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FLAME RETARDING COMPOSITION AND FABRIC TREATED THEREWITH

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This invention relates to an improved composition for imparting flame resistance to fabric materials. In another aspect it relates to improved flame and heat resistant fabrics, and more particularly to such type fabrics which possess resilience, flexibility, smoothness of texture and thermal insulating qualities following streatment at high temperatures. The present application is a continuation-in-part of my co-pending application Serial No. 449,521, filed August 12, 1954, now Patent No. 2,884,343.

Flame and heat resistant fabrics in general have been commonplace for many years. For example, it has been known that many type fabrics can be rendered flame resistant by impregnating the same with a composition consisting of a fire-proofing agent and a plasticizer. Moreover fabrics treated in such a manner usually exhibit flame-proofing characteristics. Invariably such fabrics have, as a direct result of this flame-proofing treatment, likewise manifested several most undesirable qualities which were not characteristic of the fabric before treatment. Heretofore such fabrics have, after treatment, been characterized by a loss of flexibility or resilience, and moreover a lack of ability of the fabric to slip over itself in folding or turning operations. The latter disadvantage may be designated for purposes of convenient description as a lack of smoothness of texture in the fabric. The importance of eliminating these difficulties in the finished fabric is readily realized when it is understood that such fabrics have widespread utility in the field of safety garments, such as flame-proof gloves, where loss of flexibility or lack of ability of the fabric to slip or slide over itself presents insurmountable hazards in the fabrication processes. Moreover fabrics of this type have in the past been characterized by a relatively large percentage weight loss upon exposure to the high temperatures incident to their normal conditions of use. This loss has been due to the rather high rate of volatilization or decomposition of the flame-proofing ingredients at red heat temperatures and is known as ignition loss. Trade requirements today demand that asbestos textiles meet a minimum ignition loss standard and hence the disadvantage of the aforedescribed inherent characteristic of presently known flame-proofing materials will be apparent.

As employed herein the term "asbestos-base" material or fabric is intended to mean a material or fabric containing at least 50% by weight asbestos fibers. For example, a preferred fabric for flame-proofing in accordance with the present invention is one containing approximately 83% asbestos and 17% by weight cotton.

In accordance with the present invention the disadvantages of the aforementioned flame-retardant fabric materials are overcome by providing a novel flame-proofing composition, and an asbestos-base material impregnated with this novel composition is superior not only because

of lower ignition loss but also in resiliency and smoothness of texture to flame-retardant fabrics available heretofore.

The flame-proofing composition of the present invention comprises an aqueous medium containing a fluoborate sale as the flame-retardant, an inert texture-imparting agent and a plasticizer. The plasticizer is designed to retain in the fabric the softness and resiliency which ordinarily would in part be destroyed by introduction of the flame-retardant to the fabric. The texture-imparting agent on the other hand acts partly in the nature of a dry lubricant and serves to retain in the fabric the ability to slide over itself during folding and turning operations, which normally would likewise be inhibited by the action of the flame retardant. The texture-imparting agent is an indispensable element in both the impregnating composition and the flame retardant fabric produced therefrom and comprises an inert material such as finely ground mica. The mica serves to blend the flame retardant and plasticizer into admixture in the composition, which when applied to the asbestos textile not only imparts thereto the necessary flame-proofing characteristics, but also maintains the fabric's original and desirable properties of flexibility and smoothness of texture even after the fabric has been subjected to very high temperatures.

A number of flame retardants for use in connection with texture-imparting mica and the plasticizer have been disclosed in my co-pending application Serial No. 449,521. These include borax alone and in combination with ammonium chloride or boric acid, ammonium borate, antimony oxide and zinc borate, among others, and the concentration of the flame retardant in the impregnating composition is from about 7 to 24% based on the weight of the water therein. For example, the borates to which my earlier application is particularly directed must be in the impregnating composition in amounts of at least 7 to 10% by weight of the water.

I have now found that salts containing both boron and fluorine atoms in the same molecule are much more effective than either the borates or the fluorides alone or in combination. The fluoborate salts have inherent properties which are different from other borates and compounds heretofore used as flame retardants, which make them more effective for flame and heat resistance. I have found that sodium fluoborate, for example, is much more effective than a physical mixture of borax and sodium fluoride, and to obtain approximately the same degree of flame retardancy in a treated fabric attainable with sodium fluoborate by using a physical mixture of borax and sodium fluoride the amount of the mixture required is much greater than the weight of sodium fluoborate necessary. This has a great practical advantage in that the fabric can be completely flame-proofed with but a small quantity of the treating material with the result that the fabric is not unduly weighted with the flame retardant nor is the hand of the fabric changed to an appreciable degree. Considerably less fluoborate salt is required to effect a desired degree of flame-proofing than with other flame retardants or combinations of the same.

The novel flame-proofing compositions of the present invention are aqueous media containing about 2 to 6% fluoborate salt, about 1.5 to 7% mica and about 2 to 4% plasticizer based on the weight of water present in the composition. It is not necessary that the fluoborate salt be soluble in water so long as it is sufficiently finely divided to be readily dispersed in the aqueous medium. The texture-imparting agent, mica, is of course insoluble in water which necessitates a fine state of subdivision

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in order to obtain the requisite dispersion in the composition. Among the fluoborates preferred are the water soluble alkali metal and ammonium fluoborates, and of these sodium fluoborate has been found to be most effective, almost twice as effective as potassium fluoborate. While the fluoborate salt content of the impregnating composition regardless of solubility will be between about 2 and 6% by weight of the water present, as little as 2–3% of sodium fluoborate is all that is required, with about 3% being preferred. With the less soluble or insoluble alkaline earth metal fluoborates, for example, concentrations nearer the upper end of the range are required. The present invention also contemplates the use of organic fluoborate salts as the flame retardants whether soluble or insoluble in water. It is apparently the boron-fluoride radical which is the effective flame retardant. In general organic compounds containing the boron-fluorine bond are satisfactory if they can be dissolved or dispersed in the treating medium. Examples of suitable organic compounds are boron phenyl difluoride and boron p-tolyl difluoride.

A typical impregnating composition of the present invention is:

Water	100.0 parts.
Flame retardant:	
Sodium fluoborate	3.0 parts.
Deceresol P (wetting agent)	0.3 parts.
Texture imparting agent: (mica, finely ground)	3.0 parts.
Plasticizer:	
Tricresyl phosphate, 85%	
Water, 10%	
Diglycol Stearate, 5%	
(Emulsifying agent)	3.0 emulsion.

The proportions of the various components in the impregnating composition can of course be varied within the aforementioned limits while still effecting the objects of the present invention, with the aforementioned exemplary composition merely a preferred solution.

The use of a wetting agent in the impregnating composition is often indicated and in the above composition Deceresol P was employed. It should be noted that many other well known wetting agents in approximately the same amounts are interchangeable in the composition. Among such wetting agents are those sold under the names "Aerosol," "Sulfatate," "Wetanol," "Duponol," "Tergitol," and many others. Similarly, it is noted that while diglycol stearate was used as the emulsifying agent with tricresyl phosphate, other well known emulsifying agents in approximately the same amounts may be used to equal advantage. Among these, are those sold under the names of "Tergitol NPX," "Emulgar A" and others. Several other plasticizers may be employed in place of tricresyl phosphate, for example, propylene glycol or triphenol phosphate.

The method of treating the fabric comprises the steps of first placing the proper weights as above set forth, of flame retardant, texture-imparting agent and plasticizer in a diptank and then adding water thereto while maintaining a vigorous stirring action. When the fluoborate is dissolved or completely dispersed as the case may be, and the mica uniformly suspended, the fabric is run through the tank in such manner as to accomplish total immersion thereof. During this period the treating solution is maintained in constant agitation, as by means of any well known type of recirculating pump. Following immersion the asbestos fabric is then run through a pair of squeeze rolls, preferably formed of neoprene or rubber. This last step removes excess treating solution and leaves the fabric impregnated with the flame-proofing composition. The material is then dried. Such a process results in the fine particles of mica along with the flame retardant and plasticizer becoming deposited on the fabric fibers and in the voids between fibers where they remain after treatment due to the cementing action of the plasticizer and flame retardant. The fabric is uniformly impregnated with mica even though the same is only in suspension in the treating solution because of the minute size of the mica particles and the inherent fiber structure of asbestos fabrics, and the same is true of the finely divided insoluble fluoborates.

As the result of treatment with the impregnating composition, the flame retardant, texture-imparting agent and plasticizer are deposited on the finished fabric in substantially the same ratio as they exist in the treating medium. This fact has been determined by analyses of the finished fabrics and also by analyses of the treating baths before and after production runs. Treatment of a fabric consisting of 83% asbestos and 17% cotton by weight as aforesaid with the above exemplary impregnating composition produced a flame-proof fabric of the following analysis:

	Percent
Asbestos fabric	95.2
Retardant	1.7
Mica	1.7
Plasticizer	1.4
	100.0

Experience has shown that between about 50 and 65% of the retardant, mica and plasticizer from the impregnating composition are deposited on or intermediate the fibers of the asbestos fabric.

In the resulting fabric the flame retardant of course renders the same flame-proof while the plasticizer maintains flexibility and resiliency without increasing flammability. The mica imparts smoothness to the finished fabric, as described above and renders body to that fabric. In this regard mica in finely ground form is especially satisfactory since it is inherently slick and its tiny particles slide easily over one another much in the same manner as does graphite. Moreover, when used in the combination herein set forth, it increases the tensile strength of the finished fabric after the same has been subjected to heat at relatively high temperatures. Furthermore, it increases the heat resistance of the fabric and also its thermal insulating properties. Being substantially inert the mica does not readily volatilize when subjected to flame heat and hence has an extremely low ignition loss. On the other hand the constituent elements of the flame retardant and plasticizer have a relatively high ignition loss. Therefore if these latter ingredients were to be used without the inert texture-imparting agent the overall ignition loss of the fabric would be quite high, but when the mica is added in the proportions specified that overall ignition loss is reduced by as much as 60–70%. Thus in an asbestos textile fabric treated according to the invention their weight reduction due to ignition loss is only about 2.5 to 3.8% whereas a similar asbestos fabric treated solely by a flame retardant and plasticizer will show a reduction of from 5 to 9%.

A standard test was devised to measure the concentration of flame retardant in the impregnating composition necessary to impart a particular degree of flame retardancy to a standard asbestos base fabric. The test consisted of holding a strip of the treated fabric 1 inch wide by approximately 15 inches long over a Bunsen burner, the flame of which was adjusted 6 inches high above the top of the burner, and protected against drafts, with the lower end of the strip being held in the flame so that the flame licked the bottom 2 inches of the strip. The strip was so held for 40 seconds after which it was withdrawn. If flaming or flickering continued over 5 seconds following withdrawal from the flame the fabric was not considered flame-proof. All of the retardants tested were made up in various concentrations in the impregnating composition and a variety of retardants were employed to determine the smallest percent of each in the

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impregnating composition which would give full flame-retardancy. The results of these tests appear in the following table:

Flame Retardant:	Minimum percent necessary in the treating medium for full flame retardancy
Sodium fluoborate	3
Potassium fluoborate	4.5
Borax	8.5
Ammonium borate	9.0
Boric acid	8.5
Zinc borate	8.0
Ammonium chloride	8.8
Aluminum acetate	12.0
Antimony oxide	10.0
Monoammonium phosphate	7.0
Diammonium phosphate	7.0
Ammonium sulfamate	12.0
Urea phosphate	10.0
Aluminum sulfate	9.0
Monosodium phosphate	10.0
Neoprene	9.0
Chlorinated paraffin	8.5
Vinylite	7.5

It is readily apparent that the fluoborate salts are much more effective than the conventional flame retardants tested, in fact, from more than two to four times more effective on a weight basis alone. Furthermore, the fluoborate salts produce a fabric with improved hand as well as one that is flame and heat resistant.

What is claimed and desired to be secured by Letters Patent is:

1. An aqueous composition for application to a fabric for improving the flame retarding properties thereof which comprises about 2 to 6% fluoborate salt, about 1.5 to 7% mica, about 2 to 4% plasticizer and the balance substantially water, said percentages being based upon the weight of water present in the composition.

2. An aqueous composition for application to an asbestos material for improving the flame retarding properties thereof, which comprises about 2 to 6% alkali metal fluoborate, about 1.5 to 7% mica, about 2 to 4% platicizer and the balance substantially water, said percentages being based upon the weight of water present in the composition.

3. A flexible, resilient flame and heat retardant fabric characterized by a smoothness of texture and by a low overall ignition loss after being subjected to high temperature, comprising an asbestos base material having deposited thereon and intermediate the fibers thereof about 1 to 4% fluoborate salt, about 0.75 to 4.5% mica and about 1 to 2.8% of a plasticizer, based upon the total weight of the fabric.

4. A flexible, resilient flame and heat retardant fabric characterized by a smoothness of texture and by a low overall ignition loss after being subjected to high temperature, comprising an asbestos base material having deposited thereon and intermediate the fibers thereof about 1 to 4% of a water-soluble fluoborate salt, about 0.75 to 4.5% mica and about 1 to 2.8% of a plasticizer based upon the total weight of the fabric.

5. A fabric as set forth in claim 4 wherein the soluble fluoborate salt is an alkali metal fluoborate.

6. A flexible, resilient flame and heat retardant fabric characterized by a smoothness of texture and by a low overall ignition loss after being subjected to high temperatures, comprising a dry asbestos base material which has been impregnated with an aqueous composition containing as active ingredients about 2 to 6% fluoborate salt, about 1.5 to 7% mica and about 2 to 4% plasticizer, based upon the weight of water present in said composition, with substantially the balance of said composition being incapable of enhancing the flame retarding properties of the fabric, said impregnated fabric in dry condition retaining between about 50 and about 65% of said active ingredients.

7. An aqueous composition for application to a fabric for improving the flame retarding properties thereof, which comprises at least about 2% of a fluoborate salt, at least about 1.5% mica and at least about 2% of a plasticizer and the balance substantially water, said percentages being based upon the weight of water present in the composition.

8. An aqueous composition for application to an asbestos base material for improving the flame retarding properties thereof, which comprises at least about 2% of a fluoborate salt, at least about 1.5% mica and at least about 2% of a plasticizer and the balance substantially water, said percentages being based upon the weight of water present in the composition.

9. An aqueous composition for application to an asbestos base material for improving the flame retarding properties thereof, which comprises at least about 2% of a water-soluble fluoborate salt, at least about 1.5% mica and at least about 2% of a plasticizer and the balance substantially water, said percentages being based upon the weight of water present in the composition.

10. An aqueous composition for application to an asbestos base material for improving the flame retarding properties thereof, which comprises at least about 2% of an alkali metal fluoborate salt, at least about 1.5% mica and at least about 2% of a plasticizer and the balance substantially water, said percentages being based upon the weight of water present in the composition.

11. A flexible, resilient flame and heat retardant fabric characterized by a smoothness of texture and by a low over-all ignition loss after being subjected to high temperature, comprising an asbestos base meterial having deposited thereon and intermediate the fibers thereof at least about 1% of a fluoborate salt, at least about 0.75% mica and at least about 1% of a plasticizer, based upon the total weight of the fabric.

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CERTIFICATE OF CORRECTION

Patent No. 2,948,641 August 9, 1960

John D. McCluer

It is hereby certified that error appears in the printed specification of the above numbered patent requiring correction and that the said Letters Patent should read as corrected below.

Column 1, line 20, for "streatment" read -- treatment --; column 2, line 5, for "sale" read -- salt --.

Signed and sealed this 31st day of January 1961.

(SEAL)
Attest:

KARL H. AXLINE
Attesting Officer

ROBERT C. WATSON
Commissioner of Patents

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