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METHOD OF REMOVING VESICANT PARTICLES

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This invention described herein may be manufactured and used by or for the Government for government purposes, without the payment to us of any royalty thereon.

This invention relates in general to protective clothing and more particularly has reference to a process of treating material for rendering it resistant to vesicant gases and vapors, and to the product resulting from the treatment.

Previous to this time, it has been attempted to fix chlorine on material for protecting against such gases as mustard, but the impregnated cloth has increased in weight and has been characterized by stickiness. Also, the permeability of the same has often been lowered to such an extent as to make clothing made of material so treated, uncomfortable to the wearer.

A primary object of this invention is to provide material relatively impermeable to vesicant gases or vapors.

Another object of this invention is to provide material having semi-permeable properties, that is, material which is permeable to air and inert gases, but impermeable to mustard gases and similar substances.

Another object of this invention is to devise a process for producing material having properties rendering the same impermeable to mustard gases and substances having similar properties.

Yet another object of this invention is to provide a process for fixing chlorine on textile material without appreciably affecting the desirable permeable characteristics thereof.

A further object of this invention is to provide a process for chlorinating material to render the same impervious to mustard gas and similar substances without materially increasing the weight of the material.

Still another object of this invention is to devise a process of treating clothing to render the same impervious to vesicant and toxic gases or vapors, such as mustard gas, lewisite and similar substances.

A still further object of this invention is to devise a process for fixing chlorine in material to render the same impervious to mustard gas and similar substances without appreciably changing the desirable physical characteristics thereof.

With these and other objects in view, the invention consists in the ingredients and steps to be hereinafter set forth and claimed with the understanding that the several necessary substances and compounds employed in carrying out the invention and the steps involved in the necessary procedure, may be widely varied without departing from the spirit of the invention or the scope of the appended claims.

The present invention consists briefly in directly fixing chlorine by a chemical combination on material having the characteristics of wool, so as to render the same impervious to mustard gas and similar vesicant and toxic gases. In preparing the material, wool or similar substances are chlorinated by subjecting them to treatment with hypochlorous acid, acidified hypochlorite salts or organic hypochlorites.

This invention is based on the fact that active chlorine in the form of free chlorine, hypochlorite, chloramine, or chloramide will react with mustard gas, or lewisite to form non-vesicant compounds, or compounds which are much less vesicant than mustard gas or lewisite.

Wool is considered to be composed of a protein substance, known as keratin. It has been found that, when wool is subjected to the action of chlorine, some sort of a reaction takes place between the chlorine and the wool. It has been stated that the reaction between the chlorine and the wool produces chloramines. However, it has been found that in some instances no chloramines have been formed. Regardless of the theory of the reaction which takes place between the wool and the chlorinating agent, there is a change effected in the characteristic of the wool which is desirable for certain purposes, among which is that of rendering mustard gas and similar substances non-vesicant.

We have found that, by chlorinating wool according to the various methods herein set forth, a product is obtained which is impervious to mustard gases and similar compounds employed in chemical warfare. In the processes as hereinafter set forth, we have succeeded in directly fixing active chlorine to wool by a chemical combination.

In developing the present invention, we discovered that active chlorine may be directly fixed in wool to render the same impervious to mustard gases and similar chemical compounds by three distinct types of reagents.

These chlorinating reagents are:

1. Organic hypochlorites
2. Acidified hypochlorite salts
3. Hypochlorous acid

The first of the above mentioned reagents appears to be the most satisfactory for chlorinating wool and similar material to render the same impervious to mustard gases and compounds having corresponding vesicant properties. By subjecting the wool or other material to the action of an organic hypochlorite, such as tertiary butyl hypochlorite under proper conditions, a relatively large percentage of chlorine may be incorporated therein.

Chlorination of the wool may be effected by merely subjecting it to the action of the tertiary alkyl hypochlorite for a definite period of time. It has been found, however, that the speed of the chlorinating action may be controlled by regulating the moisture content of the wool or other material under treatment prior to the chlorinating reaction.

The moisture content may be regulated by allowing the moisture in the material to come to equilibrium with an atmosphere having a controlled relative humidity. This may be accomplished by suspending the material in a chamber containing an atmosphere of a definite humidity or by subjecting the material to a moisture saturated atmosphere for a definite time. Of course, any other method of regulating the moisture content may be employed. Within certain limits, the speed of the chlorinating reaction is increased when material having a high moisture content is treated and is decreased when material having a low moisture content is treated.

The following specific examples are set forth as illustrative of the effect that the moisture content of the wool or other material undergoing treatment has on the speed and extent of a chlorinating reaction.

Relative Humidity	Time	Temperature	Active Chlorine in the Wool
Per cent	*Hours*	°*C.*	*Per cent*
67	2	40	2.4
81	½	40	1.6
81	1½	40	5.3

It will be seen from the foregoing tabulation that by controlling the moisture content of the wool or other material, as high as 5% of active chlorine may be introduced therein by subsequently subjecting it to a tertiary alkyl hypochlorite under the conditions set forth in the above tabulation. This may be accomplished without causing any appreciable damage to the material. The values given in the above tabulation were obtained by treating woolen material with tertiary butyl hypochlorite.

In one example upon which the above tabulation is based, the moisture in the woolen material was allowed to come to equilibrium with an atmosphere of a controlled relative humidity of 81% at 25° C. After this treatment, the material having a definite moisture content was immersed in a tertiary butyl hypochlorite maintained at a temperature of 40° C. The material was subjected to the chlorinating agent for about 1½ hours and subsequently to the chlorinating treatment was washed with carbon tetrachloride. In this instance, approximately 5% of active chlorine was fixed in the material. Practically identical results may be obtained by employing tertiary amyl hypochlorite as the chlorinating reagent.

It has been found advantageous to first impregnate the material to be treated with an acid inhibiting buffer. Sodium acetate serves adequately for this purpose, but we wish it to be clearly understood that other substances may be employed as found convenient. This treatment accelerates the subsequent chlorinating reaction and allows a greater amount of chlorine to be introduced into the wool or similar material without any damage thereto.

In carrying out the process involving the use of an acid inhibiting buffer, the wool or other material under treatment may be immersed in a solution of sodium acetate or other acid inhibiting buffer to incorporate the same in the material. We have found that by treating the material with a 1% solution of sodium acetate and incorporating the solution therein to the extent of 1½ times the weight of material gives satisfactory results. Any suitable methods for regulating the quantity of solution in the material may be employed. After subjecting the material to the solution of the acid inhibiting buffer, the material is dried, leaving a definite amount of the acid inhibiting buffer in the material, depending upon the concentration of the solution and the quantity of the solution taken up by the material.

Good results have been obtained by impregnating the material with about 1½% by weight of acid inhibiting buffer. While specific quantities have been mentioned above, we wish it to be clearly understood that they are given merely by way of example, and that varied quantities of the acid buffer may be employed.

After incorporating the buffer in the material to be treated, the moisture content of the dry impregnated wool may then be regulated by allowing it to come to equilibrium with the moisture in an atmosphere having a predetermined relative humidity. When a definite predetermined moisture content is established in the material, it may be immersed in a solution of tertiary butyl hypochlorite maintained at a temperature effective for chlorinating for a predetermined time. For instance, it has been found that chlorination suitable for rendering the material impervious to mustard gas and similar vesicant compounds may be effected by establishing a moisture content in the material effected by subjecting it to an atmosphere having a relative humidity of approximately 81% at 25° C., and immersing the conditioned material in a solution of tertiary butyl hypochlorite at a temperature approximating 34° C., for a period of 4 hours.

After chlorination, the wool may be washed with carbon tetrachloride to remove the excess tertiary butyl hypochlorite. It has been found that by treating wool or other materials as above set forth, as high as 8% of active chlorine (based on the weight of the wool) may be fixed in the material without damaging the same.

We wish it to be clearly understood that the conditions of chlorination may be varied over a relatively wide range. For instance, the reaction may be controlled by fixing the moisture content of the material prior to treatment and the chlorinating reaction may be further controlled by varying the time and/or temperature of the chlorinating treatment. Control of the chlorinating treatment may also be effected by diluting the tertiary alkyl hypochlorite with carbon tetrachloride or other relatively inert solvents. While in describing the process of chlorinating wool and similar materials, specific mention has been made of tertiary butyl hypochlorite, we wish it to be clearly understood that tertiary amyl hypochlorite may be just as effectively employed as tertiary butyl hypochlorite.

Another method of chlorinating wool and similar substances to impart characteristics thereto desirable for protecting materials against mustard gas and similar substances, is to subject the material to an acidified sodium hypochlorite solution. For instance, a sodium hypochlorite solution, acidified with glacial acetic acid may be employed for fixing as high as 3% of active chlorine in the material without damaging the same.

Of course, acidified solutions of other hypochlorite salts may be employed. For instance, other alkali metal salts of hypochlorous acid may be employed.

The following specific example will serve as an illustration of the procedure, but it is intended that the invention not be limited thereby, since results can be obtained by widely varying the specific compounds employed and the quantities used in carrying out the following procedure. Twelve grams of the cloth to be treated may be immersed in 1457 cc. of water, to which 240 cc. of sodium hypochlorite solution (containing 11.7% of active chlorine and 3.5% of free sodium hydroxide) is added. To this solution, 63 cc. of glacial acetic acid is immediately added while the solution is agitated. Agitation is maintained and the temperature of the solution is held substantially constant at approximately 23° C. The cloth may be allowed to remain in the solution under the above conditions for about 1¼ minutes. After subjecting the material to this treatment, it is then thoroughly washed with water. It has been found that about 3% of active chlorine may be fixed in the wool by the above procedure.

It has also been found that wool and similar materials may be chlorinated to render them substantially impervious to mustard gas and corresponding substances by subjecting the material to a pure hypochlorous acid solution. The solution employed may be either pure hypochlorous acid or hypochlorous acid prepared by the action of chlorine on water in the presence of calcium carbonate. By subjecting the wool or other material to treatment with hypochlorous acid as set forth above, we have succeeded in fixing about 3% of active chlorine in the material without appreciable damage.

It is also possible to fix active chlorine in wool and similar substances by means of free chlorine without material damage to the substance. In this instance, however, only relatively small amounts of chlorine may be fixed to the material without damaging the same. We have succeeded in fixing approximately .5% of chlorine in the material by the preceding method.

In the foregoing description, we have illustrated the invention with reference to treatment of the wool or similar material by subjecting it to the action of particular compounds and conditions. It should be clearly understood, however, that desirable and effective results may be obtained by employing equivalent compounds and by varying the condition of the material undergoing treatment, and the treating conditions over relatively wide ranges.

Material such as wool, when treated according to the methods herein set forth, serves very efficiently for protecting against mustard gases and corresponding vesicant gases. While the treated material having chlorine fixed therein is relatively impermeable or impervious to mustard gas, lewisite and similar substances, its permeability to air and gases unaffected by the chlorine content of the cloth is practically unaffected. The material acts in the nature of a semi-permeable membrane.

Desirability of this feature is obvious in that clothing made of the treated material may be worn with considerable more comfort than clothing which is impervious to all gases.

Vapors of mustard gas or corresponding vesicant compounds, upon contacting with the treated material, react with the chlorine fixed in the material to produce non-vesicant reaction products. It is believed that the reaction takes place according to the ratio of one molecule of chlorine to one molecule of mustard gas.

In the foregoing description, the processes and product have been mentioned in connection with wool and similar materials. The process is, of course, applicable to other fibres of animal and other origins.

While we have described the preferred embodiments of our invention, we wish it to be understood that we do not confine ourselves to the precise steps or ingredients set forth herein by way of illustration, as it is apparent that many changes and variations may be made therein by those skilled in the art, without departing from the spirit of the invention or exceeding the scope of the appended claims.

We claim:

1. A method of removing vesicant particles from vesicant contaminated air which comprises passing the air through woolen material having active chlorine fixed thereon by chemical combination.

2. A method of removing vesicant particles from vesicant contaminated air which comprises passing the air through woolen material which has been treated with a compound from the group consisting of organic hypochlorite, hypochlorite salts and hypochlorous acid.

3. A method, according to claim 2, in which the material has been treated with an organic hypochlorite from the group consisting of tertiary butyl hypochlorite and tertiary amyl hypochlorite.

4. A method according to claim 2, in which the material has been treated with tertiary butyl hypochlorite.

5. A method according to claim 2, in which the material has been treated with tertiary amyl hypochlorite.

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