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METHOD OF MAKING A FIRE-RESISTANT TEXTILE MATERIAL

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This invention relates, generally, to improvements in the art of imparting flame resistance to cellulosic fibers and fabrics, as well as other relatively inflammable fibers and fabrics, while simultaneously rendering the fibers and fabrics thus processed, resistant to the action of weathering and attack by bacteria and fungoid agencies, usually described under the collective title of mildew, and the invention relates, more particularly, to a novel flame resistant processed product, and to the process of preparing the same. This application is a continuation-in-part of the co-pending application, Serial No. 538,883, filed June 5, 1944, for Fire Resistant Fabric Material.

The material to be treated of most common occurence and commercial importance is cotton duck, cotton sheeting, cotton camouflage and other types of netting, and the various types of cotton clothing fabric, such as drills, twills and poplins. At present, commercially acceptable grades of fire resistance must meet the requirements of Federal Specifications CCC–D. 746, February 1939, for duck, cotton; fire, water and weather resistance, and as amended by supplementary specification (tentative) 242. Processed material that will substantially comply with the requirements of the above mentioned specifications may be considered as "fireproof" within the meaning of this invention.

Material processed as "fireproof" which will meet the requirements of Specifications 746 and 242, noted above, has hitherto been prepared by the application of a fire resistant paint formula in which ground pigments of various types are suspended in a halogenated organic material which serves as a base and as a carrier for the essential pigments. These pigment solids are preferably mixed or ground with the halogenated carrier material (almost universally a chlorinated compound), and then deposited in and on the fabric, the chlorinated material serving as a base or carrier, usually known as a vehicle.

While the flame resistance produced by the application of this fire-resistant paint meets the requirements of the aforesaid specifications, a very heavy loading of the fabric is an indispensable prerequisite, the heavy loading producing the defect of tackiness at normal temperatures and objectionable stiffness when subjected to the usual winter temperatures of the temperate zone, which stiffness is accentuated greatly when subjected to arctic temperatures. While the degree of stiffness may be materially modified by the addition of relatively large amounts of plasticizing media, the addition of plasticizing media greatly increases the degree of tackiness. Another objection, vital in many cases, lies in the tendency of the soft, fire-resistant paint to "crock" or rub off, soiling any article with which it may come in contact, a defect which renders fabrics processed with such fire-resistant paints unavailable for clothing, chair or seat covers, and affects adversely their usefulness for tentage and awning purposes, largely for the reason that they will soil any fabric which comes in contact with them, and, having a soft, tacky surface, such processed fabrics attract dirt and grime, and quickly become unsightly, although still retaining their fire-resisting characteristics.

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The present invention does not make use of a flame-resisting paint composition, but instead employs materials that are introduced into the fabric, fiber or structure to be fireproofed, in the form of solutions, which produce insolubilized material on and within the fibers of the cellulosic structure. These materials may be introduced into the fabric material in the form of aqueous solutions of the salts of the metals and compounds to be employed, or, where feasible, in a water solution of the compounds, the metallic compound being insolubilized by means of appropriate precipitants, or is reacted on to form an insoluble material by means of a compound of the alkali metals or ammonia, such as the carbonates, bicarbonates or hydroxides of the alkali metals and ammonium, or alkali salt of a weak acid.

One of the objects of the present invention is to provide a novel fire-resistant textile fabric or similar material employing a minimum amount of loading, i. e., wherein the effective loading is reduced from 40% to 60% of the loading ordinarily required in fire-resistant textile fabrics heretofore known.

Another object of the present invention is to provide a novel flame resistant textile fabric or similar material which will not crock, and from which the flame-resistant material will not rub off, as in the case of the paint-coated fabrics heretofore commonly used, whereby the material of this invention is adaptable for use for clothing, awnings, furniture and the like.

Another object of the present invention is to provide a novel flame-resistant textile fabric or similar material that is not tacky and is not stiff at low temperatures, but possesses a degree of flexibility approximating that of the untreated fabric.

A further object of the invention is to provide

a method of making a flame-resistant fabric or similar material by the use of antimony trifluoride in a water solution which is converted to antimony trioxide within the fabric or fibers thereof by a precipitating medium or converting medium introduced after the fabric or fibers have been impregnated with the antimony trifluoride.

A further object of the invention is to provide a method of making a flame-resistant fabric or textile material by the use of antimony trifluoride in aqueous solution which is converted to antimony trioxide within or on the fibers of the fabric or textile material in such a manner that the tensile strength and tear resistance of the fabric or textile material is not substantially reduced, or in other words, in such a manner that no acid is released and the fabric or textile material retains its original strength and tear resistance.

A further object of the invention is to provide a method of making a flame-resistant fabric or textile material by the precipitation of antimony trioxide within and on the fibers of the fabric or textile material in a manner such as to avoid hydrolysis of the antimony tri-fluoride employed, by the use of a compound or material, in the impregnating antimony solution, serving to drive back the dissociation or hydrolysis of the fluoride a sufficient period of time to permit saturating and drying of the fabric or textile material without the release of sufficient acid to substantially reduce the tensile strength and tear resistance of the material treated.

A further object of the invention is to provide a method of making a flame-resistant fabric or textile material by the use of antimony trifluoride in a water solution containing a soluble fluoride salt or other medium serving to prevent dissociation or hydrolysis of the antimony trifluoride for a period of at least four to six hours to permit penetration or saturation of the fabric or textile material under commercial operating conditions and permitting drying of the fabric or textile material without the release of a sufficient amount of hydrofluoric acid to tender the fabric or textile material prior to treatment with a sodium carbonate solution or precipitating medium by which antimony trioxide is precipitated in situ in the fabric structure or textile structure.

A still further object of the invention is to provide a method of making a flame-resistant fabric or textile material by the use of antimony trifluoride in a water solution containing a medium adapted to prevent substantial hydrolysis of the fluoride for a period of time sufficient to carry out commercial operations by which antimony trioxide is precipitated or deposited within the fabric or textile structure without the release of hydrofluoric acid into the atmosphere or the release of sufficient acid into the impregnating solution to reduce the tensile strength of the fabric or textile material treated and to permit washing out or removing all soluble salts from the treated material during treatment without substantially reducing the tensile strength of he said material.

With these and other objects in view, the invention comprises the various features and method steps hereinafter described and more particularly defined in the claims annexed hereto.

Ordinary paints consist of a mixture of a solid pigment and a liquid medium known as a vehicle, the latter being linseed oil or other drying oil, a drier and turpentine or other volatile thinner. When such a paint is applied to a textile fabric,

it renders the fabric hard and brittle upon drying and the fabric also becomes impervious to air, so that the same is not suitable for use as tentage, the lack of ventilating facilities and excessive heating due to the heat-absorbing nature of the painted surface preventing the use of the painted fabric for this purpose.

Furthermore, such fabric material thus coated with paints is not fireproof, owing to the use of linseed oil, and attempts have been made to replace this oil by a non-burning chlorinated hydrocarbon. Also, in order to prevent such textiles from becoming hard and brittle, the use of driers was prohibited, and while this cured the tendency of the fabric to become hard when the paint dried, nevertheless it rendered the fabric tacky even at ordinary temperatures, and this particularly was an objection in the tropics where the tackiness would make it difficult to unroll the thus-prepared material.

Moreover, by the use of paint material for flameproofing of fabrics, the fabric characteristics of the product are entirely lost, whereas the process of the present invention leaves all of the desirable fabric-like characteristics, such as drape, elasticity, softness and "hand" in the flame-proofed product.

In order to correct the tackiness of such paint-coated fabric materials, attempts were made to use "film formers," such as ethyl cellulose, urea formaldehyde resins, and phenolic condensation products, but the use of such substances tended again to deprive the fabric of its fabric-like qualities and to render the textile material rigid and stiff.

Various attempts were then made to produce a satisfactory product which would not be too tacky at normal temperature and which under aging and exposure would not become too hard and stiff, but without material success. Chlorinated paraffin was then tried, for although the same tends to become tacky at higher temperatures, it exhibits gummy characteristics rather than hardness or stiffness at lower temperatures. Unfortunately, however, chlorinated paraffins are unstable and tend to break down, evolving hydrochloric acid, which is extremely destructive of the fabric, the instability of the chlorinated paraffins increasing rapidly with increase in the chlorine content. In order to correct for this tendency, attempts were made to use calcium carbonate with the chlorinated hydrocarbon, the function of the carbonate being to neutralize any hydrochloric acid evolved from the chlorinated hydrocarbon in the use of the fabric. Unfortunately, great difficulty was experienced in getting the calcium carbonate into the interior of the fabric.

Cotton fabrics, whether of duck, twill, chain cloth, etc., are among the best of filtering media, and as fireproof paint consists of solid pigments suspended in a vehicle and solvents, which thin down the vehicle, filtration begins immediately when this mixture is applied to the fabric. The vehicle and solvent, being liquid, tend to pass into the interior of the fabric, while the suspended pigment tends to collect on the surface of the cloth being processed. Thus, upon drying, the pigment was all on the outside of the cloth, while the vehicle or binder was absorbed into the interior of the cloth, leaving no binding material to hold the pigment particles together, whereby the pigment particles either flaked off or dusted off, leaving the fabric impregnated with the vehicle or binder without sufficient binding material to

hold the pigment particles to the fabric. This condition caused so much trouble that grinding pigment, binder and solvent together became mandatory, and it was found that in order to assure reasonable impregnation of the fabric by the fire-resisting pigments, at least seventy-two hours of grinding was necessary. Even under the best of grinding conditions, filtration of the pigment by the fabric, with a tendency of the pigment to concentrate on the surface thereof and the corresponding tendency of the vehicle and solvent to accumulate within the fabric being processed still remained a grave problem.

Chlorinated paraffin is uncontrollably unstable even with but fifty per cent chlorination and must consequently be neutralized by calcium carbonate, the presence of which in the flame resistant paint, to the extent of about one-third the weight of the chlorinated paraffin used, has been made mandatory. It is clear, however, that the hydrochloric acid evolved from the chlorinated paraffin, which hydrochloric acid is destructive to the fabric or tenders the same, cannot be neutralized by the calcium carbonate unless the acid is in actual contact therewith, and if the calcium carbonate remains on the surface of the fabric, by reason of the filtering action of the cotton cloth under process, while the liquid chlorinated paraffin passes into the interior of the fabric, it is obvious that the contemplated neutralizing action of the calcium carbonate cannot take place and the evolved hydrochloric acid is thus free to damage the fabric. This is precisely what has happened in numerous cases in the use of this neutralizing agent and even the most meticulous care seems unable to cope successfully with the problem.

It has been found that the amount of antimony trioxide required to produce the necessary degree of flame-resistance by means of fireproof paint is between ten and twelve per cent of the weight of the processed goods, the amount being a somewhat variable quantity because of the uncontrollable variations of conditions. The impregnation method of incorporating antimony trioxide in fabric, as explained in the Patent No. 2,427,997, for "Fire Resistant Fabric Material," is much more efficient in preventing flame commustion than the orthodox "paint method" currently used. Actual trial and experiment have demonstrated that a content of from four and one-half to nine per cent of antimony trioxide incorporated into the fabric or textile material by the method of the co-pending application Serial No. 538,883, filed June 5, 1944, for "Fire Resistant Fabric Material" is quite as effective in preventing flame combustion as twice this amount when incorporated by means of the current orthodox "paint method." Moreover, the prevention of creeping combustion is favorably influenced by the said method of my co-pending application, above identified.

Antimony trifluoride, SbF_3, being a halogen compound of antimony which is soluble in water, can be applied as the impregnating medium for cotton goods in aqueous solution. This material may be obtained commercially as the salt, antimony fluoride, and as a mixture of antimony trifluoride with sodium fluoride, corresponding substantially to the formula $SbF_3.NaF$, and also as "De Haen salt," which is a double salt of antimony trifluoride and ammonium sulphate, $SbF_3.(NH_4)_2SO_4$. In accordance with the present invention, either antimony trifluoride alone or admixed with sodium fluoride, or in admixture with another soluble fluoride salt, or "De Haen salt" or this salt in admixture with antimony trifluoride or with sodium fluoride or other soluble fluoride salt may be applied in a water solution as the impregnating medium for the textile material to be flame-proofed in accordance with the method hereinafter set forth in detail. Antimony trifluoride can be reduced to antimony trioxide by means of sodium carbonate in a water solution, or by means of the carbonate or bicarbonate of an alkali metal or ammonium, but it is, nevertheless, entirely incompatible with mineral dye liquors, which ordinarily contain a solution of chromium, iron, calcium, magnesium or rare earth compounds, all of which are precipitated as fluorides in the presence of a soluble fluoride, as antimony trifluoride, for example. However, while antimony trifluoride may be entirely incompatible with mineral dye liquors, and cannot be impregnated into a fabric simultaneously with a mineral dye liquor, a solution of antimony trifluoride may be used to impregnate gray goods and dyed goods, employing either vat or other organic dyestuffs, and also may be used to impregnate mineral dyed goods, the conversion of the antimony trifluoride into the corresponding trioxide being then effected by means of sodium carbonate or other equivalent alkali carbonate, in accordance with the following reaction:

$$2SbF_3 + 3Na_2CO_3 \longrightarrow Sb_2O_3 + 6NaF + 3CO_2$$

Antimony trifluoride	Sodium carbonate	Antimony trioxide	Sodium fluoride	Carbon dioxide

Antimony trifluoride in the presence of a water solution of a soluble carbonate or bicarbonate of an alkali metal or ammonium is immediately decomposed with the formation of insoluble antimony trioxide together with sodium fluoride and carbon dioxide, which is evolved in accordance with the above equation. Antimony trioxide is an excellent flame inhibitor and is inert insofar as the fabric or textile material on which it is deposited is concerned.

The sequence of steps which constitute the method of the present invention are broadly as follows:

First, the fabric or textile material to be treated in accordance with the method of the invention is desized by treatment preferably with a diastase solution at a temperature of from about 140° F. to 160° F., the fabric or textile material being rolled up and put aside in the line of process, by which the starch present is converted to sugar. After the size has been converted, the fabric or textile material is rinsed so as to wash out all soluble materials, including sugars.

Second, the desized fabric or textile material is dried.

Third, the desized and dried fabric or textile material, whether of gray goods, goods dyed with organic dyes, or goods dyed with mineral compounds, is impregnated with a water solution of antimony trifluoride of any suitable strength, up to that of a saturated solution, although preferably a twenty percent solution is used in the ordinary case.

Fourth, the impregnated fabric or textile material is then squeezed between rollers and is then dried on cans in the usual manner.

Fifth, the dried fabric or textile material which is impregnated with antimony trifluoride is then passed through a water solution of a soluble carbonate or bicarbonate of an alkali metal or ammonia, although preferably a warm solution of

sodium carbonate, such as a 10% solution, is used, which serves to decompose the antimony trifluoride in the fabric or textile material and to form insoluble antimony trioxide in situ therein throughout the body of the fabric or textile material, sodium fluoride and carbon dioxide being also formed during the reaction in accordance with the above equation.

Sixth, the fabric or textile material after being thus treated is now washed with water, preferably by immersing the material in water, or otherwise by means of water sprayed onto the textile material, which serves to remove the residual carbonate or bicarbonate together with the sodium fluoride, which may have been present in the original impregnating solution or formed in the reaction, together with any other soluble material present.

Seventh, the thus processed fabric or textile material is then dried, and after drying the impregnated fabric or textile material is flameproof. While the material will not support flame, combustion can still be propagated through the fabric, but without flame effect.

The following will serve as an example of a working process, where undyed or unprocessed gray goods are used for processing:

First, the undyed or unprocessed gray goods are desized by treatment with a diastase solution at a temperature between 140° F. and 160° F. and is rolled up and put aside in the line of process, by which the starch size is converted to sugar. The goods are then rinsed to wash out all water soluble materials, including the sugars.

Second, the fabric or cellulosic material to be flameproofed is padded, that is, passed through a dye bath and squeezed through rollers or "nips," by which the material is dyed.

Third, the material is impregnated with an aqueous solution of antimony trifluoride, using preferably a twenty per cent solution thereof.

Fourth, the antimony trifluoride impregnated fabric or textile material is then dried on cans.

Fifth, the dried fabric or textile material is then padded with a dilute solution of an alkali carbonate, preferably a ten per cent solution of sodium carbonate, which is preferably hot.

Sixth, the thus treated fabric or textile material is then preferably rolled up and put aside for a sufficient length of time to permit the reaction between the sodium carbonate solution and the antimony trifluoride to take its normal course, by which antimony trioxide is formed and precipitated in situ within the body of the fabric or textile material.

Seventh, the thus treated fabric or textile material is then washed with water to remove soluble material and is subsequently dried, preferably on cans.

The resulting fabric or textile material, processed by the above steps, has now been flameproofed, but this treatment does not prevent the propagation of flameless combustion through the fabric or textile material upon the application of the necessary degree of heat. Thus, the fabric or textile material will "glow," and the zone of combustion will creep, although actual flaming is no longer possible. The prevention of "glowing," or the propagation of a creeping area of flameless combustion, requires an additional treatment.

Certain organic compounds, principal among which may be mentioned chlorinated paraffin, tricresyl phosphate, polymers of vinyl resin and various phenolic compounds, will prevent the propagation of a zone of creeping combustion, but will not, in themselves, prevent flame propagation. A combination of antimony trioxide and one of the above organic compounds will result in the production of a "fireproofed" fabric or textile material which will not only prevent flame propagation, but will also prevent creeping or "glowing" propagation of a zone of combustion.

One or more of the above mentioned organic compounds may be added to the fabric or textile material processed as above described by means of a separate and distinct operation. Thus, the fabric or textile material processed as above described may be passed through a solution of chlorinated paraffin, tricresyl phosphate, polymers of vinyl compounds, as vinyl acetate, vinyl chloride, and copolymers thereof, phenolic compounds, as phenol formaldehyde, etc., separately or in combination of one or more of these ingredients, after which the solvent may be removed by evaporation.

Should the glow-inhibiting media consist in the main of chlorinated hydrocarbons, such as chlorinated paraffin, for instance, all of which decompose under the influence of heat, weathering and exposure to ultraviolet light, which results in the liberation of hydrochloric acid, the effect of which is to weaken the fabric or textile material, frequently destroying its usefulness entirely, a neutralizing medium, such as calcium or magnesium carbonate should be incorporated into the fabric or textile material, preferably with the glow-inhibiting solution in the form of a suspension or dispersion.

In carrying out the invention above described, it has been found advantageous under some conditions of operation to use a mixture of antimony trifluoride with sodium fluoride, or potassium fluoride, or some other soluble fluoride, in place of antimony trifluoride alone. For this purpose, a mixture of antimony trifluoride and sodium fluoride may be employed which corresponds substantially to equimolar weights of antimony trifluoride and sodium fluoride, and corresponding substantially to the formula $SbF_3.NaF$.

It is well known that the solubility of antimony trifluoride at 0° C. is 384.7 grams per 100 ml. of water and the solubility increases with rise in temperature, the solubility at 20° C. being 444.7 per 100 ml. and at 30° C. is 563.6 per 100 ml. of water. The solubility of sodium fluoride at 0° C., on the other hand, is 4.0 per 100 ml. of water, the solubility also increasing with rise in temperature, the solubility at 18° C. being 4.22 grams per 100 ml. of water, and at 100° C., the solubility is about 5.0 grams per 100 ml. of water. Potassium fluoride is more soluble than sodium fluoride, the solubility at 18° C. being 92.3 grams per 100 ml. of water and at 80° C. the solubility is 150 grams per 100 ml. of water.

In carrying out the invention using a mixture of antimony trifluoride with sodium fluoride, a solution of equimolar quantities thereof containing 20 grams of antimony trifluoride and about 4.74 grams of sodium fluoride in 80 ml. of water may be used in place of the twenty per cent solution of antimony trifluoride above described. The amount or proportion of antimony trifluoride and the corresponding amount or proportion of the added soluble fluoride may be varied to a considerable extent, as will be understood by those skilled in the art, but the amount of soluble fluoride is preferably varied between the amount corresponding to about one-half the

molar weight to about two molar weights for each molar weight of antimony trifluoride. In other words for each 177.2 grams of antimony trifluoride, from 21 to 84 grams of sodium fluoride may be used although preferably the amount of 42 grams of sodium fluoride, corresponding to the molar weight thereof, is used with each molar weight of 177.2 grams of antimony trifluoride. Correspondingly, if potassium fluoride is employed, 29.1 grams of potassium fluoride, corresponding to one-half the molar weight may be used in proportion to 177.2 grams of antimony trifluoride, or up to 116.3 grams of potassium fluoride, corresponding to two molar weights thereof may be used, although preferably 58.15 grams of potassium fluoride are used for each 177.2 grams of antimony trifluoride.

In the use of a soluble fluoride salt, as sodium fluoride, or potassium fluoride, in a water solution with antimony trifluoride, the common fluorine ion is believed to drive back the dissociation of the antimony trifluoride and to prevent hydrolysis with water, thus preventing the formation of the oxy compound, or antimony oxy fluoride, although admittedly little is known about the mechanism of the reaction. It is possible that the formation of an opalescent material in water when antimony trifluoride alone is dissolved therein and is permitted to stand for some hours is due to some colloidal action and that the opalescent material is some form of a colloid, and that the action of sodium fluoride or potassium fluoride when added to a solution of antimony trifluoride is a peptizing action which dissolves or prevents the formation of a gel or colloid. This appears in fact to be the more probable explanation of the action of sodium fluoride and potassium fluoride than the theory of driving back dissociation or hydrolysis, from the observation that the same effect of preventing the formation of the opalescent material in a water solution is produced by the addition of ammonium sulphate to the solution of antimony trifluoride, which does not have a common ion, or by the use of "De Haen salt," which is a double salt of antimony trifluoride with ammonium sulphate.

In place of antimony trifluoride alone in a water solution for the impregnation of the fabric or textile material to be flameproofed, "De Haen salt" may be used alone in water solution, the molar weight of 309 grams of the double salt being equivalent to 177 grams of antimony trifluoride. The equivalent of a 20% solution of antimony trifluoride is 34.9 grams of "De Haen salt" per 65.1 ml. of water. The "De Haen salt" is particularly effective in preventing the formation of the opalescent effect in water which takes place after a period of some hours when antimony trifluoride is used alone, and in the case of "De Haen salt," the effect is believed to be due to a "peptizing" action, preventing the formation of a gel in the solution.

The action of preventing the formation of the opalescent effect or formation of antimony oxy fluoride may also be produced by the addition of both sodium fluoride, or other soluble fluoride, and ammonium sulphate, or "De Haen salt." For example, for each 177 grams of antimony trifluoride, 42 grams of sodium fluoride and 132 grams of ammonium sulphate may be used in a water solution to provide equi-molar amounts. It will be understood that the concentrations and the proportions of the added salts may be varied to a considerable extent, as will be understood by those skilled in the art, depending on the type of textile material to be treated and the length of time that the solution is to remain in contact with the textile material and other varying factors. A 20% solution of antimony trifluoride and added salts may contain 20 grams of antimony trifluoride with 2.37 grams of sodium fluoride and 7.5 grams of ammonium sulphate per 70.1 ml. of water, the added salts being in the proportions of half their molar weights in this case and serve fully to prevent the opalescent or clouding effect in the solution.

When the antimony trifluoride and sodium are used in equi-molar proportions, so as to correspond substantially to a double salt, theoretically, of sodium fluoride and antimony trifluoride, which is preferably employed in carrying out the impregnation of cotton fabrics in accordance with this invention, solutions of this mixture of salts in water can be made which contain 30% of antimony trifluoride and sodium fluoride, which is equivalent to a content of about 20% of antimony trioxide. The anhydrous double salt, or mixture, contains approximately the equivalent of 65% of antimony trioxide. Solutions of these salts or the "double salt" do not hydrolyze and attack the cotton fabric, as is the case with so many other metallic solutions. After the fabric which has been impregnated with this solution has been dried, there is no damaging effect exerted on the fabric by the dried antimony trifluoride and sodium fluoride enclosed within the body of the fabric and, moreover, the dried compound or mixture is not hygroscopic, as is the case with antimony trifluoride when used alone. Antimony trifluoride when used alone for the impregnation of textile materials in water solution, has the disadvantage of being hygroscopic and if the compound is not immediately insolubilized by the formation of antimony trioxide after the fabric or textile material has been dried, the fabric or textile material has been dried, the fabric or textile material becomes very damp through absorption of atmospheric moisture and exudates form on the surface of the fabric or textile material, which when the material is subsequently treated in a sodium carbonate solution to form the antimony trioxide from the trifluoride, unsightly blobs are formed on the surface of the fabric or textile material which disfigures the fabric or textile and produces an unsightly appearance of the dye base. The use of the equal molar mixture of antimony trifluoride and sodium fluoride overcomes the difficulty of impregnation with antimony trifluoride alone so as to render it unnecessary to speedily dry and treat the dried fabric or textile with the precipitating medium to overcome the formation of blotches or blobs. The fabric or textile when impregnated with a solution of the equal molar mixture of antimony trifluoride and sodium fluoride may be rolled up after drying without deleterious effects on the fabric or textile material even through it remains untreated with alkali carbonate to insolubilize the antimony compound by the formation of antimony trioxide therefrom for a considerable period of time, even for days.

The antimony trioxide which is produced by the reaction between the antimony trifluoride and the alkali metal insolubilizing bath is in an exceedingly fine state of subdivision, the antimony trioxide particles measuring on the average less than one micron, which is in fact on the borderline of the colloidal state of matter,

and adhere firmly to the cells of the fibers of the fabric or textile material so as not to wash out when the material is washed with water. Furthermore, the trioxide particles are so firmly fixed to the textile material that it is not removed by mechanical means, such as brushing or beating, to which the fabric or textile material may be subjected. Because of the exceedingly fine state of subdivision of the precipitated antimony trioxide, which may be considered as "semi-colloidal," the surface energies of the precipitated material are so great as to render it a strong absorbent, a fact which is of considerable importance in any dyeing operation in which the fabric or textile material, so treated, may be involved. It is well known that precipitated antimony trioxide is fusible at a temperature of 1212° F. (656° C.) and may be sublimed at a temperature of 2822° F. (1550° C.). The precipitated form of antimony trioxide is more reactive than the commercial form of the trioxide, which is usually produced by subliming the metal and then condensing the fumes.

It will be understood that when a mixture of antimony trifluoride and sodium fluoride or potassium fluoride, or other mixture herein described, is used in place of antimony trifluoride alone, the subsequent steps of the process are substantially the same as those above described in the use of antimony trifluoride alone. It is to be understood also that the proportions and concentrations of the antimony trifluoride solutions are given by way of example and may be varied as will be obvious to those skilled in the art without departing from the spirit or scope of the invention as defined in the claims annexed hereto.

In order to provide suitable impregnation of most fabrics and textile materials, relatively dilute solutions of antimony trifluoride or admixtures of antimony trifluoride with sodium fluoride, potassium fluoride, ammonium fluoride, or other soluble fluoride salt, or a salt such as "De Haen salt" ($SbF_3(NH_4)_2SO_4$), or mixtures of "De Haen salt" with antimony trifluoride, may be used, the soluble added fluoride salt or the ammonium sulphate in the "De Haen salt" serving to "peptize" the antimony trifluoride water solution, in that it apparently acts or serves to dissolve or liquefy the gel or opalescent material which would be formed in a water solution of antimony trifluoride without the added peptizing salt.

It will be understood also that antimony trifluoride, despite its relatively corrosive character toward glass in the presence of moisture, and toward most metals which are more or less corroded by the release of hydrofluoric acid in the presence of moisture, this material does not attack cellulose and does not weaken fabrics or textile materials when subjected to a solution of antimony trifluoride, particularly in the presence of a soluble fluoride salt dissolved therewith, which apparently serves to prevent decrease in tensile strength in the fabric or textile material even though it is exposed to the solution for relatively long periods of time.

It will be further understood that by the treatment and method herein described, the antimony trioxide is distributed in situ throughout the body of the fabric or textile material treated and results in a soft, flexible product in which the antimony trioxide particles are semi-colloidal and of a particle size of about one micron or slightly less, on the average, and are firmly bound to the fibers of the fabric or textile material, which after drying does not dust out and cannot be removed by soaking or washing in water or soap solutions. The precipitation of the antimony trioxide in the manner and form described does not substantially change the color of fabric or textile material, which after treatment is resistant to weak acids and alkalis.

The term "alkali carbonate" as employed in the claims is intended to mean a soluble carbonate or bicarbonate and to include sodium carbonate, sodium bicarbonate, potassium carbonate, potassium bicarbonate, ammonium carbonate and ammonium bicarbonate.

Having thus described the invention, what is claimed as new is:

1. A method for the production of a flame-resistant textile material comprising impregnating the textile material with a water solution of antimony trifluoride and a peptizing salt selected from the group consisting of sodium fluoride, potassium fluoride, and ammonium fluoride, to prevent the formation of opalescent material in the solution on prolonged standing, drying the impregnated textile material, treating the dried textile material with a water solution of an alkali carbonate to form antimony trioxide in the textile material, washing the textile material with water to remove soluble salts therefrom and drying the textile material.

2. A method for the production of a flame-resistant textile material comprising impregnating the textile material with a water solution containing from about twenty to thirty per cent of antimony trifluoride and a peptizing salt selected from the group consisting of sodium fluoride, potassium fluoride, and ammonium fluoride in the proportion of from about one-half mol to about two mols of the peptizing salt for each mol of antimony trifluoride, drying the impregnated textile material, treating the dried textile material with a water solution of an alkali carbonate to form antimony trioxide in the textile material, washing the textile material with water to remove soluble salts therefrom and drying the textile material.

3. A method for the production of a flame-resistant textile material comprising impregnating the textile material with a water solution of antimony trifluoride and a peptizing salt selected from the group of soluble fluoride salts consisting of sodium fluoride, potassium fluoride and ammonium fluoride in the proportion of from one-half mol to two mols of the fluoride salt for each mol of antimony trifluoride, to minimize the hydrolysis of the antimony trifluoride and to prevent the formation of opalescent material in the solution on prolonged standing, drying the impregnated textile material, treating the dried textile material with a water solution of an alkali carbonate to react with the antimony trifluoride in the textile material to precipitate antimony trioxide in situ therein, washing the textile material with water to remove soluble salts therefrom and drying the textile material.

4. A method for the production of a flame-resistant textile material comprising impregnating the textile material with a water solution containing from about twenty to thirty per cent of antimony fluoride and from about 4.74 to 7.12 per cent of sodium fluoride to prevent the formation of opalescent material in the solution on prolonged standing, drying the impregnated textile material, treating the dried textile material with a water solution of an alkali carbonate to form

antimony trioxide in the textile material, washing the textile material with water to remove soluble salts therefrom and drying the textile material.

5. A method for the production of a flame-resistant textile material comprising impregnating the textile material with a water solution containing from about twenty to thirty per cent of antimony trifluoride and from one-half mol to two mols of sodium fluoride for each mol of antimony trifluoride, the textile material being contacted with the impregnating solution for a sufficient time to obtain thorough impregnation, drying the impregnated textile material, contacting the dried textile material with a water solution of sodium carbonate to form antimony trioxide in the textile material, washing the textile material to remove soluble salts therefrom with water and drying the textile material.

6. A method for the production of a flame-resistant textile material as defined in claim 5 wherein the washed and dried textile material is

further passed through a solution of chlorinated paraffin to prevent propagation of a zone of creeping combustion within the textile material and then drying the fabric to remove solvent from the chlorinated paraffin solution.

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