SLOW-RELEASE INSECT-REPELLENT COMPOSITIONS AND USES

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Appl. No.: 10/010,148
Filed: Nov. 12, 2001

Related U.S. Application Data
Division of application No. 09/137,426, filed on Aug. 20, 1998, now patented, which is a continuation-in-part of application No. 08/663,267, filed on Dec. 12, 1996, now abandoned, which is a continuation-in-part of application No. 08/173,416, filed on Dec. 23, 1995, now abandoned.

Publication Classification
Int. Cl. 7 .......................... A01N 25/34; B32B 27/04; B32B 27/12
U.S. Cl. .................................. 424/411; 442/123

ABSTRACT
A slow-release insect repellent composition comprising an insect repellent, an oleophilic chemical soluble in the insect repellent, and a carbohydrate matrix wherein the combination of the insect repellent and oleophilic chemical is entrapped in the matrix such that the repellent is released from the matrix, a fabric substrate containing the composition and method for preparing the composition.
SLOW-RELEASE INSECT-REPELLENT COMPOSITIONS AND USES

CROSS-REFERENCE TO RELATED APPLICATION

[0001] This is a continuation-in-part of U.S. application Ser. No. 08/663,267, filed Dec. 22, 1994, which in turn is a continuation-in-part of U.S. application Serial No. 08/173, 416 filed Dec. 23, 1993 entitled “SLOW-RELEASE INSECT-REPELLENT COMPOSITIONS AND USES.”

FIELD OF THE INVENTION

[0002] The present invention relates to fabric substrates such as woven, non-woven, and knit substrates that are treated with an insect-repellent composition comprising amine insect repellents such as N,N'-diethyl-m-toluamide, at least two reactive silicones, and starch.

BACKGROUND OF THE INVENTION

[0003] There is a universal need for outdoor fabrics suitable for apparel use and providing for long term insect repellency. Although many insect repellents are effective when applied to the skin as a spray or lotion, their effectiveness is known to decline with time. Furthermore, in order for an insect repellent to be effective, the insect repellent must be applied in a concentrated amount to a small area of the face, neck, or hands or on selected areas of a wearer’s garment.

[0004] The effectiveness of insect repellents has been known to improve when such repellents are directly applied to fabrics which are suitable for outdoor use, and various techniques have been suggested for providing sustained release of an insect control agent.

[0005] For example, in U.S. Pat. No. 4,056,610 to Barber, et al., the invention provides for a microcapsular insecticidal composition comprising a pyrethroid and a biological synergist capable of controlling insects for up to (4) days.

[0006] In U.S. Pat. No. 5,198,287 to Samson, the invention provides for a tent fabric coated on the inside with a composition which renders it water repellent, flame retardant and insecticidal using permethrin as the insecticide. Permethrin is protected against oxygen with a plasticizer. The outside of the tent is coated with a composition which renders it water repellent and flame retardant exclusive of the insecticide. The insecticide composition has an effective life of more than six (6) months.

[0007] In U.S. Pat. No. 4,765,982 to Ronning, the invention relates to compositions, devices and methods for controlling insect activity wherein an insect control agent is self-adhered to a rough-surfaced fiber and provides extended control of insect activity. The microencapsulated insect control agents disclosed in Barber are named as the preferred insect control for use in Ronning’s invention. Ronning teaches that smooth-surfaced fibers do not act as good sites for adhesion of microencapsulated insect control agents.

[0008] In U.S. Pat. No. 5,003,635 to Peterson, the invention relates to elongated flexible insect-repellent strips secured to or retained in cavities in various articles of apparel to repel insects from the person wearing such apparel.

SUMMARY OF THE INVENTION

[0009] In U.S. Pat. No. 4,833,006 to McKinney, the invention relates to a coated fabric that is flame resistant and water repellent and includes a polyfunctional isocyanate as an adhesion promoter and binder to adhere the coating to the substrate in a flake proof manner.

[0010] In U.S. Pat. No. 5,089,298 to McNally, the invention relates to the impregnation of Battle Dress Uniforms used by the U.S. Military with amylopectin fabric wrinkle inhibitor and permethrin insect/arthropod repellent in combination.

[0011] In U.S. Pat. No. 3,859,121 to Yeadon et al., the invention relates to textiles impregnated with insect-repellent compositions including piperonyl butoxide and pyrethrin, wetting agent, thickener and an agent for preventing migration of the piperonyl butoxide and pyrethrin repellent into foods stored in contact with the textiles and for otherwise retaining the repellent in the textiles.

[0012] An amine insect repellent such as, for example, N,N'-diethyl-m-toluamide (DEET) is an effective insect repellent. In addition, it is believed that DEET is generally environmentally safe and leaves no or minimal harmful residues. DEET has been found to be stable at both reduced and elevated temperatures and under storage conditions. DEET is effective on biting flies, chiggers, deerflies, fleas, lice, mosquitoes, and ticks. Several factors influence the efficacy of DEET as an insect repellent, such as, for example, extreme environmental conditions, the extent of absorption and elimination, the type of repellent composition, and the avidity of the test species. Typically, the efficacy of DEET tends to be reduced by increased perspiration and external water sources such as rain.

[0013] One object of the present invention is to provide an insect-repellent composition and process of making same such that the insect repellent is slowly released. It is a further object of this invention to provide for slow-release insect-repellent compositions and a process of manufacturing same that may be applied to a fabric substrate such as woven, non-woven and knit substrates and result in slow-release of the insect repellent from the fabric.

[0014] The present invention relates to a fabric composition comprising a fabric substrate and an insect-repellent composition, wherein the insect-repellent composition comprises an amine insect repellent, at least two reactive silicones, and starch.

[0015] The present invention relates to a fabric composition comprising: (a) a fabric substrate; and (b) an application of an insect-repellent composition formed by an insect-repellent composition consisting essentially of an amine insect-repellent, at least two reactive silicones, and starch. The reactive silicones, of the insect-repellent composition may comprise a substituted organosilane and silanol functional polymer. The starch of the insect-repellent composition may be modified corn starch. The amine insect-repellent of the insect-repellent composition may be N,N'-diethyl-m-toluamide. The fabric substrate may be selected from the group consisting essentially of woven, non-woven and knit substrates. In one embodiment, the fabric composition further comprises the finishing agent which may be selected from the group consisting of at least {i} two reactive silicones, and/or {ii} a finishing resin, cationic softener and/or wax emulsion.
In another embodiment, the present invention relates to a fabric composition comprising a fabric substrate and an application of an insect-repellent composition, wherein the application of the insect-repellent composition comprises an aqueous dispersion consisting essentially of an amine insect-repellent, at least two reactive silicones, and starch. The aqueous dispersion is spray dried to form a powder and then the powder is applied to the fabric substrate to form the fabric composition that provides insect-repellency.

In a further embodiment, the present invention relates to a fabric composition comprising a fabric substrate and an application of an insect-repellent composition, (i) wherein the application of the insect-repellent composition comprises the steps of: (a) mixing an aqueous dispersion consisting essentially of N,N-diethyl-m-toluamide, a substituted organosilane and silanol functional polymer, and modified corn starch; (b) spray drying the aqueous dispersion of step (a) to form a powder; (c) adding an effective amount of the powder formed in step (b) to a dye bath and dyeing said fabric substrate; (d) adding an effective amount of the powder formed in step (b) to a print paste and printing on the fabric substrate of step (c); and (e) adding an effective amount of the powder formed in step (b) to a finishing solution comprising at least two reactive silicones, and/or finishing resin and applying the finishing solution onto the fabric substrate of step (d) to form the fabric composition; (ii) wherein said fabric composition provides insect-repellency.

In still a further embodiment, the present invention relates to a method of manufacturing a fabric substrate comprising the steps of: (a) providing a fabric substrate; (b) mixing an aqueous dispersion consisting essentially of an amine insect-repellent, at least two reactive silicones, and a starch; (c) spray drying the aqueous dispersion of step (b) to form a powder; and (d) applying the powder of step (c) to the fabric substrate to form a fabric composition that provides insect repellency.

DETAILED DESCRIPTION OF THE INVENTION

Oleophilic silicone compounds, heavy alcohols (such as, for example, polyvinyl alcohol), polyvinyl acetate, liquefied synthetic rubber, or acrylic copolymers and the insect repellent N,N-diethyl-m-toluamide (DEET) can be combined in an aqueous solution and then dried to form an insect-repellent composition. The composition has novel properties including the time-release of DEET. The rate of release of DEET may be controlled by varying the amounts of oleophilic compounds in the composition. The period of time may span from about 24 hours to about several months and may be affected by the method of storage of the powder and exposure to light and air.

Other novel properties of the composition include dispersability in water and water-based compounds (such as for example lotions, creams, latex paints, acrylic-based paints and sealers) as well as in oil, hydrocarbon solvents and the like.

It is believed that the time-release of DEET of the present invention may be attributed to the entrapment of DEET in the resulting structure of the capsule-forming compounds and the entrapping effect of the oleophilic compounds. In one embodiment, the time-release property of DEET and the overall stability of the insect-repellent composition of the present invention may be affected by further coating the composition with, for example, a waxy compound, gelatin, polyethylene glycol and derivatives, for an intermediate coating or with, for example, clay, china or ivory, for a harder coating or a protective layer. For purposes of the present invention the term “capsule,” “matrix,” and “latex-like structure,” will be used interchangeably and are equivalent.

In one embodiment, the insect-repellent composition of the present invention may be applied to a wide range of fabric substrates without being limited by the coarseness or smoothness of the fabric substrate. Furthermore, the insect-repellent composition of the present invention may be combined with a wide range of fabric treatment compositions. The resulting formulation may be applied to any suitable fabric substrate depending on the intended use of the fabric and the fabric treatment process such as, for example, cottons, knits, polyesters or blends, fiberglass, wovens or non-wovens and heat-sensitive substrates such as acetates and acrylates and nylon. In order to lower the insect-repellent volatility and control the loss of insect-repellent activity, the compositions may be applied such that multiple layers of super thin laminates are produced.

The insect-repellent composition of the present invention can be prepared by first formulating the insect repellent N,N-diethyl-m-toluamide with an oleophilic chemical such as a silicone compound such as, for example, elastomeric or monomeric silicone, and then it is believed by mechanical and/or chemical entrapping of the resulting compound in a carbohydrate matrix, such as, for example, a starch compound including, but not limited to, modified corn starch, potato starch, technical starch, rice starch and any of the synthetic starches. Silicone is dissolved with DEET which is also immiscible with water. The DEET-silicone solution is mixed into a starch solution with the resulting solution being miscible in water. It is believed that the DEET molecule is entrapped in the lattice-like structure of the starch molecule that has the effect of lowering the vapor pressure of the insect repellent thus reducing its volatility. DEET is slowly released from the lattice-like structure of the starch with the net effect of the formation of an insect-repellent composition.

In one embodiment, the silicone compound used in the present invention contains at least two reactive silicones. For example, the silicone compound comprises a substituted organosilane and a silanol functional polymer. One example of a silanol functional polymer is silandiol. An example of a material that contains two reactive silicones is a product with a trade name “Hydrolast 284M.”

It is believed that the silicone compounds in the above embodiment provides at least two functions. First, during the mixing of the aqueous solution containing DEET, the silicone compound and the starch, the objective is to form a homogeneous emulsification. During this step, it is believed that the silicone compound acts like a surfactant by effectively emulsifying the starch, which is hydrophilic, and DEET, which is oleophilic. As such, in one embodiment, suitable silicones are oxodervative silicones, for example, trioxo-silane and silanol. Since in one embodiment, the
mixing of this aqueous solution occurs at ambient temperature, a DEET/starch/silicone emulsion is essentially produced.

[0026] Subsequently, in this embodiment, the DEET/starch/oxo-derivative silicones are dried at elevated temperature such as during spray drying. During this step, it is believed that the reactive silicones, such as trioxo-organosilane and silanediol react at this elevated temperature. More specifically, in one embodiment, it is believed that, at elevated temperatures during drying, the reactive silicones undergo a condensation reaction to produce a cross-linked polymer. It is believed that the reaction progresses as follows:

\[
\begin{align*}
\text{(RO)}_3\text{Si} \rightarrow & \text{R} \rightarrow \text{(HO)}_3\text{Si} \rightarrow \text{R} \\
\text{(HO)}_3\text{Si} - \text{R} + \text{HO} \rightarrow & \text{(SO)}_2\text{Si} - \text{OH} \\
\text{HO} - \text{Si} \rightarrow \text{(OS)}_2\text{O} \rightarrow & \text{Si} - \text{R'} \\
\text{HO} - \text{Si} \rightarrow \text{(OS)}_2\text{O} \rightarrow \text{Si} - \text{OH + H}_2\text{O} \\
\end{align*}
\]

[0027] a) \((\text{RO})_3\text{Si} \rightarrow \text{R} \rightarrow (\text{HO})_3\text{Si} \rightarrow \text{R}''

[0028] b) condensation of silane and silanol:

[0029] where R is any functional group.

[0030] In addition, it is believed that the end product may further undergo self-condensation. It is also believed that a cross condensation reaction with the starch may also occur.

[0031] In yet another preferred embodiment, the insect-repellent composition consists of from about 40% to about 75% weight starch, and of from about 10% to about 35% weight DEET, and of from about 15% to about 25% weight silicone. In another embodiment, the insect-repellent composition consists of from about 50% to about 72% weight starch, and of from about 20% to about 32% weight DEET and of from about 4% to about 10% weight silicone, and from about 4% to about 8% weight water. Following spray-drying, the insect-repellent composition may have a particle size from about 0.005 mm to about 1.800 mm.

[0032] In one embodiment, the insect-repellent composition of the present invention is prepared by first dissolving starch in water at its boiling point. The starch solution is then cooled to room temperature by further addition of water and the amine insect-repellent DEET and silicone are slurried into the starch solution. Following slurring, the composition is spray dried using conventional spray drying equipment such that the slurry produces a fine while powder comprising DEET. It is to be understood that the term powder used herein includes particles ranging from about 0.0005 mm to about 1.8 mm. For purposes of the present invention, “emulsified,” “slurried,” “homogeneous mixture,” and “dispersed” are equivalent.

[0033] As the following embodiments describe, the insect-repellent composition of the present invention may be formulated for application on fabric substrate in high yields without affecting the functionality, esthetic appearance, hand or feel of the treated substrate and leaving minimal residual odor.

[0034] In one embodiment, the insect-repellent composition may be combined with a cationic softener and applied as a finishing rinse to a fabric substrate that has been already dyed. A finishing rinse is the last rinse step in a dyeing procedure during the processing of textile material. For example, the insect-repellent composition may be combined into a formulation comprising of from about 2% to about 10% on the weight of the fabric (owf) of the insect-repellent composition of the present invention and of from about 2% to about 6% (owf) cationic softener. The term “owf” refers to on (o) weight (w) of fabric. This formulation may be exhausted onto the fabric substrate under mildly acidic conditions, for example using acetic acid such that the pH is of from about 5.50 to about 6.60 with the insect-repellent composition adhering to the cationic sites in dyeing and the cationic softener adhering to the fabric substrate. It should be understood that the term “finishing agent” includes chemicals or compositions added during the dyeing, printing and/or finishing step of a fabric.

[0035] In another embodiment, the insect-repellent composition may be combined in a composition comprising of from about 90% to about 150% (owf) of a padding composition and of from about 2% to about 10% (owf) of the insect-repellent composition. A padding composition refers to the mixture of 2% (owf) of the cationic softener and the insect-repellent composition that is applied to the fabric in an immersion tank and squeezed into the fabric with two or more squeeze rollers. Padding is a process of applying any chemical composition to any fabric using an immersion tank and a squeeze roller.

[0036] In yet another embodiment, the insect-repellent composition may be combined in a composition comprising of from about 90% to about 150% (owf) of a pigment printing paste composition and of from about 2% to about 10% (owf) of the insect-repellent composition. A pigment printing paste is referring to a colloidal suspension of water, melamine or acrylic binding or holding resin, synthetic thickener, pigment color or colors, and catalysts, alcohols or ammonias used during the screen printing of textiles and other substrates. It is believed that the insect-repellent composition cross-links with the pigment binder during pigment dyeing and printing to link on to the cellulose groups of the fabric substrate.

[0037] In yet a further embodiment, the insect-repellent composition of the present invention may be combined in a composition comprising of from about 90% to about 150% (owf) of water and of from about 2% to about 10% (owf) of the insect-repellent composition.

[0038] In another embodiment, the insect-repellent composition of the present invention may be used in a final rinse composition. Fabric substrates after dyeing, pigment padding and printing, with the appropriate composition which comprises the insect-repellent composition of the present invention, may be given a resin finish for hand, appearance and dimensional stability. In this particular embodiment, it is believed that the resin finish may act as a super thin laminate and provide the treated fabric with an insect-repellent finish. More particularly, when the resin used is utra-formaldehyde, it is believed that cross-linking of the molecular structure of the cotton fiber to the cellulose fiber occurs during resin curing conditions. It is further believed that the insect-repellent composition is trapped during the reaction.
of the urea-formaldehyde with the cellulose molecule in the cotton fabric. In one embodiment, substrate fabrics of 100% cotton and a 50%-50% polyester/cotton blend may be treated with a resin finish comprising of from about 2% to about 5% (owf) of the insect-repellent composition of the present invention and of from about 95% to about 150% (owf) of a resin finish composition.

[0039] In another embodiment, the insect-repellent composition of the present invention may be used in heat-transfer printing using a wide range of substrate fabrics. For example, from about 2% to about 10% (owf) of the insect-repellent composition may be combined with of from about 2% to about 5% (owf) of the ink dispersion and with of from about 85% to about 96% (owf) of the extender. An ink dispersion is usually used in ink jet printing, hand screen, and semi-automatic screen printing to produce textiles with designs affixed to the textiles. An extender is usually a water-soluble alcohol or vinyl emulsion that is added to the ink to control the color level of the ink being applied and to link the ink onto the product or textile being printed. The extender can also be an organic alcohol or resin that is solvent-based. The fabric was processed by heat-transfer printing, i.e., by vaporization of the ink from the paper to the fabric at the sublimation temperature of the ink. In another embodiment, the ink dispersion can be omitted and about 100% (owf) of the extender can be used. In both cases, the adhesion of the heat transfer paper to the fabric prevents the insect repellent from volatilizing and escaping resulting in greater durability.

[0040] In one embodiment, of from about 2% to about 10% (owf) of the insect-repellent composition of Example I may be combined with an extender. The fabric was processed by heat-transfer printing, i.e., by vaporization of the ink from the paper to the fabric at the sublimation temperature of the ink.

EXAMPLE I

[0041] In this example, an insect-repellent composition may be prepared by mixing into a starch slurry a mixture of silicone of about 4% weight/weight (w/w) and the insect-repellent DEET of about 8% (w/w). The starch slurry is formed by dissolving about 8% (w/w) starch into about 8% (w/w) water at its boiling point. The mixture of insect repellent and silicone is added to the starch mixture after cooling the slurry to room temperature.

[0042] The resultant slurry is spray dried in a spray drying chamber at an inlet temperature of about 230°F using an atomizing force of from about 2400 to about 3200 psig from an atomizer. The slurry produces a fine white powder comprising of from about 0.1% to about 32% (w/w) DEET and with of from about 4% to about 8% (w/w) moisture.

EXAMPLE II

[0043] From about 2% to about 10% (owf) of the insect-repellent composition of Example I can be combined with of from about 3% to about 6% (owf) cationic softener and exhausted on a fabric substrate that has been dyed on a jigg, beck, beam or jet at about 120°F, for about fifteen (15) minutes and at a pH of from about 5.50 to about 6.60, adjusted with acetic acid.

EXAMPLE III

[0044] From about 2% to about 10% (owf) of the insect-repellent composition of Example I can be combined with a pigment padding composition of about 0.06% (owf) of a aqueous ammonia, about 4.78% (owf) of a padding emulsion (for example, padding emulsion no. 9098 from BASF), about 2.39% (owf) of a anti-migrant (for example, anti-migrant no. 909-95915 from BASF), about 0.48% (owf) ammonium sulphate, water, and a variable weight percent of a pigment dye depending on the shade. The resulting composition is padded onto the fabric at from about 85% to about 150% (owf) wet pickup, dried at about 275°F and cured at about 340°F for about thirty (30) seconds.

EXAMPLE IV

[0045] From about 2% to about 10% (owf) of the insect-repellent composition of Example I can be combined with a pigment printing composition of about 4.7% (owf) of a dispersion of an acrylic copolymer in mineral oil (for example, Allied DP-5205 from Allied Colloids, Inc.), about 10% (owf) of a white aqueous-based acrylate copolymer emulsion (for example, Allied PB-8A from Allied Colloids, Inc.), about 83.3% (owf) water, and a variable weight percent of a pigment print dye depending on the shade. The resulting composition is printed onto the fabric in all colors of the pattern and cured at about 340°F for about sixty (60) seconds.

EXAMPLE V

[0046] From about 2% to about 10% (owf) of the insect-repellent composition of Example I can be combined with a water repellent composition of from about 3% to about 6% (owf) silicone softener, about 6% (owf) wax emulsion (for example, fluoropolymer wax emulsion PEL-TEK 508, Hydrolabs, Inc.), and of from about 4% to about 7% (owf) of a glyoxal reagent (for example, REACTEX #7222 from Ivax Industries, Inc.). The resulting composition is padded onto the fabric from about 85% to about 150% (owf) wet pickup, dried at about 275°F and cured at about 340°F for about twenty (20) seconds.

EXAMPLE VI

[0047] From about 2% to about 10% (owf) of the insect-repellent composition of Example I can be combined with a resin finishing composition suitable for treating 100% cotton and comprising of from about 3% to about 6% (owf) of the cationic softener, of from about 1% to about 2% (owf) of the silicone softener, and of from about 4% to about 7% (owf) of the glyoxal reactant.

[0048] From about 2% to about 10% (owf) of the insect-repellent composition of Example I can also be combined with a resin finishing composition suitable for treating 50%/50% polyester/cotton and comprising of from about 3% to about 6% (owf) of the cationic softener, of from about 1% to about 2% (owf) of the silicone softener, and of from about 2% to about 4% (owf) of the glyoxal reactant.

[0049] The resin finishing composition may be applied to a fabric substrate that has been dyed, pigment padded, and/or printed as in the above Examples. The resin finish acts as a super thin laminate and provides an additional insect-repellent coating.

[0050] With both formulas and fabrics, the resulting composition is padded onto the fabric at from about 85% to about 150% (owf) wet pickup, dried at about 275°F and cured at about 340°F for about twenty (20) seconds.
EXAMPLE VII

[0051] From about 2% to about 10% (owf) of the insect-repellent composition of Example I can be combined with about 20% (owf) of the ink dispersion and about 75% (owf) of the extender. The fabric was processed by heat-transfer printing, i.e., by vaporization of the dye from the paper to the fabric at the sublimation temperature of the dye.

EXAMPLE VIII

[0052] From about 2% to about 10% (owf) of the insect-repellent composition of Example I can be combined with about 100% extender. The fabric was processed by heat-transfer printing, i.e., by vaporization of the dye from the paper to the fabric at the sublimation temperature of the dye.

EXAMPLE IX

[0053] An insect-repellent composition was prepared by first dissolving about 8% (w/w) of modified corn starch ("CAPSUL" from National Starch and Chemical) into about 80% (w/w) water at its boiling point. After cooling the starch slurry to room temperature, about 8% (w/w) of DEET and about 4% (w/w) of reactive silicone containing substituted silane and silanol ("Hydrolast 284M") were added. The mixture was agitated until a homogeneous mixture was obtained.

[0054] The resultant slurry was spray dried in a spray drying chamber at about 230°F. The slurry produced the insect-repellent composition in the form of a white powder comprising between about 20% to about 32% (w/w) of DEET.

[0055] The insect-repellent composition was added to a dye bath to produce a dye bath containing about 3% (owf) of DEET. A white fabric substrate of 100% cotton was immersed in the dye bath and allowed to come to equilibrium with the dye bath. The fabric substrate was removed from the dye bath.

[0056] Subsequently, the dyed fabric containing about 3% (owf) of DEET was combined with a pigment printing formulation having the insect-repellent composition containing about 3% (owf) of DEET. The resulting formulation was printed onto the fabric in all colors to produce a camouflage pattern.

[0057] The fabric composition was then combined with a resin finishing composition containing about 3% (owf) of Hydrolast 284M, about 5% (owf) of urea-formaldehyde resin, and the insect-repellent composition containing about 5% (owf) of DEET. The fabric composition was squeeze-rolled and dried at about 300°F.

[0058] It should be understood that Example IX above can be modified to involve applying the insect-repellent composition of the present invention at one or more of the individual steps—dyeing, printing, and/or finishing.

[0059] Field Testing:

[0060] The insect-repellent compositions of the present invention were found to afford the wearer complete protection under most types of weather and infestation conditions. Furthermore, the insect-repellency effectiveness of the fabric substrate treatment was found to span up to a period of about several hours to at least several weeks.

[0061] To test the stability of the fabric treatment, garments were produced from treated fabric. Fabric substrates which were laundered from ten (10) to fifty (50) launderings continue to exhibit insect-repellent properties. It is to be noted that typically the industry standard number of washings for apparel is three (3), five (5), or ten (10) for consumer apparel and fifty (50) washings for military or certain specialty requirements such as, for example, flame retardancy.

[0062] To test the effectiveness of the treatment, laboratory size knit and woven fabric samples were processed using the compositions described above. A first batch of the samples were tested for direct insect repellency using fire ants and fruit flies as test insects. For several hours the fire ants and fruit flies were directly repelled by the fabric samples. A second batch of the fabric samples were tested by weavers who wore shirts made from the fabric sample. Mosquitoes and blackflies were repelled for at least eleven (11) hours during a fishing trip.

[0063] A production trial was initiated on four (4) fabric substrates, knit and woven cotton, poly/cotton and poly/nylon. The fabrics were dyed with the insect-repellent composition applied during dyeing and/or resin finishing. Another group of knit and woven fabrics were predyed, printed and resin finished with the insect-repellent composition applied at the dyeing, printing and resin finishing steps.

[0064] Garments such as shirts, hats, and the like were produced from the treated fabric substrates. The garments were tested under conditions encountered during outdoor activities. The garments were found to repel insects under outdoor conditions during activities such as hiking, hunting, and fishing in coastal, piedmont and mountain conditions during day and night wear. The treated garments were compared to untreated counterparts under the same conditions. In one particular field trial, garments were tested on mosquitoes. The mosquitoes would land on exposed skin and promptly bite. By contrast, when the mosquitoes landed on the treated fabric at any location on the garment, they would only remain for about four (4) to about six (6) seconds before taking off.

[0065] This present invention and many of its attendant advantages will be understood from the foregoing description, and it will be apparent that various modifications and changes can be made without departing from the spirit and scope of the present invention or sacrificing all of its material advantages, the process hereinafter described being merely preferred embodiments.

[0066] For example, the insect-repellent composition of the present invention can incorporate different insect control agents such as, for example, alkylamines and alkylisocyanates. Additionally, the insect-repellent composition may be applied on a wide range of fabric substrates including but not limited to textile blends, woven and non-woven, knits, fibers, leather and synthetic adaptations of leather, flocked-fabrics, wood and wood derivatives, plastic and laminates, cable, sheeting, film fiberglass and plexiglass. The insect-repellent composition may also be comprised in moth proofing composition, packaging material, and paint compositions. Furthermore, a longer-term repellency can be obtained when such capsules are used in the manufacture of commercial finishes such as caulking compositions, paint seal-
ers, wall and floor coverings and the like. Additionally, the compositions of the present invention can have a wide applicability in situations requiring a lower concentration of DEET. As an example, the insect-repellent composition may be incorporated with a scent or a fragrance or pheromones or with any other compositions that could be conducive to applications in sporting, farming, or hunting situations. Conversely, higher concentrations of up to from about 20% to about 25% of the insect-repellent composition can be used in caulking compounds.

[0067] The present invention may be embodied in other specific forms without departing from its spirit or essential attributes. Accordingly, reference should be made to the appended claims, rather than the foregoing specification, as indicating the scope of the present invention.

What is claimed is:

1. A slow-release insect-repellent formulation for a fabric substrate, said formulation comprising an insect repellent, an oleophilic chemical soluble in said insect repellent, a carbohydrate matrix wherein the combination of said insect repellent and said oleophilic chemical is entrapped in said matrix such that said insect repellent is released from said matrix.

2. The formulation of claim 1 wherein the weight ratio of said insect repellent to said oleophilic chemical is of from about 0.13:0.2 to about 0.875:0.635 per weight of said carbohydrate matrix.

3. The formulation of claim 2 wherein said insect repellent is encapsulated in a microcapsule with a diameter of from about 0.005 mm to about 1.800 mm and wherein said insect repellent is from about 10% to about 35% of the weight of said formulation.

4. The formulation of claim 3 further comprising a fabric treatment formulation, said fabric treatment formulation comprises from about 2% to 6% (owf) cationic softener.

5. The formulation of claim 4 wherein said fabric treatment formulation is a padding formulation of from about 90% to about 98% (owf).

6. The formulation of claim 4 wherein said fabric treatment formulation is a pigment printing paste formulation of from about 90% to about 98% (owf).

7. The formulation of claim 4 wherein said fabric treatment formulation is a pigment printing paste formulation of from about 90% to about 98% (owf).

8. The formulation of claim 4 wherein said fabric treatment formulation is a water repellent formulation of from about 90% to about 98% (owf).

9. The formulation of claim 4 wherein said fabric treatment formulation is a finishing rinse formulation of from about 2% to about 5% (owf).

10. The formulation of claim 4 wherein said fabric treatment formulation is an ink formulation comprising from about 2% to about 5% (w/w) ink dispersion and from about 85% to about 96% (w/w) extender.

11. The formulation of claim 4 wherein said fabric treatment formulation comprises about 100% (w/w) extender.

12. The formulation of claim 4 wherein said insect repellent is N,N'-diethyl-m-toluamide.

13. A process for the manufacture of an insect-repellent formulation comprising the steps of combining an insect repellent with an oleophilic chemical soluble in said insect repellent; slurrying said combination in a water solution comprising said carbohydrate matrix such that said combination is entrapped in said matrix and slowly released from said matrix; and spray drying said slurry to form a powder comprising microcapsules of said insect repellent.

14. The process of claim 13 wherein the weight ratio of said insect repellent to said oleophilic chemical is of from about 0.13:0.2 to about 0.875:0.635 on a weight basis with said carbohydrate matrix.

15. The process of claim 14 wherein said microcapsules are of from about 0.005 mm to about 1.800 mm in diameter and wherein said insect repellent is of from about 3% to about 8% on a weight basis with said microcapsules.

16. The process of claim 15 which further comprises the step of combining with said powder a fabric treatment formulation.

17. The process of claim 16 wherein said fabric treatment formulation comprises of from about 2% to about 6% (owf) cationic softener.

18. The process of claim 16 wherein said fabric treatment formulation is a padding formulation of from about 90% to about 98% (owf).

19. The process of claim 16 wherein said fabric treatment formulation is a pigment printing paste formulation of from about 90% to about 98% (owf).

20. The process of claim 16 wherein said fabric treatment formulation is a water repellent formulation of from about 90% to about 98% (owf).

21. The process of claim 16 wherein said fabric treatment formulation is a finishing rinse formulation of from about 2% to about 5% (owf).

22. The process of claim 16 wherein said fabric treatment formulation is an ink formulation comprising from about 2% to about 5% (w/w) ink dispersion and from about 85% to about 96% (w/w) extender.

23. The process of claim 16 wherein said fabric treatment formulation comprises about 100% (w/w) extender.

24. The process of claim 16 wherein said insect repellent is N,N'-diethyl-m-toluamide.

48. A fabric substrate comprising the slow-release insect-repellent formulation comprising an insect repellent, an oleophilic chemical soluble in said insect repellent, and a carbohydrate matrix wherein the combination of said insect repellent and oleophilic chemical is entrapped in said matrix such that said insect repellent is released from said matrix, said fabric substrate having an insect repellency of up to twelve (12) months.

49. The fabric substrate 48 wherein the weight ratio of said insect repellent to said oleophilic chemical is of from about 0.13:0.2 to about 0.875:0.635 per weight of said carbohydrate matrix.

50. The fabric substrate of claim 49 wherein said insect repellent is comprised in microcapsules with a diameter of from about 0.005 mm to about 1.800 mm and wherein said insect repellent is of from about 3% to about 8% on a weight basis with said microcapsules.

51. The fabric substrate of claim 50 wherein said insect repellent is N,N'-diethyl-m-toluamide.

52. A garment manufactured with the fabric substrate of claim 48.

53. The formulation of claim 1 wherein said formulation is applied onto the fabric substrate.

54. The formulation of claim 53 wherein said formulation is contacted with insects.

55. The formulation of claim 1 wherein said fabric substrate consists of woven, nonwoven and knit substrates.
56. The formulation of claim 1 wherein said oleophilic chemical is silicone.
57. The formulation of claim 56 wherein said silicone is an elastomeric silicone.
58. The formulation of claim 56 wherein said silicone is a monomeric silicone.
59. The formulation of claim wherein said silicone is from about 15% to about 25% of the formulation.
60. The formulation of claim 1 further comprising silicone.
61. The process of claim 13 wherein said oleophilic chemical is silicone.
62. The process of claim 11 further comprising the step of combining silicone with said insect repellent and said oleophilic chemical.
63. The fabric substrate of claim 48 wherein said oleophilic chemical is silicone.
64. The fabric substrate of claim 63 wherein said silicone is an elastomeric silicone.
65. The fabric substrate of claim 63 wherein said silicone is a monomeric silicone.
66. The fabric substrate of claim 48 wherein said formulation further comprises silicone.