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PROCESS FOR SIMULTANEOUSLY CLEANING AND DISINFECTING TEXTILE

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5 Claims

ABSTRACT OF THE DISCLOSURE

In the process of drycleaning textiles which comprises the steps of immersing soiled textiles in a cleaning liquor based on organic solvents with a low water content for a time sufficient to remove the soil from the textile and recovering cleaned textiles, the improvement which consists adding to said cleaning liquor a condensation product of substantially equimolar amounts of formaldehyde and ethanolamine whereby the textiles are recovered disinfected as well as cleaned. Drycleaning concentrate compositions including the aforesaid condensation product are also part of the invention.

THE PRIOR ART

Washing and cleaning processes conducted in aqueous liquors generally give textiles which are not only clean but at the same time free of germs. Long treatment time at temperatures close to the boiling point and in the presence of certain chemicals contained in the detergents effect here a reliable disinfection of the material being washed. Contrary to this, the dry cleaning processes in themselves are not disinfecting. The solvents used there, such as, chlorinated hydrocarbons or heavy gasoline are, neither alone nor with the addition of the conventional cleaning intensifiers, able to effect during an acceptable treatment time a sufficient reduction of germs. For this reason the present drycleaning processes give indeed macroscopically unobjectionable textiles, but they do not lead to a microbiological sanitation. Since the cleaning liquors have to be used repeatedly there is the additional danger that germs of earlier cleaned goods will be transferred to later cleaning loads and that thus a new infection series could develop. The insufficient disinfection of textiles in such cleaning liquors is a particular problem in regard to the dry cleaning of clothes, woolen blankets and other textiles from hospitals, hotels and common lodgings.

It has already been suggested to couple the cleaning process with a disinfection step in order to improve the hygienic conditions in dry cleaning. The conventional used process done by addition of formaldehyde to the organic solvent requires, however, an after treatment in an ammonia bath because of the pungent odor which adheres to the textiles after cleaning. In practice it has, however, been found that also with such an aftertreatment an offensive odor cannot always be avoided. Further disadvantages of this process are the additional work, time and expenses of the aftertreatment and in the increased wear and tear of the textiles caused by the additional mechanical stress.

Condensation products from aldehydes and aliphatic amines or hydroxyalkylamines are known disinfectants and preservatives. For example, in German Auslegeschrift 1,148,706 the use of condensation products of aliphatic aldehydes with primary or secondary aliphatic hydroxyalkylamines is described for the disinfection and preserva-

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tion of aqueous technical solutions and emulsions, as, for example, cutting oils.

OBJECTS OF THE INVENTION

An object of the present invention is the development of a simultaneous process for the drycleaning and disinfecting of textiles in a one-step operation.

Another object of the present invention is the development in the process of drycleaning textiles, which comprises the steps of immersing soiled textiles in a cleaning liquor based on organic solvents with a low water content for a time sufficient to remove the soil from the textiles and recovering cleaned textiles, the improvement which consists adding to said cleaning liquor a condensation product of substantially equimolar amounts of formaldehyde and ethanolamine whereby the textiles are recovered disinfected as well as cleaned.

A further object of the present invention is the obtaining of a concentrate drycleaning composition for addition to organic solvent drycleaning baths to effect simultaneous cleaning and disinfecting of textiles consisting essentially of from 40% to 60% by weight of cleaning intensifiers selected from the group consisting of anionic surface active compounds and nonionic surface active compounds, from 5% to 20% by weight of the condensation product of substantially equimolar amounts of formaldehyde and ethanolamine, from 10% to 15% by weight of a lower alkanol, from 10% to 20% by weight of water, from 0% to 2% by weight of a corrosion inhibitor, and the balance to 100% by weight of an organic solvent.

These and other objects of the invention will become more apparent as the description thereof proceeds.

DESCRIPTION OF THE INVENTION

Surprisingly, it now has been found that of the above-mentioned disinfectants and preservatives, the condensation product from substantially equimolecular amounts of formaldehyde and ethanolamine gives in the organic solvent conventionally used in dry cleaning a far greater germ-reducing effect than is obtained in an aqueous media. The basis of the invention is an improved process for the simultaneous cleaning and disinfection of textiles in cleaning liquors based on organic solvents with a low water content. The new procedure is free of the above-described disadvantages of the known processes for the disinfecting drycleaning and it is characterized that to the cleaning liquors known in themselves the condensation product from substantially equimolecular amounts of formaldehyde and ethanolamine is added as a disinfectant.

The invention also resides in a concentrate drycleaning composition for addition to organic solvent drycleaning baths to effect simultaneous cleaning and disinfecting of textiles consisting essentially of from 40% to 60% by weight of cleaning intensifiers selected from the group consisting of anionic surface active compounds and nonionic surface active compounds, from 5% to 20% by weight of the condensation product of substantially equimolar amounts of formaldehyde and ethanolamine, from 10% to 15% by weight of a lower alkanol, from 10% to 20% by weight of water, from 0 to 2% by weight of a corrosion inhibitor, and the balance to 100% by weight of an organic solvent.

The condensation product from substantially equimolecular amounts of formaldehyde and ethanolamine used as a disinfectant in the process of the invention is produced by reacting formaldehyde or a precursor liberating formaldehyde in an aqueous media with ethanolamine at temperatures below 60° C. The condensation product

produced can be utilized directly in its aqueous solution or recovered therefrom by distillation. While equimolecular amounts of reactants are preferred for the reaction, as much as a 10% excess of either of the reactants may be employed. In other words, by substantially equimolecular amounts is meant the use of formaldehyde in molecular ratios of from 9 to 11 for 10 mols of ethanolamine.

Preferably in the preparation of the formaldehyde-ethanolamine condensation product disinfectant, 650 gms. (10.5 mols) of ethanolamine are added to 810 gms. of a 37% by weight aqueous formaldehyde solution (10 mols formaldehyde) under agitation and cooling. The addition of the ethanolamine is done slowly so that the temperature of the reaction mixture does not surpass 60° C. Subsequently, the agitation is continued for another hour. In this way a 52.7% by weight solution of the effective disinfectant ingredient is obtained which is ready for use. The pure condensation product can be obtained by fractional distillation from the reaction mixture. It distills at a temperature of 74° to 76° C. under a pressure of 0.01 torr (1 mm. Hg/cm.²) and has a refractive index $n_D^{20} = 1.5156$. The preparation of the formaldehyde-ethanolamine condensation product is not the subject of this application.

The condensation product from substantially equimolecular amounts of formaldehyde and ethanolamine is added to the cleaning liquors of the cleaning bath in a concentration of from 0.01 to 1.0 gm./l., preferably from 0.1 to 0.5 gm./l.

For the improvement of the cleaning effect known cleaning intensifiers based on anionic and/or nonionic surface-active agents are generally added, such as, higher molecular alkylsulfonates of a C₁₂ to C₂₂ chain length, petroleum sulfonates, alkylbenzenesulfonates whose alkyl chains have 8 to 14 carbon atoms, preferably dodecylbenzenesulfonate, also fatty alcohol sulfates having a fatty chain of from 8 to 22 carbon atoms, fatty acid alkanolamides having a fatty acid chain of from 8 to 22 carbon atoms, ethyleneoxide and propyleneoxide adducts of higher molecular compounds with an active hydrogen, such as, fatty alcohols of a C₁₂ to C₁₈ chain length, fatty amides of a C₁₂ to C₁₈ chain length, alkylphenols having an alkyl chain of 8 to 14 carbon atoms, preferably nonylphenols, and the like.

The cleaning intensifiers are usually added to the cleaning liquors in the form of concentrates which contain, in addition to the surface-active compound, solvents such as chlorinated hydrocarbons or mineral oil as well as, if necessary, a dissolving aid such as a lower alkanol, for example isopropanol, and water. The condensation product from formaldehyde and ethanolamine can be added to these concentrates and can be measured into the organic solvent cleaning bath with the cleaning intensifiers.

Preferably the concentrate drycleaning composition for addition to organic solvent drycleaning baths to effect simultaneous cleaning and disinfecting of textiles consists essentially of from 40% to 60% by weight of cleaning intensifiers selected from the group consisting of anionic surface active compounds and nonionic surface active compounds, from 5% to 20% by weight of the condensation product of substantially equimolar amounts of formaldehyde and ethanolamine, from 10% to 15% by weight of a lower alkanol, from 10% to 20% by weight of water, from 0 to 2% by weight of a corrosion inhibitor, and the balance to 100% by weight of an organic solvent.

It is also preferable in the carrying out of the cleaning process of the invention to add a sufficient amount of water to the cleaning liquors so that during the cleaning procedure the relative humidity in the vapor phase above the liquor is at least 70%.

In certain cases it has proven advantageous to add corrosion inhibitors to the cleaning liquors, for example, compounds from the series of benzothiazoles, in order to prevent an attack of the liquor upon the vessel material

of the cleaning equipment. If desired, the inhibitors can be mixed into the cleaning intensifier concentrates.

The cleaning liquors used contain as the main ingredient the conventional drycleaning organic solvents and solvent mixtures, such as, chlorinated hydrocarbons and gasoline. Especially suitable as solvents are perchloroethylene, heavy gasoline with a boiling point in the range between 140° to 200° C. and monofluorotrichloromethane.

In practice, the cleaning of the textiles is done in the commercial machines for drycleaning.

The textiles cleaned according to the invention process with the use of the condensation product of formaldehyde and ethanolamine as disinfectant, are odorless and show no discoloration. The disinfection effected during the process is as good against pathogenic bacteria as against yeasts and fungi.

A considerable advantage of the formaldehyde-ethanolamine condensation product, as used as a disinfectant, is the lack of any skin irritation. The skin tolerance of the product was tested in comparison to a 37% by weight formalin solution. Once daily, a 52.7% by weight solution of the formaldehyde-ethanolamine condensation product and a 37% by weight formaldehyde solution were applied to dehaired mice by brushing. The formaldehyde solution caused, after only two days, a strong necrotic change in the skin while the condensation product from formaldehyde and ethanolamine was tolerated without irritation during a test period of 14 days.

The following examples are illustrative of the practice of the invention without being limitative in any respect. The examples demonstrate the increased microbiological activity of the condensation product from substantially equimolecular amounts of formaldehyde and ethanolamine in organic solvents in comparison to its effectiveness in an aqueous solution, as well as the practical carrying out of the process of the invention.

Example 1.—Laboratory experiments

(A) Bactericidal effectiveness in perchloroethylene.—1 cm.² large sterile wool patches (weight 550 gm./m.²) were infected with suspensions of *Staphylococcus aureus* SG 511 (10¹⁰ germs/ml.), *Pseudomonas aeruginosa* (10¹⁰ germs/ml.) and *Candida albicans* (10⁷ germs/ml.) and subsequently dried at 30° C.

The disinfectant experiments were conducted in perchloroethylene liquors which contained either (a) 5 gm./l. of a commercial cleaning intensifier based on a petroleum sulfonate and nonylphenol polyglycoether, or (b) 5 gm./l. of the same cleaning intensifier and 0.3 gm./l. of the above described formaldehyde-ethanolamine condensation product. The infected patches were placed in the perchloroethylene liquors held at 20° C., and agitated therein for 10 or 15 minutes with the aid of a magnetic stirrer. Subsequently, the patches were rinsed twice with a 0.1% by weight sodium sulfite solution in order to avoid a possible after-inhibition effect. One part of the patches infected with bacteria was placed into a Merck-Standard-I-Bouillon (small tube test), the other part spread on a Merck-Standard-I-Bouillon-Agar and covered with a layer of the same culture medium (Plate test). With the patches infected with *Candida albicans* the small tube test was done with wort, the plate test with wort agar. To avoid a bacteriostatic after-effect the liquid and solid culture media each contained 0.5% per weight of histidine. The small tubes and plates were incubated for 8 days at 37° C. and subsequently macroscopically examined for growth. The small tube test results were checked by smears on solid culture media. The results obtained with the perchloroethylene liquors (a) and (b) are reproduced in Table I. This table demonstrates that with an addition of 0.3 gm./l. of the formaldehyde-ethanolamine condensation

product a duration of 10 minutes is already sufficient completely to inhibit a germ growth after the treatment.

TABLE I

Bactericidal effectiveness of the condensation product from equimolecular amounts of formaldehyde and ethanolamine in a perchloroethylene liquor

Concentration of the effective ingredient:
liquor (a) 0 gm./l.;
liquor (b) 0.3 gm./l.

Test germ	Liquor	Reaction time			
		10		15	
		Plate	Small tubes	Plate	Small tubes
<i>Staphylococcus aureus</i>	(a)	+	+	+	+
	(b)	+	+	+	+
<i>Pseudomonas aeruginosa</i>	(a)	+	+	+	+
	(b)	+	+	+	+
<i>Candida albicans</i>	(a)	+	+	+	+
	(b)	-	-	-	-

NOTE: +=Growth; -=Sterile.

(B) Bactericidal effectiveness in aqueous solution.— The determination of the bactericidal effectiveness in aqueous liquor was done with solutions of the formaldehyde-ethanolamine condensation products in various concentrations. All solutions contained 5 gm./l. of the above-mentioned commercial cleaning intensifiers. As test germs *Staphylococcus aureus* SG 511 was used.

The wool patches were infected in the same way as in the experiments conducted with the perchloroethylene liquors. They were spread in small Petri dishes and 10 ml. of the test solutions were poured over each dish. After the selected exposure times of 5, 10, 15, 20, 30, 45 and 60 minutes, patches were taken out and rinsed twice with a 0.1% by weight sodium sulfite solution. Subsequently, the patches were put into Merck-Standard-I-Bouillon or on Merck-Standard-I-Bouillon-Agar and covered with the same culture medium. The culture media always contained 0.5% of histidine for the avoidance of a possibly after-inhibition effect.

construction (Böwe R 9). In four charges, each time 6 to 8 kg. of woolen blankets were cleaned, to which in muslin bags were attached containing infected wool patches of 1 cm.² size. The cleaning was done in a three-stage, single-bath process with 100 l. perchloroethylene as cleaning agent. In the first charge, the cleaning liquor contained only 5 gm./l. of a commercial cleaning intensifier and no disinfectant. To the same liquor were added for the second charge 0.3 gm./l. of the formaldehyde-ethanolamine condensation product described above and sufficient water so that the relative humidity in the vapor phase above the liquor was higher than 70%. In order to prevent a depletion of effective material in the liquor, at each successive machine loading, another 0.1 gm./l. of disinfectant was added. Subsequently, the textiles were centrifuged for 2 minutes and then dried for 15 minutes at 60° C.

As in the laboratory experiments, the wool patches were rinsed twice with 0.1% by weight of sodium sulfite solution, brought into the nutrient solution or spread on the culture media and covered with the media, incubated for 8 days at 37° C. and then examined for sterility. The liquid and solid culture media contained again 4.5% histidine.

As test germs in these experiments the following micro-organisms were used:

- (1) *Staphylococcus aureus* SG 511
- (2) *Escherichia coli*
- (3) *Pseudomonas aeruginosa*
- (4) *Mycobacterium phlei*
- (5) *Candida albicans* and
- (6) *Trichophyton mentagrophytes*

The results found are summarized in Table 3.

The same disinfection results are obtained if the cleaning procedure is conducted with heavy gasoline (B.P. 140–200° C.) or monofluorotrichloromethane as solvents.

TABLE 3

Microbicidal effect of the condensation product from equimolecular amounts of formaldehyde and ethanolamine with dry cleaning in a perchloroethylene liquor

Concentration of the effective ingredient:

Charge I—0 gm./l.
Charge II—0.3 gm./l.
Charge III—Addition to 0.1 gm./l.
Charge IV—Addition 0.1 gm./l.

Test Germ	Charge I		Charge II		Charge III		Charge IV	
	Plate	Small tube	Plate	Small tube	Plate	Small tube	Plate	Small tube
<i>Staphylococcus aureus</i>	+	+	-	-	-	-	-	-
<i>Escherichia coli</i>	+	+	-	-	-	-	-	-
<i>Pseudomonas aeruginosa</i>	+	+	-	-	-	-	-	-
<i>Mycobacterium phlei</i>	+	+	-	-	-	-	-	-
<i>Candida albicans</i>	+	+	-	-	-	-	-	-
<i>Trichophyton mentagrophytes</i>	+	+	-	-	-	-	-	-

NOTE: +=Growth; -=Sterile.

The small tubes and plates were incubated for 8 days at 37° C. and then examined macroscopically for growth. The results of the small tube tests were checked by smears on solid culture media. The results reproduced in Table 2 show that even at a concentration of the effective ingredient of 30 gm./l. and a treatment time of 60 minutes the test germs were not killed.

TABLE 2

Bactericidal effectiveness of the condensation product from equimolecular amounts of formaldehyde and ethanolamine in aqueous liquor

Test germ: *Staphylococcus aureus*

Concentration gm./l.	Exposure time (minutes)						
	5	10	15	20	30	45	60
20.....	+	+	+	+	+	+	+
25.....	+	+	+	+	+	+	+
30.....	+	+	+	+	+	+	+

NOTE: +=Growth.

Example 2.—With drycleaning equipment

The examples of actual practice of the invention were conducted in a drycleaning equipment of conventional

These results demonstrate the effectiveness of the simultaneous cleaning and disinfecting of the process of the invention using from 0.01 to 1.0 gm./l. of the condensation product of substantially equimolecular amounts of formaldehyde and ethanolamine as disinfectant in the conventional drycleaning baths based on organic solvents with a low water content.

The preceding specific embodiments are illustrative of the practice of the invention. It is to be understood, however, that other expedients known to those skilled in the art may be employed without departing from the spirit of the invention or the scope of the appended claims.

We claim:

1. In the process of drycleaning textiles which comprises the steps of immersing soiled textiles in a cleaning liquor based on drycleaning organic solvents with a sufficient water content so that the relative humidity in the vapor phase above said cleaning bath liquor is at least 70%, for a time sufficient to remove the soil from the textiles and recovering cleaned textiles, the improvement which consists of adding to said cleaning liquor

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from 0.01 to 1.0 gm./l. of a condensation product of substantially equimolar amounts of formaldehyde and ethanolamine whereby the textiles are recovered disinfected as well as cleaned.

2. The process of claim 1 wherein said concentration of said condensation product in said cleaning liquor is from 0.1 to 0.5 gm./l.

3. The process of claim 1 wherein said cleaning liquor contains drycleaning intensifiers selected from the group consisting of alkylsulfonates of a C₁₂ to C₂₂ chain length, petroleum sulfonates, alkylbenzenesulfonates having alkyls with 8 to 14 carbon atoms, fatty alcohol sulfates having 8 to 22 carbon atoms, fatty acid alkanolamides having a fatty acid of from 8 to 22 carbon atoms, ethylene oxide and propylene oxide adducts of fatty alcohols of a C₁₂ to C₁₈ chain length, ethylene oxide and propylene oxide adducts of fatty amides of a C₁₂ to C₁₈ chain length and ethylene oxide and propylene oxide adducts of alkylphenols having an alkyl of 8 to 14 carbon atoms.

4. The process of claim 1 wherein said organic solvent is selected from the group consisting of perchloroethylene,

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heavy gasoline with a boiling range between 140° C. and 200° C., and monofluorotrichloromethane.

5. The process of claim 1 wherein said condensation product is the condensation product of 9 to 11 mols of formaldehyde with 10 mols of ethanolamine.

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MAYER WEINBLATT, Primary Examiner

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