and moisture wicking to the treated textile.

[0002] The present disclosure is directed to a molecule having a hyperbranched polyethyleneimine core to which is attached, at a minimum, one or more hydrocarbon groups. Optionally, linking compounds may connect the hydrocarbon groups to the hyperbranched polyethyleneimine core. Additionally, other organic compounds may also be used to “cap” the branches of the hyperbranched polyethyleneimine that are unreacted with hydrocarbon groups.

[0003] In one embodiment, the hydrocarbon groups comprise up to 20% of the weight of the hyperbranched polyethyleneimine derivative. In such embodiment, the resulting treated textile exhibits wicking properties that are desired for textiles used in apparel and other applications. To produce a treated textile whose finish is capable of withstanding multiple launderings, it is preferable to use a cross-linking agent or compound to secure the hyperbranched polyethyleneimine derivative to the textile surface.

[0004] In a second embodiment, the hydrocarbon groups comprise up to 20% and about 80% of the weight of the hyperbranched polyethyleneimine derivative. In such embodiment, it has been found that durability may be achieved without the use of a separate cross-linking agent or compound, although one may be incorporated if so desired for certain applications. Textiles treated with derivatives having a greater amount of hydrocarbon groups tend to exhibit finishes that are more water-repellent, which may be useful in some circumstances.

BACKGROUND

[0005] It has long been desirable to produce a textile substrate having durable odor adsorption capabilities. In particular, the abatement of human sweat odors is useful in a variety of different applications. For instance, hunters are interested in preventing their body odors from reaching animals being pursued. In perhaps a more common application, apparel that may be worn several times before requiring laundering would provide considerable benefits to users thereof.

[0006] Manufacturers have used a variety of approaches to solve the problem of odor abatement. A first approach is the treatment of the textile article with antimicrobial compounds or the incorporation of antimicrobial compounds into the yarns used to make the substrate. Antimicrobial-treated textiles function to reduce odors by controlling or preventing the growth of microorganisms. When microorganisms grow, they degrade materials into volatile organic compounds, which are often malodorous. While preventing the growth of microorganisms, approaches using antimicrobials fail to address the issue of odor control once volatile organic compounds are present in the textile.

[0007] Another approach to the problem of odor control is to incorporate carbon black particles, granules, fibers, or cloth into a textile. Depending on the chosen pore size and source material, carbon black is generally effective at absorbing odors when dry, but it tends to lose some of its efficacy when wet (as the surface area becomes blocked by water or other aqueous contaminants). A second problem with carbon black is that it must be bound to the textile by adhesives or other binders, often reducing the breathability of the treated textile. Finally, carbon black imparts a black color to the textile surface being treated, which is unsuitable in many situations (for example, with light-colored apparel).

[0008] Other particles, such as zeolites, have also been used for textile odor reduction. Many of these exhibit the same problems associated with carbon black, while being functionally less effective.

[0009] More recently, manufacturers have used cyclodextrins and cycloextrin derivatives in an effort to produce odor-reducing textiles. However, as will be demonstrated herein, cyclodextrins are not particularly efficacious in reducing odors associated with human sweat, nor are they particularly durable to repeated launderings.

[0010] Thus, a need exists for a wash-durable odor abatement chemistry that preferably imparts benefits such as wrinkle resistance, moisture wicking, and softness. The present treatment provides a solution to such needs providing a specific hyperbranched polyethyleneimine structure, which is linked to one or more hydrocarbon groups having linear portions of between 5 and 30 carbon atoms and which may additionally be linked to one or more organic “capping” compounds.

SUMMARY

[0011] This disclosure is directed to treatments for synthetic textile goods that impart odor control properties to the treated textiles. Additionally, the chemical treatment provides benefits in terms of reduced drying time, reduced wrinkling, and, in some circumstances, improved moisture wicking.

[0012] The present treatment comprises a modified hyperbranched polyethyleneimine compound, also referred to herein as a “hyperbranched polyethyleneimine derivative.” The hyperbranched polyethyleneimine derivative comprises at least one hyperbranched polyethyleneimine (a “hydrophobic component”) that has been linked to one or more hydrocarbon groups having between 5 and 30 carbon atoms linearly arranged (“hydrophobic component(s)”, which may be linked to the hyperbranched polyethyleneimine using any of a number of different linkages). In another embodiment, the chemical treatment also comprises one or more additional organic “cap” compounds attached to the hyperbranched polyethyleneimine core.

[0013] Preferably, the hydrophobic components (that is, hydrocarbon groups and optional linking compounds) are electrophilic, so that they react with the nucleophilic hyperbranched polyethyleneimine molecule. Any of a number of acceptable linking groups may be used to link the hydrocarbon groups to the hyperbranched polyethyleneimine, as will be described herein, or the hydrocarbon groups may link directly to the hyperbranched polyethyleneimine molecule.

[0014] In one embodiment, the hydrocarbon groups comprise up to 20% of the weight of the hyperbranched polyethyleneimine derivative. In such embodiment, the resulting treated textile exhibits wicking properties that are desired for textiles used in apparel and other applications.
treated textile whose finish is capable of withstanding multiple launderings, it is preferable to use a cross-linking agent or compound to secure the hyperbranched polyethyleneimine derivative to the textile surface.

[0015] In a second embodiment, the hydrocarbon groups comprise up about 20% and about 80% of the weight of the hyperbranched polyethyleneimine derivative. In such embodiment, it has been found that durability may be achieved without the use of a separate cross-linking agent or compound, although one may be incorporated if so desired for certain applications. Textiles treated with derivatives having a greater amount of hydrocarbon groups tend to exhibit finishes that are more water-repellent, which may be useful in some circumstances.

[0016] The disclosure is further directed to the process for treating textiles with the present chemical treatment, wherein the chemical treatment is applied to at least a portion of the fiber, yarn, textile, or composite. In a presently preferred embodiment, the target fabric is placed into the chemical treatment (e.g., by dipping), then padded and dried in a single continuous process.

[0017] This disclosure is further directed to the fibers, yarns, fabrics, textiles, finished goods, or nonwovens (encompassed herein under the terms “textiles” and “webs”) treated with the subject hyperbranched polyethyleneimine derivatives. Such textiles and webs exhibit greatly improved odor control properties, even after multiple launderings. Further, such treated textiles also exhibit enhanced moisture wicking and reduced wrinkling and drying time.

DETAILED DESCRIPTION

[0018] The present chemical treatment is especially well-suited for use with synthetic substrates. The term “synthetic” refers to any man-made fiber type, including, without limitation, polyester, polyamide (e.g., nylon), acrylic, polyethylene, polypropylene, aramids (e.g., NOMEX® and KEVLAR®) and the like. Preferably, the substrate comprises a majority of synthetic content and may include a combination of synthetic fiber types or a combination of synthetic and natural fiber types.

[0019] Of principal utility in the present chemical treatment is the hyperbranched polyethyleneimine (h-PEI) molecule, which may also be referred to herein as the hydrophobic component of the compound. In schematic terms, the h-PEI molecule can be described as having a central core surrounded by a plurality of molecular branches, with each branch projecting outward from the core and having a highly reactive end group. It is to be expected that partial linkage of the branches to themselves often occurs. The molecule typically exhibits a very high charge density per area, meaning that there are a high number of positive charges clustered densely together around the molecular core. This configuration makes the molecule very capable of interacting with a wide range of other molecules, many of which will be described herein. The number of molecules that may be attached to the h-PEI molecule depends on the number average molecular weight \( M_n \) of the h-PEI, which reflects the number of branches available for attachment.

[0020] For the applications that are contemplated herein, hyperbranched polyethyleneimines having a number average molecular weight \( M_n \) of between about 300 and about 2 million are preferred, with \( M_n \) of between about 1,000 and about 75,000 being more preferred.

[0021] To the h-PEI molecule are attached at least one, and preferably more than one, hydrocarbon groups to increase the hydrophobicity of the resulting compound (i.e., the h-PEI derivative). These hydrocarbon groups, together with any linking compounds which may be used to attach them to the h-PEI molecule, are collectively referred to as the “hydrophobic components” of the dye-reactive molecule. These hydrocarbon groups may be linear molecules or may contain branched or aromatic portions, which have an electrophilic group capable of reacting with the nucleophilic h-PEI. Preferably, regardless of the structure of the hydrocarbon, the linear portion of the hydrocarbon group contains between about 5 and about 30 carbon atoms and, more preferably, between about 4 and about 24 atoms. Mixtures of various length hydrocarbons may also be used.

[0022] Examples of electrophilic hydrocarbons include, without limitation, carboxylic acids, ketone dimers, formates, acetyl halides (such as acetyl chloride), esters, anhydrides, alkyl halides, epoxides, isocyanates, and the like. Preferred examples include stearic acid, hydroxy stearic acid, isostearic acid, and palmitic acid.

[0023] In a first embodiment, the weight ratio of h-PEI to hydrophobic groups is from about 100:1 to about 5:1 and, more preferably, is from about 100:1 to about 10:1. In a second embodiment, the weight ratio of h-PEI to hydrophobic groups is from about 5:1 to about 1:10 and, more preferably, is from about 2:1 to about 1:5, depending on the \( M_n \) of the h-PEI. Most preferably, weight ratios of h-PEI to hydrophobic group from about 1:1 to about 1:4 are used.

[0024] The present h-PEI derivatives possess the structure shown below:

\[
\text{R}_x \text{-h-PEI-(A)_y}
\]

where \( R \) is a non-hyperbranched hydrocarbon group (for example, such as alkyl, alkenyl, arylalkyl, and arylalkenyl groups, where the number of carbon atoms in the linear portion of the hydrocarbon is between 5 and 30 carbon atoms), \( x \) is a number from 1 to about 10,000 (depending on the \( M_n \) of the h-PEI), where h-PEI is a hyperbranched polyethyleneimine, where \( A \) is a small organic “capping” compound, where \( y \) is a number from 0 to 500, and wherein \( R \) is present in an approximate amount of between about 0.1% and about 80% by weight of the molecule.

[0025] It is understood that, in the synthesis of molecules such as those fitting the general structure provided above, the actual product exhibits a polydispersity (a distribution of ratios) and the molar ratio of each molecule will vary somewhat around the target ratio.

[0026] Optionally, small organic molecules (generically shown as “A”) in the structure above may be used to “cap” the unreacted branches of the hyperbranched polyethyleneimine. Generally speaking, the capping molecule “A” has from one to four carbon atoms. Any caps that will react with the amine (NH or NH₂) portion of the h-PEI molecule may be used, including, without limitation, epoxides, anhydrides, esters, acids, carbonates, sulfates, formates, isocyanates, and mixtures thereof. Specific examples of such caps include, without limitation, ethylene oxide, propylene oxide, methyl bromide, acetic acid, vinyl sulfonates, trihydroxyacetic acid, and succinic anhydride. Mixtures of different “cap” molecules may be used.

[0027] Ethylene oxide (EO) or propylene oxide (PO) chains are especially useful as capping compounds in the present treatment to prevent the treated substrate from yel-
lowing when exposed to high manufacturing temperatures (for example, temperatures greater than 350° F). The addition of such chains is not required to achieve odor control and other benefits of the present treatment, but merely to impart additional benefits. For example, it has been found that the addition of EO or PO chains within the h-PEI derivative improves moisture wicking and produces softness in the treated substrate.

[0028] One potentially preferred “R” group has a C_{10}H_{22} structure, which in the above structure forms a stearic amide. When using this R group and an h-PEI having an M_n of 1200, representative molar ratios of h-PEI to R are 1:2, 1:4, 1:6, 1:8, 1:10, and 1:12. Similarly, when the M_n of the h-PEI molecule is about 10,000, representative molar ratios of h-PEI to R are 1:100, 1:100, 1:160, 1:80, and 1:40, respectively. Where the M_n of the h-PEI molecule is about 75,000, representative molar ratios of h-PEI to R are 1:400, 1:500, and 1:600.

[0029] The chemical synthesis of the present treatment molecules is conducted by reacting the h-PEI molecule with a hydrophobic R-containing electrophilic molecule in the presence of nitrogen. It has been found that mechanical agita-
tion of the reagents in a vessel under nitrogen at a temperature of about 150° C. produces the h-PEI derivatives described herein. The time necessary to complete the reaction depends on the amount of reagents that are being reacted and the size of the reaction vessel. The resulting compounds, referred to herein as “h-PEI derivatives”, are typically in the form of an oily liquid or waxy solid.

[0030] In one preferred embodiment, h-PEI derivatives have hydrophobic agents present in an amount of at least 0.1% to about 20% of the weight of the h-PEI derivative, and more preferably, from about 2% to about 15% of the weight of the h-PEI derivative. In an alternate embodiment, h-PEI derivatives have hydrophobic agents present in an amount of at least 20% to about 80% of the weight of the h-PEI derivative, and more preferably, from about 30% to about 75% of the weight of the h-PEI derivative.

[0031] To prepare a treatment bath for textiles using the h-PEI derivatives described herein, one approach is to heat the h-PEI derivative to its melting point, so that it may be poured into a vessel where it is combined, via high speed and high shear agitation, with hot water. In this instance, the phrase “hot water” refers to water having a temperature equal to or greater than the melting point of the h-PEI derivative. Suitable equipment for achieving high speed and high shear agitation includes propeller-type mixers, Jago®-type agitators, homogenizers, roll mill, ball mill, microfluidization, and the like.

[0032] The dispersion that results from the forcible introduction of the h-PEI derivative into water may be assisted and stabilized by an additional surfactant (e.g., a surfactant or a surfactating agent), the amount of which depends on the molecular weight of the h-PEI and the molar ratio of h-PEI to hydrophobic components. Acetic acid is one potentially preferred acid for this purpose (excess acid being evaporated off during subsequent drying of the treated textile substrate). Amounts of greater than 0.1% acid, by weight of solution, may be used successfully. Preferably, the amount of acid will be in the range of about 0.1% to about 50% of the weight of the h-PEI derivative.

[0033] The fiber, the yarn, the fabric, or the finished garment may be dyed using conventional processing, after which it is exposed (by methods known in the art such as by soaking, spraying, dipping, padding, foaming, exhausting, and the like) to the aqueous dispersion of the treatment chemistry. In some instances, it may be possible to introduce the treatment chemistry onto a garment by replacing the softening chemistry (normally applied during the laundering process) with the treatment described herein. Alternatively, the treatment chem-

[0034] The treated web is then removed from the solution and dried, preferably at temperatures between room temperature and 400° F, and more preferably, at temperatures between about 100° F. to about 380° F. The typical add-on weight of the h-PEI derivative is from about 0.1% of the weight of the fabric to about 10% of the weight of the fabric and, preferably, is from about 0.2% of the weight of the fabric to about 5% of the weight of the fabric.

[0035] The treated substrates exhibit durable odor control, even after multiple launderings. Additionally, and surprisingly, the substrates also have durable softness, reduced wrin-

[0036] Without wishing to be bound by theory, it is hypo-
thesized that the h-PEI derivatives disclosed herein possess a molecular configuration that facilitates odor absorption. Spe-
cifically, the h-PEI derivatives have a hydrophilic core sur-
rounded by a hydrophobic “shell” that is formed by the plu-
rality of hydrocarbon groups attached to the core h-PEI mole-
cule. Such a configuration results in numerous voids within the derivative molecule, in which volatile odor mol-
ecules having different polarities may be trapped. Addition-
ally, secondary interactions—such as, for example, Van der Waals forces, hydrogen bonding, and ionic interactions—may also contribute to the odor-trapping ability exhibited by textiles treated with the present derivatives.

[0037] In one embodiment (particularly when the hydro-
carbon groups are present in an amount of between 0.1% and 20% of the weight of the h-PEI derivative), a separate cross-linking agent is incorporated into the aqueous solution or dispersion to enhance treatment durability. Suitable cross-
linking agents for this purpose include epoxides, chlorotrimethylenes and their derivatives, azetidines, blocked isocyanates, and melamine derivatives, which may further enhance the durability of the treatment chemistry. When used, the cross-
linking agent is preferably present in an amount of between about 0.05% and about 5% of the weight of the treated textile. Preferably, the ratio of h-PEI derivative to cross-linking agent is from about 1:0.1 to 1:1.

[0038] Other finishing agents may also be used, such as wetting agents, softeners, soil release agents, flame retardants, and the like.

[0039] In order to further illustrate the present derivatives and advantages thereof, the following specific examples are given, it being understood that the same are intended only as illustrative and are in no way limiting.

**COMPARATIVE EXAMPLE A**

[0040] A 100% polyester knit fabric was used as the subs-
strate for Comparative Example A. A treatment solution was created, which contained 7.5% (by weight) of a hydroxypro-
pyl beta-cyclodextrin (available from Wacker Chemical un-
der the tradename CAVATEX® W7 HPTL) and 2.0% (by weight) of a blocked isocyanate cross-linking agent (available from Clariant Corporation under the tradename ARKO-
PHOB® DAN). The knit fabric was dipped into the treatment solution and padded at a pressure of 40 p.s.i. to remove excess
treatment solution. The treated fabric was then dried at about 370°F for about 3 minutes (until dry).

**COMPARATIVE EXAMPLE B**

[0041] A woven fabric containing 52% nylon by weight and 48% cotton by weight was used as the substrate for Comparative Example B. For this Comparative Example, the fabric was dipped into a bath containing only water and then padded at a pressure of about 40 p.s.i. to remove excess water. The fabric was then dried at about 310°F for about 10 minutes (until dry).

**COMPARATIVE EXAMPLE C**

[0042] The fabric from Comparative Example B was used in Comparative Example C. For this Comparative Example, the fabric was dipped into a bath that contained 7.5% (by weight) of a hydroxypropyl beta-cyclodextrin (available from Wacker Chemical under the tradename CAVATEX® W7 HPTL) and 2.0% (by weight) of a blocked isocyanate cross-linking agent (available from Clariant Corporation under the tradename ARKOPHOB® DAN) and then padded at a pressure of about 40 p.s.i. to remove excess. The treatment solution used in Comparative Example C was the same as that used in Comparative Example A. The fabric was dried at about 310°F for about 10 minutes (until dry).

**COMPARATIVE EXAMPLE D**

[0043] The fabric from Comparative Example B was used in Comparative Example D. For this Comparative Example, the fabric was dipped into a bath that contained about 5.0% (by weight) of a melamine-based retentive material (available from Ciba Specialty Chemical under the tradename PHOBO-TEX® JVA) and 2.0% (by weight) of a blocked isocyanate cross-linking agent (available from Clariant Corporation under the tradename ARKOPHOB® DAN). The fabric was then padded at a pressure of about 40 p.s.i. to remove excess, after which the fabric was dried at about 310°F for about 10 minutes (until dry).

**COMPARATIVE EXAMPLE E**

[0044] A woven fabric containing 52% nylon by weight and 48% cotton by weight was used as the substrate for Comparative Example E. The fabric was dipped into a bath that contained about 6.0% (by weight) of a hydroxypropyl beta-cyclodextrin (available from Wacker Chemical under the tradename CAVATEX® W7 HPTL) and 1.0% (by weight) of a blocked isocyanate cross-linking agent (available from Clariant Corporation under the tradename ARKOPHOB® DAN) and then padded at a pressure of about 40 p.s.i. to remove excess. The fabric was then dried at about 330°F for about 4 minutes (until dry).

**EXAMPLE 1**

[0045] To a round-bottom flask with a mechanical agitator were added 200.0 grams of hyperbranched polyethyleneimine (sold under the name EPOMIN® SP012 by Summit Specialty Chemical, New Jersey) and 94.83 grams of steric acid (sold by Aldrich, Wis.). The hyperbranched polyethyleneimine had a Mw of about 1200. The mixture was heated under nitrogen, with agitation, at a temperature of about 150°C for about 3 hours. At the end of the 3 hours, an aliquot was removed and analyzed using FT-IR, which indicated that no acid remained and that the reaction was complete. The resulting product was a waxy solid.

[0046] The h-PEI derivative was dispersed into hot water via high speed and high shear agitation. To solubilize the h-PEI derivative, acetic acid was added to the dispersions to achieve a pH level of about 5. The h-PEI derivative comprised about 3.0% by weight of the dispersion. Also added to the dispersion was 2.0% by weight of a blocked isocyanate cross-linking agent (available from Clariant Corporation under the tradename ARKOPHOB® DAN).

[0047] The 100% polyester knit fabric used in Comparative Example A was dipped into the dispersion and padded at a pressure of about 40 p.s.i. to remove excess. The fabric was then dried at a temperature of about 370°F for about 3 minutes (until dry).

**EXAMPLE 2**

[0048] To a round-bottom flask with a mechanical agitator were added 100.0 grams of hyperbranched polyethyleneimine (sold under the name EPOMIN® SP200 by Summit Specialty Chemical, New Jersey) and 170.7 grams of isostearic acid (sold under the name PRISORINE® by Uniqema, Del.). The hyperbranched polyethyleneimine had a Mn of about 10,000. The mixture was heated under nitrogen, with agitation, at a temperature of about 150°C for about 3 hours. At the end of the 3 hours, an aliquot was removed and analyzed using FT-IR, which indicated that no acid remained and that the reaction was complete. The resulting product was a viscous liquid.

[0049] The h-PEI derivative was dispersed into hot water via high speed and high shear agitation. To solubilize the h-PEI derivative, acetic acid was added to the dispersions to achieve a pH level of about 5. The h-PEI derivative comprised about 3.0% by weight of the dispersion. Also added to the dispersion was 2.0% by weight of a blocked isocyanate cross-linking agent (available from Clariant Corporation under the tradename ARKOPHOB® DAN).

[0050] The 100% polyester knit fabric used in Comparative Example A was dipped into the dispersion and padded at a pressure of about 40 p.s.i. to remove excess. The fabric was then dried at a temperature of about 370°F for about 3 minutes (until dry).

**EXAMPLE 3**

[0051] To a round-bottom flask with a mechanical agitator were added 100.0 grams of hyperbranched polyethyleneimine (sold under the name LUPASOL® WF by BASF, New Jersey) and 284.5 grams of stearic acid (sold by Aldrich, Wis.). The hyperbranched polyethyleneimine had a Mn of about 10,000. The mixture was heated under nitrogen, with agitation, at a temperature of about 150°C for about 3 hours. At the end of the 3 hours, an aliquot was removed and analyzed using FT-IR, which indicated that no acid remained and that the reaction was complete. The resulting product was a waxy solid.

[0052] To a stainless steel reactor with agitator and temperature controller were added 300.0 grams of the h-PEI derivative formed above. The h-PEI derivative was heated to about 250°F, after which 42.0 grams of ethylene oxide slowly were added until all of the ethylene oxide was reacted, as measured by the hydroxyl number. The resulting capped h-PEI derivative was a paste at room temperature.
[0053] The capped h-PEI derivative was dispersed into hot water via high speed and high shear agitation. To solubilize the h-PEI derivative, acetic acid was added to the dispersions to achieve a pH level of about 5. The h-PEI derivative comprised about 5.7% by weight of the dispersion. Also added to the dispersion was about 2.0% by weight of a blocked isocyanate cross-linking agent (available from Clariant Corporation under the tradename ARKOPHOB® DAN).

[0054] The 100% polyester knit fabric used in Comparative Example A was dipped into the dispersion and padded at a pressure of about 40 p.s.i. to remove excess. The fabric was then dried at a temperature of about 370°F for about 3 minutes (until dry).

EXAMPLE 4

[0055] To a round-bottom flask with a mechanical agitator were added 200.0 grams of hyperbranched polyethyleneimine (sold under the name EPOMIN® SP012 by Summit Specialty Chemical, New Jersey) and 94.83 grams of stearic acid (sold by Aldrich, Wis.). The hyperbranched polyethyleneimine had a Mₘ of 1200. The mixture was heated under nitrogen, with agitation, at a temperature of about 150°C for about 3 hours. At the end of the 3 hours, an aliquot was removed and analyzed using FT-IR, which indicated that no acid remained and that the reaction was complete. The resulting product was a waxy solid.

[0056] The h-PEI derivative was dispersed into hot water via high speed and high shear agitation. To solubilize the h-PEI derivative, acetic acid was added to the dispersions to achieve a pH level of about 5. The h-PEI derivative comprised about 3.0% by weight of the dispersion. Also added to the dispersion was about 2.0% by weight of a blocked isocyanate cross-linking agent (available from Clariant Corporation under the tradename ARKOPHOB® DAN). This is the same treatment chemistry that was used in Example 1.

[0057] The nylon/cotton woven fabric used in Comparative Examples B-D was dipped into the dispersion and padded at a pressure of about 40 p.s.i. to remove excess. The fabric was then dried at a temperature of about 310°F for about 10 minutes (until dry).

EXAMPLE 5

[0058] To a round-bottom flask with a mechanical agitator were added 100.0 grams of hyperbranched polyethyleneimine (sold under the name EPOMIN® SP200 by Summit Specialty Chemical, New Jersey) and 24.61 grams of stearic acid (sold by Aldrich, Wis.). The hyperbranched polyethyleneimine had a Mₘ of 10,000. The mixture was heated under nitrogen, with agitation, at a temperature of about 150°C for about 3 hours. At the end of the 3 hours, an aliquot was removed and analyzed using FT-IR, which indicated that no acid remained and that the reaction was complete. The resulting product was a viscous liquid.

[0059] The h-PEI derivative was dispersed into hot water via high speed and high shear agitation. To solubilize the h-PEI derivative, acetic acid was added to the dispersions to achieve a pH level of about 5. The h-PEI derivative comprised about 3.0% by weight of the dispersion. Also added to the dispersion was about 2.0% by weight of a blocked isocyanate cross-linking agent (available from Clariant Corporation under the tradename ARKOPHOB® DAN).

[0060] The nylon/cotton woven fabric used in Comparative Examples B-D was dipped into the dispersion and padded at a pressure of about 40 p.s.i. to remove excess. The fabric was then dried at a temperature of about 310°F for about 10 minutes (until dry).

EXAMPLE 6

[0061] To a round-bottom flask with a mechanical agitator were added 100.0 grams of hyperbranched polyethyleneimine (sold under the name LUPASOL® WF by BASF, New Jersey) and 14.23 grams of stearic acid (sold by Aldrich, Wis.). The hyperbranched polyethyleneimine had a Mₘ of 10,000. The mixture was heated under nitrogen, with agitation, at a temperature of about 150°C for about 3 hours. At the end of the 3 hours, an aliquot was removed and analyzed using FT-IR, which indicated that no acid remained and that the reaction was complete. The resulting product was a viscous liquid.

[0062] The h-PEI derivative was dispersed into hot water via high speed and high shear agitation. To solubilize the h-PEI derivative, acetic acid was added to the dispersions to achieve a pH level of about 5. The h-PEI derivative comprised about 2.3% by weight of the dispersion. No cross-linking agent was included with this formulation.

[0063] The nylon/cotton woven fabric used in Comparative Example E was dipped into the dispersion and padded at a pressure of about 40 p.s.i. to remove excess. The fabric was then dried at a temperature of about 330°F for about 4 minutes (until dry).

EXAMPLE 7

[0064] To a round-bottom flask with a mechanical agitator were added 100.0 grams of hyperbranched polyethyleneimine (sold under the name LUPASOL® WF by BASF, New Jersey) and 14.23 grams of stearic acid (sold by Aldrich, Wis.). The hyperbranched polyethyleneimine had a Mₘ of 10,000. The mixture was heated under nitrogen, with agitation, at a temperature of about 150°C for about 3 hours. At the end of the 3 hours, an aliquot was removed and analyzed using FT-IR, which indicated that no acid remained and that the reaction was complete. The resulting product was a viscous liquid.

[0065] The h-PEI derivative was dispersed into hot water via high speed and high shear agitation. To solubilize the h-PEI derivative, acetic acid was added to the dispersions to achieve a pH level of about 5. The h-PEI derivative comprised about 2.3% by weight of the dispersion. Also added to the dispersion was about 1.5% by weight of a blocked isocyanate cross-linking agent (available from Clariant Corporation under the tradename ARKOPHOB® DAN).

[0066] The nylon/cotton woven fabric used in Comparative Example E was dipped into the dispersion and padded at a pressure of about 40 p.s.i. to remove excess. The fabric was then dried at a temperature of about 330°F for about 4 minutes (until dry).

EXAMPLE 8

[0067] To a round-bottom flask with a mechanical agitator were added 100.0 grams of hyperbranched polyethyleneimine (sold under the name LUPASOL® WF by BASF, New Jersey) and 14.23 grams of stearic acid (sold by Aldrich, Wis.). The hyperbranched polyethyleneimine had a Mₘ of 10,000. The mixture was heated under nitrogen, with agitation, at a temperature of about 150°C for about 3 hours. At the end of the 3 hours, an aliquot was removed and analyzed
using FT-IR, which indicated that no acid remained and that the reaction was complete. The resulting product was a viscous liquid.

[0068] The h-PEI derivative was dispersed into hot water via high speed and high shear agitation. To solubilize the h-PEI derivative, acetic acid was added to the dispersion to achieve a pH level of about 4. The h-PEI derivative comprised about 2.3% by weight of the dispersion. Also added to the dispersion was about 1.5% by weight of a melanine-based cross-linking agent (available from Cytec Industries under the tradename CYMEL® 385).

[0069] The nylon/cotton woven fabric used in Comparative Example E was dipped into the dispersion and padded at a pressure of about 40 p.s.i. to remove excess. The fabric was then dried at a temperature of about 35° C., for about 4 minutes (until dry).

Odor Abatement Testing

[0070] A fabric sample (typically a 2-inch by 2-inch square) was positioned inside a glass vial having an internal volume of about 20 ml. and having a silicone cap. 1 microliter of an odor molecule mixture was injected into the vial. The vial was held at a temperature of about 40 C. for about 1 hour. The chemical composition of the void space at the top of the vial (that is, the "headspace") was evaluated using samples drawn from the vial and analyzed using a GC/MS. The numbers shown in the TABLES below are relative within each individual table for a given volatile compound. The data are reduced representations of the areas obtained directly from the GC peaks for a given compound. Higher numerical values reflect greater amounts of a particular volatile organic compound in the vapor phase. Lower numerical values reflect fabric samples having greater adsorption of odor-causing compounds.

[0071] For each of the Comparative Examples and the Example fabrics, head space analysis was conducted to determine the presence of three different odor-related molecules (isoamyl alcohol, isovaleric acid, and limonene), which had been added to the vials in approximately equal amounts by volume. Fabrics were evaluated after initial preparation (that is, after 0 washes) and after multiple washes had occurred (for example, after 5 washes). The term "wash" refers to laundering in a standard washing machine, using TIDE® powdered detergent, according to AATCC Method 130 and subsequent drying in a hot air dryer.

**TABLE 1**

<table>
<thead>
<tr>
<th>Sample ID</th>
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<th>5 washes</th>
<th>10 washes</th>
<th>20 washes</th>
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<td></td>
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<td></td>
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<td></td>
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<td>Example 3</td>
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**TABLE 1-continued**

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</thead>
<tbody>
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<td></td>
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**TABLE 2**

<table>
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</thead>
<tbody>
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<tr>
<td>Comp. Ex. B</td>
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<td>936</td>
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<td>Comp. Ex. C</td>
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<td>Comp. Ex. D</td>
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<td>606</td>
<td>868</td>
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<td>0</td>
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<td>Example 5</td>
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<td>58</td>
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<td>Comp. Ex. C</td>
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</tr>
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<td>Comp. Ex. D</td>
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<td>316</td>
<td>316</td>
</tr>
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<td>Example 4</td>
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<td>Example 5</td>
<td>191</td>
<td>243</td>
<td>313</td>
</tr>
<tr>
<td><strong>VOLATILE COMPOUND: LIMONENE</strong></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Comp. Ex. B</td>
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<tr>
<td>Comp. Ex. C</td>
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<td>8115</td>
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<td>Comp. Ex. D</td>
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[0072] As shown from the test data above, the fabrics of Examples 1, 2, and 3 adsorbed more volatile compounds from the headspace of their respective vials than did Comparative Example A. The treatment chemistry was particularly effective at adsorbing isovaleric acid, which is a primary component of human sweat odors. The treatment chemistry also worked exceptionally well at adsorbing isobutyraldehyde, and performed better than the Comparative Example at adsorbing limonene. It should also be noted that the superior performance of Examples 1-3 was especially evident after the samples had been subjected to multiple launderings.

[0073] As shown from the test data above, the fabrics of Examples 4 and 5 performed better than, or equivalent to, Comparative Examples B, C, and D at adsorbing volatile compounds from the headspace of their respective vials. The treatment chemistry was particularly effective at adsorbing isovaleric acid. The treatment chemistry also worked well at adsorbing isobutyraldehyde and limonene, although the performance was not as dramatic as with the polyester fabric samples discussed above. Again, the decrease in odor adsorption with multiple washes was less pronounced with Examples 4 and 5, as compared to Comparative Examples B-D.
TABLE 3
MEASURE OF VOLATILE COMPOUNDS PRESENT IN HEADSPACE OF STAINS CONTAINING COMPARATIVE EXAMPLE E AND EXAMPLES 6, 7, AND 8 (NYLON COTTON WOVEN FABRIC)

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>0 washes</th>
<th>5 washes</th>
</tr>
</thead>
<tbody>
<tr>
<td>VOLATILE COMPOUND: ISOCALERIC ACID</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Comp. Ex. E</td>
<td>1803</td>
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<tr>
<td>Example 6</td>
<td>5</td>
<td>76</td>
</tr>
<tr>
<td>Example 7</td>
<td>0</td>
<td>2</td>
</tr>
<tr>
<td>Example 8</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>VOLATILE COMPOUND: ISOBUTYRALDEHYDE</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Comp. Ex. E</td>
<td>250</td>
<td>239</td>
</tr>
<tr>
<td>Example 6</td>
<td>41</td>
<td>158</td>
</tr>
<tr>
<td>Example 7</td>
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<td>82</td>
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<td>Example 8</td>
<td>42</td>
<td>102</td>
</tr>
<tr>
<td>VOLATILE COMPOUND: LIMONENE</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Comp. Ex. E</td>
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<td>8625</td>
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<td>Example 6</td>
<td>9142</td>
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<td>Example 7</td>
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</tr>
<tr>
<td>Example 8</td>
<td>8849</td>
<td>7806</td>
</tr>
</tbody>
</table>

[0074] As shown from the test data above, the fabrics of Examples 6, 7, and 8 adsorbed more volatile compounds from the headspace of their respective vials than did Comparative Example E. The treatment chemistry was particularly effective at adsorbing isocaleric acid, and also worked exceptionally well at adsorbing isobutyraldehyde. Further, the treatment chemistry performed better than Comparative Example E, after both had been washed 5 times, at adsorbing limonene. It should also be noted that the superior performance of Examples 7 and 8 was especially evident after the samples had been subjected to multiple launderings.

[0075] The results shown above indicate that the present chemical treatment provides odor abatement and that the chemical treatment is durable to laundering, both of which represent a useful advance over the prior art.

We claim:

1. A treated textile substrate, said treated textile fabric comprises:
   (a) a synthetic-containing textile substrate; and
   (b) a treatment applied to at least one side of said textile substrate, said treatment comprising:
      (i) a hyperbranched polyethyleneimine derivative of the formula:
         \[
         (R_1)^m \cdot h\text{-PEI}\cdot (A)_n
         \]
         where \( R \) is a non-hyperbranched hydrocarbon group, said hydrocarbon group having at least one linear portion, said linear portion having between 5 and 30 carbon atoms; where \( x \) is a number from 1 to 10,000; where \( h\text{-PEI} \) is a hyperbranched polyethyleneimine; where \( A \) is an organic compound having from 1 to 4 carbon atoms; where \( y \) is a number from 0 to 500; and wherein \( R \) is present in an amount of between about 0.1% and about 20% by weight of said hyperbranched polyethyleneimine derivative; and
      (ii) a cross-linking agent.

2. The treated textile substrate of claim 1, wherein said substrate is a fabric.

3. The treated textile substrate of claim 1, wherein said treatment is applied to said substrate at an add-on level of between about 0.1% to about 10%, based on the weight of said substrate.

4. The treated textile substrate of claim 3, wherein said treatment is applied to said substrate at an add-on level of between about 0.2% to about 5%, based on the weight of said substrate.

5. The treated textile substrate of claim 1, wherein said h-PEI has a number average molecular weight (\( M_n \)) in the range of about 300 to about 2 million.

6. The treated textile substrate of claim 5, wherein said h-PEI has a number average molecular weight (\( M_n \)) in range of about 1,000 to about 75,000.

7. The treated textile substrate of claim 1, wherein said linear portion of said R group has between about 10 and about 24 carbon atoms.

8. The treated textile substrate of claim 1, wherein said h-PEI and said R group are present, in a weight ratio, of from about 100:1 to about 10:1.

9. The treated textile substrate of claim 1, wherein said A group is at least one compound selected from the group consisting of ethylene oxide, propylene oxide, butylene oxide, methyl, acetate, vinyl sulfonate, trifluoroacetate, and trialkyl silyle.

10. The treated textile substrate of claim 9, wherein said A group is ethylene oxide or propylene oxide.

11. The treated textile substrate of claim 1, wherein said R group has a \( C_3 H_{15} \) structure, resulting in a stearic amide linkage between said R group and said h-PEI.

12. The treated textile substrate of claim 1, wherein said cross-linking agent is selected from the group consisting of isocyanate cross-linking agents, protected isocyanate cross-linking agents, and melamine formaldehyde cross-linking agents.

13. A process of treating a textile substrate, said process comprising:
   (a) providing a synthetic-containing textile substrate;
   (b) providing a dispersion comprising a hyperbranched polyethyleneimine derivative and a cross-linking agent, said hyperbranched polyethyleneimine derivative being of the formula:
      \[
      (R_1)^m \cdot h\text{-PEI}\cdot (A)_n
      \]
      where \( R \) is a non-hyperbranched hydrocarbon group, said hydrocarbon group having at least one linear portion, said linear portion having between 5 and 30 carbon atoms; where \( x \) is a number from 1 to 10,000; where \( h\text{-PEI} \) is a hyperbranched polyethyleneimine; where \( A \) is an organic compound having between 1 to 4; where \( y \) is a number from 0 to 500; and wherein \( R \) is present in an amount of between about 0.1% and about 20% by weight of said hyperbranched polyethyleneimine derivative; said dispersion containing said hyperbranched polyethyleneimine derivative, said dispersion containing said hyperbranched polyethyleneimine derivative being produced by:
      (i) providing said hyperbranched polyethyleneimine derivative, a solubilizing agent, and water, said water having a temperature at least equal to the melting point of said hyperbranched polyethyleneimine derivative; and
      (ii) subjecting said hyperbranched polyethyleneimine derivative, said solubilizing agent, and said water to high speed and high shear agitation;
(c) applying said dispersion to said textile substrate; and
(d) drying said textile substrate.

14. The process of claim 13, wherein said textile substrate
is a fabric.

15. The process of claim 13, wherein said solubilizing
agent is acetic acid.

16. The process of claim 13, wherein heat is applied during
step (i) or step (ii) to facilitate formation of said dispersion.

17. A treated textile substrate, said treated textile fabric
comprises:
(a) a synthetic-containing textile substrate; and
(b) a treatment applied to at least one side of said textile
substrate, said treatment comprising:
a hyperbranched polyethyleneimine derivative of the
formula:
(R)\text{h-PEI-(A)}\text{h}

where R is a non-hyperbranched hydrocarbon group, said
hydrocarbon group having at least one linear portion,
said linear portion having between 5 and 30 carbon
atoms; where x is a number from 1 to 10,000; where A is
an organic compound having from 1 to 4 carbon atoms;
where y is a number from 0 to 500; where h-PEI is a
hyperbranched polyethyleneimine; and wherein R is
present in an amount of between about 20% and about
80% by weight of said hyperbranched polyethyleneimine
derivative.

18. The process of claim 17, wherein said substrate is a
fabric.

19. A process of treating a textile substrate, said process
comprising:
(a) providing a synthetic-containing textile substrate;
(b) providing a dispersion comprising a hyperbranched
polyethyleneimine derivative and a cross-linking agent,
said hyperbranched polyethyleneimine derivative being
of the formula:
(R)\text{h-PEI-(A)}\text{h}

where R is a non-hyperbranched hydrocarbon group, said
hydrocarbon group having at least one linear portion,
said linear portion having between 5 and 30 carbon
atoms; where x is a number from 1 to 10,000; where A is
an organic compound having from 1 to 4 carbon atoms;
where y is a number from 0 to 500; where h-PEI is a
hyperbranched polyethyleneimine; and wherein R is
present in an amount of between about 20% and about
80% by weight of said hyperbranched polyethyleneimine
derivative, said dispersion containing said hyper-
branched polyethyleneimine derivative being produced by:
(i) providing said hyperbranched polyethyleneimine
derivative, a solubilizing agent, and water, said water
having a temperature at least equal to the melting
point of said hyperbranched polyethyleneimine
derivative; and
(ii) subjecting said hyperbranched polyethyleneimine
derivative, said solubilizing agent, and said water to
high speed and high shear agitation;
(c) applying said dispersion to said textile substrate; and
(d) drying said textile substrate.

20. The process of claim 19, wherein said substrate is a
fabric.

21. The process of claim 19, wherein heat is applied during
step (i) or step (ii) to facilitate formation of said dispersion.

* * * * *