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2,911,322

FIELD TREATMENT FOR MAKING CELLULOSIC FABRICS FLAMEPROOF AND RESISTANT TO CERTAIN CHEMICAL WARFARE GASES

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

This invention relates to a combination of treatments which render cellulosic materials resistant to the propagation of flame and to the passage of certain of the chemical warfare gases. In particular this invention provides cotton textiles impregnated in the field with two separate treatments which render them resistant to burning and to certain warfare gases.

Copending patent application Serial No. 693,212, filed on Oct. 29, 1957, describes a combined treatment for textile materials which renders them resistant to burning and to the passage of certain chemical warfare gases. The present invention provides a process for producing such a combined treatment on the textile materials which may be employed by troops in the field.

Heretofore many compositions and treatments have been developed to render textile materials flameproof. Similarly, treatments have been devised to prevent the passage of certain chemical warfare gases through these textile fabrics. However, none of the flameproof treatments which give a permanent finish can be applied in the field, and the known treatments which can be applied in the field involve ionic materials which cannot be used as a substrate for activated carbon gas adsorbent.

A primary object of this invention is to provide a field treatment for textile clothing material such as cotton sateen which will provide an air permeable cloth suitable for the usual textile applications and sufficiently adsorptive or destructive of certain chemical warfare gases or liquid agents to protect a person covered by the treated textile and permit him to find shelter from the gas or other agents. An additional object is to provide this protection against such gases under conditions which would normally result in burning of the fabric, such as would be the case under attack by incendiary devices.

The flameproofing agents utilized in this combined treatment comprise those polymeric flameproofers for cellulose which result from the reaction of tetrakis (hydroxymethyl) phosphonium chloride with methylol compounds such as the methylol melamines, methylol urea, etc. in the presence of acidic catalysts.

The gas-proofing agents may be any of a large number of such materials which have been developed for resistance to chemical warfare gases. They may protect by undergoing chemical reaction with the attacking gas and so converting it to a harmless derivative or they may adsorb the gas and prevent it from penetrating the treated fabric.

The combined treatment described has been designed for use with cellulosic materials, such as fabrics made from cotton, rayon, linen, jute, and hemp; they may be employed with some success to animal fibers such as silk and wool.

The essential part of the flameproofing step of this

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process appears to be the spontaneous decomposition of the chosen catalyst to give acidic substances which bring about the reaction between the tetrakis-(hydroxymethyl) phosphonium chloride and methylol compounds. When the tetrakis-(hydroxymethyl) phosphonium chloride resin mixtures are prepared as prescribed by the literature (W. A. Reeves and J. D. Guthrie, Textile World, vol. 104, No. 2, page 101, February 1954), applied to fabrics by dripping and pressing between rollers, and air dried, the resulting polymer is not wash fast, and does not make the fabric flameproof. A high temperature curing step, which is impractical for use by troops in the field, is necessary to secure a satisfactory treatment as disclosed in the cited article. But, when the non-volatile urea and triethanolamine are replaced with ammonium hydroxide as the acid acceptor, the air-dry treated fabric, after rinsing in water to remove ionic materials, still retains sufficient flameproofing resin to allow the sample to pass the standard vertical flame test. It is speculated that the spontaneous decomposition of the tetrakis (hydroxymethyl) phosphonium chloride to liberate HCl and the evaporation of the excess ammonia operate to produce a condition of sufficiently low pH to bring about the polymerization reaction between tetrakis (hydroxymethyl) phosphonium chloride and the methylol compound.

The fabric so treated with the flameproofing agent may then be further treated by depositing on the surface of the treated fabric a reagent which either adsorbs or reacts with the chemical warfare gas so that its function is not hindered by the presence of other deposited material. Cotton fabrics treated in the proper manner have acceptable char lengths when treated with the standard vertical flame test and although permeable to air will prevent the passage of chemical warfare gases, e.g., chloropicrin, for acceptable lengths of time. This protective agent is chosen from a group of such materials which are known to resist the passage of specific chemical warfare agents by virtue of either adsorbing or reacting with them. For instance, activated charcoal suspended in a suitable binder may be applied for the purpose of adsorbing chloropicrin and high molecular weight organic gases or liquids in general, or N,N dichloro-bis (2,4,6 trichlorophenyl) urea may be applied in order to neutralize mustard gas by reacting with it.

It should be noted that these materials do not form an impervious coating on the surface of the fabric, but rather impregnate the thread, where they serve to adsorb or neutralize the chemical warfare agents while permitting the passage of air through the interstices. Other specific treatments may be applied as the need arises.

The following examples are illustrative of the details of the method of practicing the invention.

Example 1

As prescribed in the literature (W. A. Reeves and J. D. Guthrie, Textile World, vol. 104, No. 2, February 1954), an aqueous solution was made containing 16% tetrakis (hydroxymethyl) phosphonium chloride, 9% trimethylolmelamine, 10% urea and 3% triethanolamine. Samples of cotton sateen were padded to an 80% wet pickup and allowed to air dry overnight to give about a 20% dry pickup.

One of these samples was worked in a suspension containing: 1% wetting agent (Daxad 11, a water soluble sodium salt of a sulfonated naphthalene-formaldehyde condensate), 3.5% suspending agent (Elchem 1273, a vinylacetate-crotonic acid co-polymer), 27% activated carbon and 68.5% water. After working, the sample was padded to about 60% wet pickup and air dried. The finished sample was flameproof when tested by the stand-

ard vertical flame test but resisted the passage of chloropicrin for only 1.5 minutes.

When a similar sample of the flameproof sateen was rinsed in water to remove contaminating ionic materials, and then treated with the carbon suspension, the gas adsorption was improved but the sample was no longer flameproof.

Example 2

An aqueous solution was made containing 16% tetrakis (hydroxymethyl) phosphonium chloride, 10% methylolmelamine and 1.5% aqueous ammonia (28% NH_3). The solution had pH of 8.5. Samples of cotton sateen were worked, padded, and allowed to air dry under conditions which gave a dry pickup of about 25%. The samples were rinsed in 100 parts of water for every part of fabric and allowed to air dry. The dried weight showed a gain of 17.5%. These samples were then treated with carbon suspension described in Example 1, and allowed to air dry. The pickup of the carbon suspension was calculated to be equal to 17.8% of the original weight of the fabric. The finished sample was flameproof when tested with the standard vertical flame test and resisted the passage of chloropicrin for 12.2 minutes.

While the above examples show the details of the application of only one gas-proofing treatment, it will be understood that other similar treatments can be employed. For example, stabilized N,N dichloro-bis (2,4,6 trichlorophenyl) urea may be applied as described in Example 3 of our application, Serial No. 693,212, filed of even date herein and referred to above. Moreover, there is disclosed in application Serial No. 585,583, filed May 17, 1956, now Patent No. 2,875,167, by Francis W. Minor et al., a process for impregnating fabric with activated carbon which comprises treating the fabric with an aqueous suspension of activated carbon, polymeric carboxylic acid, preferably a copolymer of monosecondarybutyl maleate and styrene, and an emulsified polymer,

preferably a butadiene-styrene copolymer latex. This treatment can be used instead of the treatment with the carbon suspension described in Examples 1 and 2.

We claim:

1. A process suitable for use by troops in the field for rendering a cellulosic fabric flameproof, which comprises impregnating said fabric with an aqueous solution consisting essentially of tetrakis (hydroxymethyl) phosphonium chloride, a methylol melamine, ammonium hydroxide, and water, rinsing said impregnated fabric with water, and air drying said impregnated fabric.

2. A process as defined in claim 1 and further comprising impregnating said flameproofed fabric, following said air drying step with a second material which renders said fabric resistant to chemical warfare agents, said second material comprising an active agent selected from the group consisting of activated carbon and N,N dichloro bis (2,4,6 trichlorophenyl) urea.

3. The process of claim 1 wherein the cellulosic material is cotton.

4. The process of claim 1 wherein said second material comprises activated carbon.

5. A process suitable for use by troops in the field, for rendering fabric flameproof and resistant to chemical warfare agents comprising impregnating said fabric with a solution consisting essentially of tetrakis (hydroxymethyl) phosphonium chloride, a methylol melamine, and ammonium hydroxide, air drying said fabric, thereafter impregnating said fabric in an aqueous suspension comprising a vinyl acetate-crotonic acid copolymer and activated carbon, and air drying said fabric.

References Cited in the file of this patent

UNITED STATES PATENTS

2,810,701 Reeves et al. Oct. 22, 1957

FOREIGN PATENTS

575,379 Great Britain May 21, 1940