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3,697,220

BACTERIA GROWTH INHIBITING TEXTILES AND DRY CLEANING AND DISINFECTING COMPOSITIONS AND PROCESSES

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11 Claims

ABSTRACT OF THE DISCLOSURE

Disinfection of, and production of bacteria growth inhibiting fabrics or textiles is accomplished by simultaneous dry cleaning and disinfection procedures involving contact of the textile with an antimicrobial agent containing drycleaning solution in the form of an emulsion of the "water-in-oil" type and which contains a fat solvent in the continuous phase and an aqueous solution of 1 - (3 - chloroallyl - 3,5,7 - triaza - 1 - azoniaadamantane chloride in the dispersed phase.

This application is a continuation-in-part of my co-pending application Ser. No. 852,491, filed Aug. 22, 1969, now abandoned.

This invention relates to the drycleaning of fabrics or textiles and more particularly to certain chemically disinfected textiles that contain a bacteria growth inhibitor and to certain drycleaning and disinfecting compositions and methods.

There is a false popular belief that the drycleaning of textiles or woven fabrics involves a germicidal process which is destructive of the vegetative forms of pathogenic or disease causing microorganisms. It is well established however, that such procedures have only limited antibacterial effects, (see "Microbiology of Dry Cleaning" by Robert R. Banville and Ethel McNeil. "Appl. Microbiol." 14:1-7, 1966) and that many bacteria carry through the drycleaning process on the fabrics or textiles and/or are redeposited on the fabrics and textiles during the cleaning procedures. Such carry through and redepositions are, of course, undesirable because there is the resultant spread of disease producing bacteria which, if destroyed during the drycleaning procedure, would be incapable of causing subsequent human infection.

The inventor is unaware of any commercialized drycleaning procedure that simultaneously cleans and disinfects textiles or fabrics. On the other hand, it is known to fumigate textiles with formaldehyde vapors as a step separate and apart from the cleaning step and as a means for disinfecting the textiles by the destruction of the pathogenic bacteria therein.

Procedures for simultaneously cleaning and disinfecting textiles have been proposed in the past but suffer from one or more disadvantages which have prevented their commercialization insofar as the inventor is aware. For example, it is known to incorporate formaldehyde or Formalin, as a disinfectant or germicide, in organic fat solvents that may be used in drycleaning procedures. The compositions, however, suffer the disadvantage that during the cleaning process, the formaldehyde imparts a pungent odor to the textile and which requires removal by a subsequent neutralization step. This added step of course, substantially increases the time and cost of cleaning and disinfecting the fabrics. The incorporation of

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other antimicrobial agents in the fat solvents suitable for use in commercial drycleaning processes has also been proposed. Among the problems which have confronted investigators in this field, however, is that of finding an antimicrobial agent which is compatible with the solvents and detergents used in commercial drycleaning procedures and which is also germicidally effective and economical to use in treating the textiles under the normal operating conditions which are encountered in modern commercial drycleaning establishments.

The inventor has discovered that 1-(3-chloroallyl)-3,5,7-triaza-1-azoniaadamantane chloride is not only compatible with the commercially employed organic fat solvents, such as perchloroethylene and stoddard solvent, and with the usual nonionic and anionic detergents that are employed in the commercial drycleaning procedures, but that the compound serves as an effective antimicrobial agent in system involving such fat solvents and detergents and is capable of accomplishing the disinfection of the textiles under the operating conditions which are normally encountered in commercial drycleaning plants.

The antimicrobial agent advocated for use herein is insoluble in such organic fat solvents as perchloroethylene and stoddard solvent, but has a high degree of solubility in water that amounts to better than 2.2 grams of the 1 - (3 - chloroallyl) - 3,5,7 - triaza - 1 - azoniaadamantane chloride per gram of water. This has led to the discovery that the antimicrobial agent can be introduced as an additive to the current day drycleaning compositions and will therein become soluble in the small amounts of water which normally enter the system with the textiles during the cleaning procedures. This introduced water becomes emulsified and colloiddally dispersed by the synthetic detergents and consequently, the compositions of the invention are emulsions of the "water-in-oil" type and wherein the antimicrobial agent is present in an aqueous solution that constitutes the dispersed phase of the emulsion while the organic fat solvent provides the continuous phase of the emulsion. Textiles which are contacted by the compositions of the invention, as by being immersed and agitated with the compositions in accord with conventional drycleaning procedures, and which are thereafter dried are not only cleaned and disinfected but have been found to thereafter inhibit bacterial growth as will be subsequently apparent.

A general object of the invention is to provide improvements in the field of drycleaning.

Yet another object of the invention is to provide drycleaning and disinfecting compositions which can be used for the drycleaning of textiles and woven fabrics in accord with conventional drycleaning techniques and without the need for modifying or changing the equipment used in the current commercial drycleaning procedures.

Yet another object of the invention is to provide a process for drycleaning and disinfecting fabrics or textiles and which can be carried out in commercial drycleaning equipment without the modification of such equipment or changes in the drycleaning techniques that are usually employed.

Yet a further object of the invention is to provide drycleaning solutions which contain an antimicrobial agent which without imparting odors to the textiles is capable of effectively disinfecting textiles that are cleaned in such solutions.

Yet another object of the invention is to provide compositions for use in drycleaning and disinfecting textiles and which can be easily prepared and maintained by people which are basically unskilled in the formulation

of chemical compositions and which can also be economically used for such purposes.

A further object of the invention is to provide a drycleaning and disinfecting process which not only disinfects fabrics and other textiles subjected to the process but which imparts bacteria growth inhibiting properties to the textiles thus subjected to the process.

Another object of the invention is to provide textiles which have bacteria growth inhibiting properties.

In accord with the invention, the textile drycleaning and disinfecting compositions are emulsions of the so-called "water-in-oil" type and comprise a liquid organic fat solvent that forms the medium or continuous phase of the emulsion, an aqueous solution of 1-(3-chloroallyl)-3,5,7-triaza-1-azoniaadamantane chloride which is colloiddally dispersed in the fat solvent, and an emulsifier in amounts sufficient to provide a stable emulsion so that an antimicrobial agent containing dispersed phase will be brought into effective contact with textiles subjected to cleaning procedures involving the use of the compositions.

The fat solvent may be one or more of the liquid organic solvents that are suitable for drycleaning purposes, such as the chlorinated lower aliphatic hydrocarbons, exemplified by perchloroethylene, carbon tetrachloride, trichloroethylene, etc. and the aromatic and saturated aliphatic hydrocarbons such as stoddard solvent, benzene, benzol and the gasolines. In modern drycleaning plant practices stoddard solvent or perchloroethylene are by far the most widely used and preferred.

Information currently available indicates that emulsions which are suitable for the cleaning and disinfecting of textiles under commercial drycleaning plant practices should have at least 0.1 ounce (weight) of the additive present per one hundred gallons of the emulsion in order to effectively disinfect textiles which are cleaned in the compositions. Available information also indicates that the antimicrobial agent adheres to and impregnates the textiles during the cleaning procedures and is accordingly progressively removed from the emulsions with each batch of garments that are treated by the solutions. Accordingly, in plant practice the 1-(3-chloroallyl)-3,5,7-triaza-1-azoniaadamantane chloride content of the emulsions is replenished periodically on the basis of the weight of the textiles treated in the emulsions and in this respect, it has been found that one hundred gallons of an emulsion which initially contains one ounce of the 1-(3-chloroallyl)-3,5,7-triaza-1-azoniaadamantane chloride will effectively clean and disinfect nine hundred pounds of garments before the concentration of the additive diminishes to an unacceptable level for satisfactory disinfection of the further treated textiles.

Insofar as the water content of the emulsions is concerned, the amount of water need be no more than enough to dissolve effective amounts of the antimicrobial agent. In this respect, the weight of water in the emulsion should preferably exceed 45% of the weight of the 1-(3-chloroallyl)-3,5,7-triaza-1-azoniaadamantane chloride content of the emulsion in order to insure complete solution of the antimicrobial agent and its dispersement throughout the emulsion. In commercial drycleaning plant practices the drycleaning compositions usually contain from one pint to as much as a quart of water per hundred gallons of the cleaning solution because of the water content of the garments that are charged to the solutions. The amount, of course, varies in accord with existing humidity conditions but it has been found that the amount of water which exists in such compositions is more than adequate to dissolve the amounts of the antimicrobial agent and which would normally be added during the drycleaning procedures.

Insofar as the emulsifier in the composition is concerned, the emulsifier can be anyone or more of the soaps or synthetic detergents that are useful as cleaning aids or boosters in commercial drycleaning solutions and which, among other things, serve to disperse the water content of

such solutions and provide stable emulsions. Examples of suitable emulsions forming surfactants of the nonionic type are the products formed by condensing the alkalene oxides, for example, with the high molecular weight fatty alcohols, amines, carboxylic acids, amides, and alkylphenols to mention a few. Examples of the emulsion forming surfactants of the anionic type are the soaps, fatty alcohol sulfates, alkane sulfonates, alkylaryl sulfonates, succinic acid ester sulfonates, and fatty acid ester sulfonates to mention a few. The surfactants which serve as the emulsifier are used in amounts which are sufficient to provide a stable emulsion and are preferably employed in small amounts between about .5 and 2.0 percent by weight of the emulsion.

In carrying out the process aspects of the invention, the textiles are immersed and agitated in the drycleaning compositions advocated herein so as to thoroughly contact the textile with the drycleaning and disinfecting composition and the contact between the composition and the textiles is maintained for a period of time which is sufficient to destroy the vegetative forms of the pathogenic microorganism introduced with the textiles. The contact time or period will, of course, vary in accord with the type and strain of bacteria adhering to or otherwise incorporated with the textile, but even with the more resistant strains of bacteria, a contact time of fifteen minutes under the usual drycleaning conditions which exist in commercial drycleaning plant practices has been found to provide satisfactory garment disinfection. As will be subsequently seen, compositions contemplated herein have shown one hundred percent germicidal activity against *Salmonella choleraesuis* where contact has been maintained for fifteen minutes and similar activity against strains of *Staphylococcus aureus* where contact has been maintained for five minutes. Following the contact with the compositions, the textiles need only be dried in the usual manner.

Textiles treated in accord with the process are impregnated with bacteria growth inhibiting amounts of the antimicrobial agent and current information indicates that the growth of vegetative forms of such bacteria as *Staphylococcus aureus* may be inhibited in fabrics or garments containing residual amounts in excess of about .05 gram of the antimicrobial agent per pound of fabric.

EXAMPLE I

100 gallons of a basic commercial drycleaning solvent essentially consisting of perchloroethylene, 1½% (wt.) charge of detergent material, and a small amount (about 1 quart) of water emulsified therein, was modified by the addition of 1 ounce of 1-(3-chloroallyl)-3,5,7-triaza-1-azoniaadamantane chloride to provide a drycleaning and disinfecting composition for use in comparative testing of the germicidal activity of the base solvent and the additive containing emulsified composition in a 100 gallon commercial drycleaning plant.

The testing method used was that of Banville & McNeil (Applied Microbiology, vol. 14, No. 1, 1966) and the test culture was *Staphylococcus aureus* (FDA #209) grown in a nutrient broth as specified in Official Methods of Analysis, A.O.A.C.

1 cc. of nutrient broth containing 100×10⁶ organisms per cc. was absorbed in each of two 1 square inch cotton toweling swatches which were then air dried for 12 hours. One of the dried swatches was agitated for 5 minutes in 50 cc. of the base solvent while the other swatch was agitated for an identical period in 50 cc. of the disinfecting composition after the composition had been previously used on a commercial scale to clean and disinfect 200 lbs. of garments. After a 1 hour drying time, the swatches were separately pulverized in a blender and diluted to 1 liter with sterile water. 0.01 cc. samples of each diluted solution were then streaked onto nutrient agar plates in triplicate, and the plates were then incubated at 37° C. and examined after 24 hours, 48 hours and 72 hours. The

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colonies were counted at each examination with the results shown below.

Incubation period.....	Colonies		
	24 hours	48 hours	72 hours
Base solvent:			
Control plate 1.....	16.0	17.0	17.2
Control plate 2.....	20.0	20.8	21.0
Control plate 3.....	18.0	18.8	19.0
Disinfectant composition:			
Test plate 1.....	0	0	0
Test plate 2.....	0	0	0
Test plate 3.....	0	0	0

¹ Times 10⁶.

EXAMPLE II

1 ounce of 1-(3-chloroallyl)-3,5,7-triaza-1-azoniaadamantane chloride was added to 100 gallons of a basic commercial drycleaning solvent which was in use in a 100 gallon commercial size drycleaning plant and which essentially consisted of perchloroethylene, a 1½% (wt.) charge of detergent material, and a small amount (about 1 quart) of water emulsified therein to provide a drycleaning and disinfecting composition with respect to test cultures of *Staphylococcus aureus* and *Salmonella choleraesuis* grown in nutrient broth.

The official A.O.A.C. use-dilution method, modified to utilize 1 square inch cotton toweling swatch as carriers was used, with 30 swatches being used for each test and 10 swatches for each control. Procedurally 1 cc. of broth, containing 100×10⁶ organisms per cc. was placed on each swatch and the swatched air dried for 24 hours. The swatches used in the tests were agitated for 5 minutes, 10 minutes and 15 minutes in respective 200 cc. samples of the drycleaning and disinfecting composition after the composition had been previously used on a commercial scale to clean and disinfect 460 pounds of garments, whereas, the swatches used in the control runs were agitated for like periods in 200 cc. samples of the basic solvent.

All swatches were aseptically transferred to a vacuum-desiccator and after 1 hour of desiccation transferred to nutrient broth tubes and incubated at 37° C. for 48 hours. Each tube was then examined for growth with the results shown below.

	Test, 30 tubes ¹	Control runs, 10 tubes ¹
<i>Staphylococcus aureus</i> contaminated swatches: 5 minutes agitation.....	30 neg.....	10 plus.
<i>Salmonella choleraesuis</i> contaminated swatches:		
5 minutes agitation.....	21 neg.—9 plus.	Do.
10 minutes agitation.....	28 neg.—2 plus.	Do.
15 minutes agitation.....	30 neg.....	Do.

¹ Neg.=no growth; Plus=growth.

EXAMPLE III

To determine the bacterial growth inhibiting properties of textiles cleaned and disinfected in drycleaning and disinfecting compositions containing 1-(3-chloroallyl)-3,5,7-triaza-1-azoniaadamantane chloride and to determine the replenishment cycle for compositions initially containing 1 ounce of 1-(3-chloroallyl)-3,5,7-triaza-1-azoniaadamantane chloride per 100 gallons of composition, two runs were made in a 100 gallon commercial size dry-cleaning plant where the equipment was successively charged with weighed and usually approximately 20 lbs. of garments, that were agitated in the equipment for 20 minutes with the solvent composition before being removed and dried. The composition at the beginning of each run consisted essentially of perchloroethylene, a

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small amount (about 1 quart) of water, 1½% (wt.) of detergent material, and 1 ounce of 1-(3-chloroallyl)-3,5,7-triaza-1-azoniaadamantane chloride.

The method used is in Federal Specification #UU-P-510, Paragraphs 4.4.2.1–4.4.2.4 and the test culture was *Staphylococcus aureus* (F.D.A. #209), grown in nutrient broth as specified in Official Methods of Analysis, A.O.A.C.

Procedurally, and at selected intervals during each run, 1 square inch test swatches of cotton toweling were laundered in the emulsified drycleaning and disinfecting composition along with other garments. After air drying, each test swatch was placed on the surface of a pour plate that had been freshly prepared and inoculated with 0.1 cc. of the test culture. The specimens were then allowed to incubate at 37° C. for 24 hours before the zone of inhibition, as measured in millimeters from the edge of the swatch to the edge of the bacterial growth, was recorded, the results being shown below and wherein it is evident that the compositions have a satisfactorily long life before the need for replenishment of the 1-(3-chloroallyl)-3,5,7-triaza-1-azoniaadamantane chloride arises and that the swatches have bacteria growth inhibiting properties.

Total garment poundage preceding test swatch:	Test swatch zone of inhibition, mm.
First run:	
100	2
200	2
340	2
870	1
1,370	(1)
Second run:	
600	2
700	1
800	1
900	½
1,000	(1)

¹ No zone.

EXAMPLE IV

Similar results may be secured by substituting stoddard solvent for perchloroethylene in the prior examples.

While the above examples have been set forth as illustrative of the invention, it should be understood that it is intended herein to cover all such modifications as fall within the true spirit and scope of this invention.

What is claimed as new and what it is desired to secure by Letters Patent of the United States is:

1. A composition for use in drycleaning and disinfecting textiles, said composition being in the form of an emulsion of the "water-in-oil" type and consisting essentially of an organic drycleaning solvent, an aqueous solution of 1-(3-chloroallyl)-3,5,7-triaza-1-azoniaadamantane chloride colloiddally dispersed in the solvent, and an emulsifying agent in an amount sufficient to stabilize the emulsion; said solution being present in an amount ranging from about one pint to about one quart per 100 gallons of the composition and said chloride being present in an amount exceeding 0.1 ounce per 100 gallons of the composition.

2. A composition in accord with claim 1 where the solvent is stoddard solvent.

3. A composition in accord with claim 1 where the solvent is perchloroethylene.

4. A method for simultaneously drycleaning and disinfecting a textile comprising contacting the textile for a period of time in excess of five minutes with a stable emulsion consisting essentially of an organic drycleaning solvent, an aqueous solution of 1-(3-chloroallyl)-3,5,7-triaza-1-azoniaadamantane chloride colloiddally dispersed in the solvent, and an emulsifying agent in an amount sufficient to stabilize the emulsion, and thereafter removing the textile from the emulsion and drying the textile; said emulsion containing about one pint to one quart of water and said chloride being present in the emulsion in an amount exceeding 0.1 ounce per 100 gallons of the emulsion.

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5. A method in accord with claim 4 where the solvent is stoddard solvent.

6. A method in accordance with claim 4 where the solvent is perchloroethylene.

7. The method for simultaneously drycleaning and disinfecting a textile of claim 4 wherein the textile is contaminated with *Staphylococcus aureus*.

8. A composition for use in drycleaning and disinfecting textiles in accord with claim 1 where said emulsifying agent consists essentially of a mixture of anionic and non-ionic surfactants.

9. A method for simultaneously drycleaning and disinfecting textiles in accord with claim 4 wherein said emulsifying agent consists essentially of a mixture of anionic and nonionic surfactants.

10. The composition of claim 1 wherein the emulsion contains at least about one ounce per 100 gallons of emulsion of 1-(3-chloroallyl)-3,5,7-triaza-1-azoniaadamantane chloride.

11. The method of claim 4 wherein the emulsion contains at least about one ounce per 100 gallons of emulsion of 1-(3-chloroallyl)-3,5,7-triaza-1-azoniaadamantane chloride.

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