

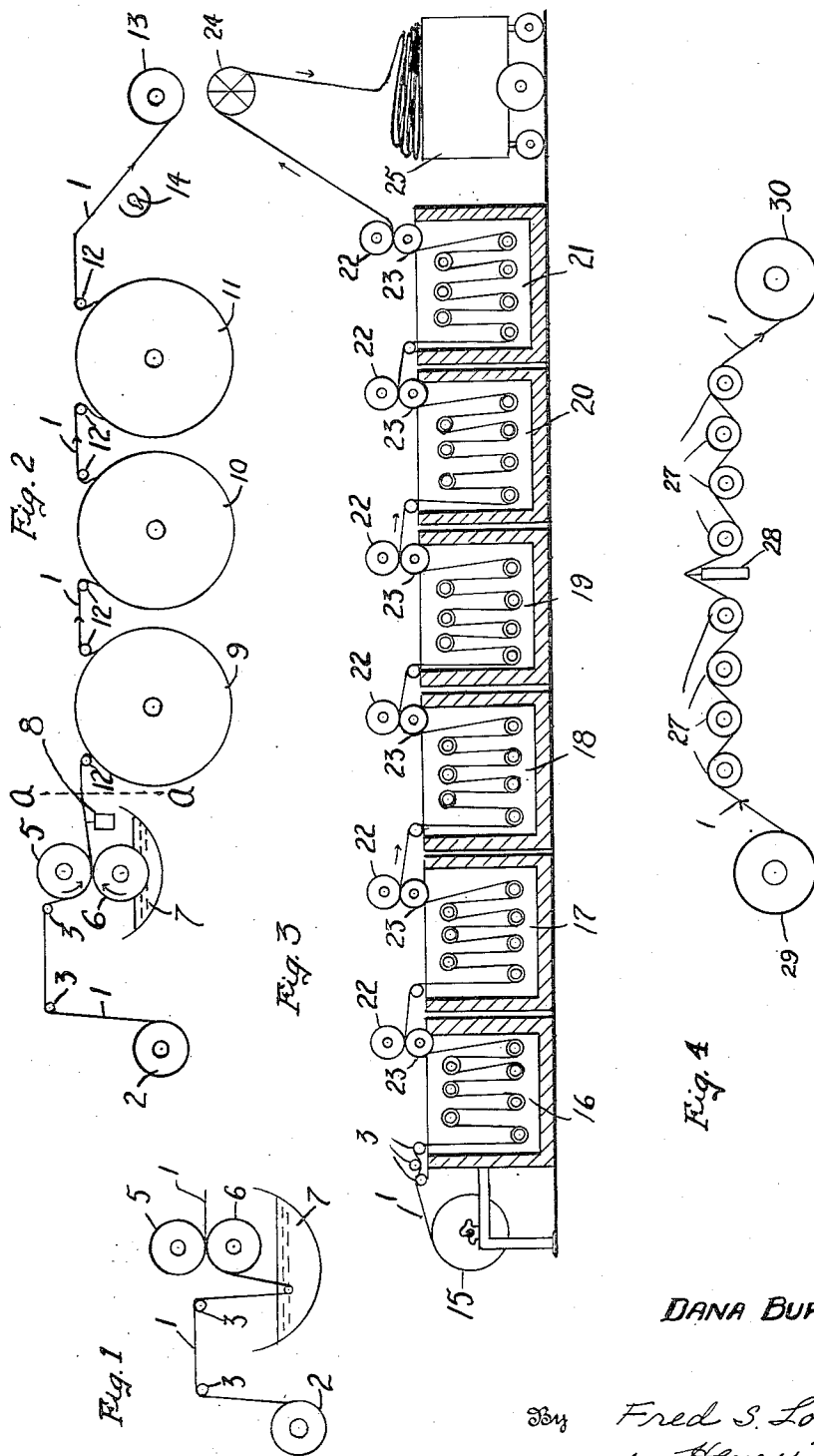
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D. BURKS, JR

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ANTIVESICANT FILTER FABRIC

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Inventor
DANA BURKS, JR.

By Fred S. Lockwood
and Henry Berk Attorneys

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ANTIVESICANT FILTER FABRIC

Dana Burks, Jr., Walpole, Mass., assignor, by mesne assignments, to the United States of America as represented by the Secretary of War

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1 Claim. (Cl. 117—143)

The invention described herein may be manufactured and used by or for the Government, for governmental purposes, without the payment to me of any royalty thereon.

This invention relates to protective fabrics and process of producing same. This invention has for an object the provision of a flexible and air permeable fabric which is resistant to penetration by persistent chemical warfare agents such as mustard gas.

The persistent chemical warfare agents, e. g. mustard gas, will readily penetrate ordinary fabrics so that serious burns may be produced on the skin of a person wearing clothing constructed from such fabrics. Several methods have been proposed in the past for treating fabrics to render them resistant to penetration by persistent chemical warfare agents. However, the fabrics and processes that have been proposed in the past have not been entirely satisfactory. For example, certain chemicals containing active chlorine have been used to impregnate fabrics to render them resistant to penetration by certain vesicants. However, the tensile and tear strength of the fabric is often adversely affected thereby and the protective value of the fabric is markedly reduced after the fabric is laundered or dry cleaned several times. In the case of fabrics impregnated with chemicals of this character, it is necessary that they be reimpregnated after washing or cleaning. It has also been proposed that a fabric be treated with activated carbon in association with a binder such as rubber latex or a formaldehyde-hardened casein composition. The use of rubber latex, however, is not satisfactory for the reason that the resulting fabric is unduly weighted and is substantially impervious to air. This is a disadvantage when the fabric is used in the manufacture of garments; also, the rubber degrades on storage and tends to poison the carbon. The use of a formaldehyde-casein binder for the activated charcoal is not satisfactory for the reason that the resulting fabric has a marked tendency to crock and the carbon is washed out of the fabric during washing or dry cleaning operations.

Accordingly, it is one object of this invention to provide a fabric resistant to penetration by vesicants which is capable of being laundered or dry cleaned a number of times without substantial loss of the protective action of the fabric.

An additional object of this invention is the provision of a fabric resistant to penetration by vesicants which does not have a tendency to crock and which will retain its protective properties for a long period of time.

An additional object of this invention is the provision of a means for providing a fabric with vesicant-penetration resisting properties without substantially interfering with the strength and flexibility and air permeability of the fabric.

A further object of this invention is the provision of a fabric of the character indicated which may be readily and inexpensively prepared.

A still further object of this invention is the provision

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of a process whereby the above indicated fabric may be prepared.

Further and additional objects will appear from the following description and the accompanying claim.

- 5 In accordance with this invention, a fabric highly resistant to penetration by vesicants, such as mustard, may be prepared by treating a base fabric with particulate activated carbon dispersed in a cellulosic binder whereby the carbon particles are firmly affixed to the base fabric.
- 10 The carbon serves to resist the penetration of the fabric by vesicants; and the cellulosic binder, when properly fixed, firmly attaches the activated carbon to the base fabric without destroying the flexibility and air permeability of the fabric. Cellulosic binders which have been found particularly useful for the purpose are hydroxyethyl cellulose and cellulose regenerated from a viscose solution. However, as will be apparent from the following description, other cellulosic materials may be used such as the cellulose ethers, cellulose esters, the alkali soluble celluloses such as degraded alpha and beta cellulose, cupric ammonium cellulose, etc. By the term "cellulosic" as used in this specification and in the accompanying claim, it is intended to mean any binder which is prepared from cellulose or its derivatives, and more particularly the cellulosic plastics.

- 20 In accordance with preferred embodiments of this invention, particles of activated carbon may be suspended in any suitable cellulosic solution. This mixture is then applied to a fabric either by impregnation or by coating.
- 30 In the impregnation process, the fabric may be totally immersed in the carbon-containing mixture, while in the coating process, the mixture may be applied to one or both sides of the fabric by a standard coating method. After treatment of the fabric with the cellulosic mixture, the latter is fixed by suitable means. In the event that a viscose or a hydroxyethyl cellulose solution is employed, the binder may be fixed by passing the treated fabric through a bath containing a dilute mineral acid such as sulfuric acid. The resulting treated fabric may thereafter
- 40 be subjected to washing, drying, breaking, preshrinking, and other finishing operations to impart desired properties to the fabric.

- 45 For a more complete understanding of this invention reference is now made to the drawing and the following specific examples. It will be understood that the examples are for illustrative purposes only and this invention is not to be limited thereto.

In the accompanying drawing:

- 50 Figure 1 is a flow plan of an impregnating step; Figure 2 is a flow plan of a coating procedure, the coating step being an alternate for the step shown in Fig. 1 up to line A—A in Fig. 2;

Fig. 3 is a flow plan of a fixing procedure; and

Fig. 4 is a flow plan of a breaking procedure.

- 55 Referring to Fig. 2, an uncoated fabric web 1 unwound from roll 2 is passed over guide rolls 3, then between a controlled set of mangle rolls 5 and 6, spaced a suitable distance apart, e. g., 0.05". The upper roll 5 revolves in a counter-clockwise direction and the lower roll 6 revolves in a clockwise direction. The lower roll 6 is partly immersed in a bath 7 of coating material so that it is capable of transferring the coating material to the under surface of the fabric. The wet coated fabric leaving the mangle rolls 5 and 6 is passed over a smoothing knife 8 then over a series of steam heated drums 9, 10 and 11, and over guide rolls 12, which may be water cooled. The dry coated fabric is wound on roll 13 following inspection in front of an inspection light 14.

- 70 In an impregnating process, or in coating both sides of the fabric, as shown in Fig. 1, the fabric web 1 may be passed from roll 2 into the impregnating bath 7 by guide

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rolls 3 before being passed through a mangle roll assembly 5 and 6 for squeezing out excess impregnating solution.

The coated fabric after a preliminary drying, when necessary, is passed through a series of coagulating and washing solutions as illustrated in Fig. 3. The coated fabric is unwound from roll 15 to be passed first through acid baths 16 and 17, next through water washing baths 18 and 19, next through an alkaline neutralizing bath 20, and finally through a water washing bath 21. In passing through each of the baths the fabric may be passed between sets of squeeze rolls 22 and 23 for squeezing out liquid. After the final water washing, the fabric may be partly air dried in being led over reel 24 and then be folded into layers on a truck 25 by means of a conventional automatic folding mechanism, not shown. The fixed and washed product is then to be dried in a standard drying apparatus, and thereafter may be subjected to the action of a breaking unit.

The breaking unit illustrated in Fig. 4 comprises a series of button breaker rolls 27 and a breaking knife 28 over which the dried coated fabric is passed under tension in being unwound from roll 29 and rewound on roll 30.

Example 1

A stock solution of hydroxyethyl cellulose was prepared. The hydroxyethyl cellulose employed was one known to the trade as "Ceglin," and the stock solution had the following compositions:

	Lbs.
Ceglin (viscosity No. 2)-----	3.9
Sodium hydroxide (50% solution)-----	14.5
Ice -----	38.8
Water -----	42.8

The dry "Ceglin" was added to the sodium hydroxide solution in a mechanical mixer. Ice was then added and the resulting mass was mixed until the dispersion was complete. The mass was then diluted with the additional quantity of water to form the desired stock solution.

To form the impregnating mixture, 300 parts by weight of finely divided activated charcoal was added to 1200 parts of the above stock "Ceglin" solution in a mechanical mixer and the resulting mass was mixed until dispersion was complete. The final composition of the impregnating mixture was as follows:

	Percent by weight
Hydroxyethyl cellulose (Ceglin)-----	3.1
Activated charcoal-----	20.0
Sodium hydroxide (NaOH)-----	5.8
Water -----	71.1

The charcoal used was an activated charcoal of a type adapted as a base in gas mask canisters. The particle size distribution was as follows:

	Microns
19.6 percent-----	under 0.5
36.4 percent -----	0.5 to 1.0
26.0 percent -----	1.0 to 2.0
16.5 percent -----	2.0 to 3.0
1.5 percent -----	3.0 to 5.0

The above-referred-to impregnating mixture was applied to several different fabrics by impregnation. Base fabrics used were flannel napped on both sides and mercerized, and unmercerized twill. The impregnation may be effected by any standard process. The cloth may be completely immersed in the mixture and the excess mixture may be removed by passing the impregnated fabric between a pair of squeeze rolls 5 and 6, as shown in Fig. 1. If desired, the fabric may be immersed several times in succession to insure thorough impregnation.

After complete impregnation, the fabric was then immersed in a 5% solution of sulfuric acid for three minutes at 25° C. to coagulate or fix the hydroxyethyl cellulose. The fabric was then washed free of acid in water at 25° C. and finally dried on a swing frame by radiant heat and subsequently cured for ten minutes at 160° C. The re-

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sulting fabric was found to be highly resistant to penetration by mustard vapors. It was flexible and air permeable as the result of the swing framing operation which served to disrupt the continuous film of carbon and binder formed in the coagulating procedure. It also retained substantially its original tensile strength. The weight of the charcoal on a flannel fabric, treated as above indicated, was about 12.0 to 13.0 mg. per square cm. On the mercerized and unmercerized twill fabrics, the weight of charcoal was between about 3.6 and 5.5 mg. per square cm.

Satisfactory fabrics also have been prepared by applying the above indicated hydroxyethyl cellulose mixture to only one side of the fabric by a coating procedure followed by a three minute treatment with a 5% sulfuric acid solution and washing, drying under conditions whereby the continuous film is disrupted to improve flexibility and air permeability, and subsequent curing steps.

Example 2

About 150 lbs. of wood cellulose pulp (Novacell) were saturated with 18% NaOH for two to three hours. The sodium hydroxide was then pressed out of the treated pulp and the amount of sodium hydroxide retained was about 20% by weight of the pulp. The pressed pulp was then disintegrated and allowed to stand eighteen hours at 70-80° F. Thereafter the pulp was reacted with 60 lbs. of carbon disulfide at 60-70° F. This reaction product was then dispersed in sodium hydroxide and treated in a water jacketed disintegrator to form a final composition containing 5% cellulose and 5% NaOH having a viscosity of 2700 centipoises at 75° F. Finely divided activated carbon, of a type useful as a base in gas mask canisters but with particle sizes substantially all below three microns, was added to the viscose solution in a standard paint-pigment mixer. The amount of carbon added was such that the ratio of carbon to cellulose was about three to one. After one hour of mixing the viscosity of the resulting product was about 9000 centipoises at 75° F.

A base fabric comprising a standard herringbone twill was napped on standard napping equipment on one side. To the napped side was applied a coating of the above referred to carbon-viscose mixture by a standard knife coating procedure, e. g., as illustrated in Fig. 2. After coating, the fabric was passed over a plurality of steam heated drums 9, 10, and 11 to effect a preliminary drying of the coated fabric at a suitable temperature in the range of about 300 to 350° F. and at cloth speed of about 20 yds. per minute. The coated and dried fabric was then passed through a series of acid coagulation and washing baths, as shown in Fig. 3. The first two baths, 16 and 17, in this series contained 10% sulfuric acid maintained at 160° F. The second two baths, 18 and 19, contained water. The fifth bath, 20, contained a dilute solution of sodium carbonate, and the final bath, 21, contained wash water.

The washed product was then dried on a standard drying frame, not shown, and thereafter subjected to the action of a "breaker," as illustrated in Fig. 4, to reduce the stiffness of the product. The "breaker" comprises a series of button breaker rolls and a breaking knife which flexes the fabric thus reducing its stiffness. The coated fabric is run back and forth, under controlled tension at a cloth speed of about 50 to 250 yds. per minute, through the breaking unit until the desired degree of softness has been attained. Ordinarily six passes through the unit will suffice.

The stiffness and tendency to crock, of the thus softened fabric, may be further reduced by a washing operation. This may be effected by first wetting out the material with cold water, thereafter washing for about twenty-five minutes at 170° F. with a 0.2% of an inorganic detergent followed by warm water and cold water rinses.

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After washing, the fabric is dried and may be subjected to a standard preshrinking or Sanforizing operation.

It has been found that the fabric produced as indicated above retains, to a large degree, its tensile and tear strength. The carbon content of the cloth is between about 3.5 and 3.8 milligrams per square centimeter and its efficiency toward the action of mustard vapors compares very favorably to chemically impregnated fabrics. Also, the fabric is soft and has a considerable air porosity. It is indicated to be very satisfactory for use in protective clothing.

An advantage of the coating process indicated in Example 2 is that only one side of the fabric is coated with the carbon. Accordingly, the opposite side retains its original color and the garments made therefrom will not be unsightly.

While in the foregoing only two specific examples have been given for preparing the fabric of this invention, it will be apparent to one skilled in the art that various modifications may be made. For example, other cellulosic binders may be employed and activated carbon of varying degrees of particle size may be employed. Likewise the amount of carbon deposited on the fabric may be varied within wide limits and the ratio of carbon to cellulosic binder may also be varied if desired. However, it has been found, in the case of viscose, that the ratio of carbon to regenerated cellulose is preferably between two to one and four to one, and more particularly about three to one. With hydroxyethyl cellulose, the ratio may be somewhat higher, namely between about six to one and seven to one. Also, if desired, pigments may be mixed with the carbon-cellulose mixture in order to alter the color of the surface of the fabric. Inorganic pigments, non-poisonous to the carbon, are preferably employed.

Although modifications may be employed for coating a fabric in accordance with this invention, particular process steps disclosed in the examples and particularly in Example 2 have been found to produce very satisfactory products which may otherwise be unobtainable. For example, the breaking step and the final washing step are desirable in connection with the preceding steps in order to produce a fabric which will have the desired degree of softness, air permeability, and which will not crock excessively.

The air permeability of the fabric rendered resistant to penetration by vesicants appears to be dependent upon the discontinuous nature of the cellulosic binder, for if the binder is present on or in the fabric as a continuous film, the air permeability is extremely low. The process of this invention disrupts to a large extent whatever con-

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tinuity is possessed by the binder as it is initially applied. In very satisfactory products, interstices between interwoven threads of a textile base fabric cloth may be largely free of binder while pile or napped filaments extending from the interwoven threads have a predominant amount of the binder in a discontinuous coating.

The retention efficiency of the nap coated fabrics has been found to be surprisingly comparable to that of fabrics impregnated with vesicant destroying or absorbing chemicals. A composite fabric made by bonding together two layers of base fabric having their adhering adjacent sides coated discontinuously with a cellulosic binder holding particulate activated charcoal between the layers gave the result of greater than twice the retention efficiency of a single layer of the same fabric coated on one side.

In general, protective fabrics made in accordance with the present invention, have excellent adsorptive capacity for vesicant vapors and other agents, including some which do not readily react with known impregnating chemicals. They have satisfactory tear and tensile strength. They possess desirable storage stability and wearing qualities.

While several specific examples have been given in the foregoing description, it is not intended that this invention shall be limited thereto. Many modifications will be readily apparent to one skilled in the art and it is intended that the invention be limited only by the scope of the appended claim.

I claim:

An antivesicant filter fabric consisting of a porous, flexible cellulose fabric and a discontinuous crocked coating adhesively secured on said cloth in a manner adapted to leave said cloth porous and flexible, said coating being a binder of hydroxyethyl cellulose having particles of activated carbon dispersed therein, the proportion by weight of said hydroxyethyl cellulose to said carbon being in the range of 1:6 to 1:7.

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