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FIRE RESISTANT COMPOSITION AND
FABRIC

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The subject of invention is an improvement in the impregnation of cellulosic fibers and fabrics and similar relatively inflammable fibers and fabrics; to render them resistant to the action of fire, water and deterioration by mildew or weathering and the invention further relates to the resulting impregnated product and to the composition with which it is impregnated. This application is in part a continuation of our co-pending application, Serial #290,752, filed August 18, 1939.

The material to be treated which is of most general occurrence and of most importance commercially is cotton duck and the standard, with which the product must comply in order to be acceptable commercially is Federal Specification CCC-D-746, February 1939, for Duck, cotton, fire, water, and weather resistant.

An important provision of this specification relating to the subject of the invention is that ten specimens 2 x 12½ inches shall be cut with their long dimension extending in the direction of the warp, and ten with their long dimension extending in the direction of the filling, one half of each lot be subjected to fire tests by application of a specified Bunsen burner flame and the other half to accelerated weathering. The average length of char must not be more than 3½ inches and no specimen of the treated duck shall continue flaming for more than two seconds after the burner flame is withdrawn. The specimens subjected to weathering must thereafter meet the requirements as to fire resistance and specified requirements as to flexibility and strength. It is the main purpose of the treatment herein-after described to produce on an economical basis treated duck which shall comply with the above specification. Herein, fireproof, and weather resisting, indicates compliance with said standard.

The treatments described and claimed in United States Patents Number 2,172,698; and Number 2,194,690, applied to cotton duck, produce fabrics which conform to said standard, but it is found difficult to treat the heavier weights of duck as numbers 2, 4 and 6, according to the methods and formulas of said patents, and to thus obtain without excessive increase in weight, of the fabric a product which will comply with said specification, and at reduced cost.

An object accomplished by the invention being described is the provision of a method and formula adapted to the treatment of the heavier grades of cotton duck which will serve to produce with uniformity and without undue increase in weight a satisfactory fire, water and weather resistant duck in the heavier grades of material above identified. These formulas and method are also adapted to the treatment of the lighter grades of duck and to the treat-

ment of cellulose fibers and the like in various forms with equally satisfactory results. In all instances and with all available weights of untreated material the product complies with the above standard and is highly satisfactory.

This fire resistant duck which represents the object or accomplishment of the invention is adapted, like that of the above cited patents, to use as a covering for bus and truck tops and for tents and tarpaulins, awnings, cab curtains, boat covers, screens, canopies, roofing, decking and side walls for buildings, etc., and the treated fiber may be used in rope and cord, and as caulking material and fiber padding and it is of particular importance that the treated fabric is almost equally elastic, pliable, flexible and quite as strong as the untreated fabric. In fact, all of these qualities are maintained or increased by the treatment.

In the treating composition used in the practice of the invention a halogenated organic material is employed as the base and as a carrier for solid ingredients. The chlorinated materials being most easily obtainable on a commercial scale have been used almost exclusively in actual practice. The solids include as an important constituent substantially water insoluble finely divided compounds of arsenic, antimony or bismuth which are also substantially insoluble in the chlorinated organic materials and in the solvents of said materials. These solids are preferably mixed or ground with the chlorinated material and deposited in and on the fabric, the chlorinated material serving as a base. In this manner a fireproofing treatment is accomplished which is highly efficient and particularly adapted to the heavy weight duck in the fireproofing of which difficulty has previously been encountered as well to the lighter materials. The present treatment in fact represents an important advance in the fireproofing of cellulosic fabrics in general.

It has been found in actual practice that chlorinated carriers of extremely high chlorine content, used in this way with the solids suggested, give a very satisfactory fire resistant test but the fabric thus treated, most particularly in the heavier weights, and notably where the chlorinated material is mainly chlorinated paraffin, has been subject to the defect of objectionable stiffness at the low temperatures incident to winter weather in the temperate zone. This property makes it less desirable where flexibility at low temperature is required but there are various uses to which the fabric may be applied in which flexibility at such low temperatures is unimportant. While the low temperature stiffness can be overcome by use of plasticizers, it is found that so much of the plasticizer may be

required that a tacky finish results, together with undue expense of treatment.

In the practice of the invention, while much more highly halogenated materials are usable, it is found that halogenated carriers of which chlorinated paraffin is a good example, approximately 40% chlorine content being preferred give a satisfactory result when applied to the fabric in combination with finely divided water insoluble compounds of arsenic, antimony and/or bismuth, and that the fire, water and weather resistant fabrics thus produced have the desired degree of flexibility at low temperatures and that they meet the other requirements of the said Federal Specification CCC-D-746 above set forth. In addition to the increased flexibility, it is found that with this practice the cost of the treatment, particularly of the heavier grades of duck, is considerably reduced as hereinafter fully explained and the product is improved.

The chlorinated material, when heated to the combustion temperature of the fabric, gives off a chlorine containing gas mainly hydrogen chloride which or part of which combines with the metal, forming volatile compounds which exclude oxygen and practically eliminate flaming.

The compositions to be described are mechanical mixtures but it is believed that chemical compounds can be synthesized from chlorinated paraffin or other chlorinated organic substances with arsenic, antimony or bismuth which would flameproof, waterproof, and weatherproof cellulosic fabrics and other cellulosic materials. In either case the important point is that the product forms under the heat of the flame a fireproofing compound of arsenic, antimony or bismuth in an amount sufficient to render the fabric, to which the compound is applied, non-inflammable.

An important factor in the cost reduction provided by the treatment herein described is the fact that with these compounds of arsenic, antimony and bismuth used in combination with chlorinated carriers, it is possible to reduce the amount of solid material deposited per unit of weight of the fabrics, of the various counts or numbers, by impregnation according to the invention, and still meet the test requirements above recited.

The following examples show approximately the increase in weight of the different numbers or counts of cotton duck when treated in accordance with the invention being described, so as to possess the qualities required in and by said Specification CCC-D-746.

No. of duck	Weight prior to treatment	Percent gain in weight from treatment, when using methods disclosed herein	Maximum increase allowed under Specification CCC-D-746
	Oz. sq. yd.	Percent	Percent
2.....	27.12	26	60
4.....	23.93	29	60
6.....	20.74	32	70
8.....	17.55	35	70
10.....	14.35	40	70
12.....	11.16	44	80
8 oz. Army.....	9.85	44	80
10 oz. Army.....	12.29	41	70
12 oz. Army.....	14.77	38	70
15 oz. Army.....	18.48	37	60

These percentages are approximately only and subject to considerable variation with the change of formula due to differences of color desired, different degrees of flexibility required, thickness or weight of ducks treated, availability of the dif-

ferent constituents, and the like. The increase permitted by the specification is about twice that which actually occurs in the practice of the invention.

It is also found that it is possible to produce awning fabrics which are striped or otherwise colored with the identical oleo resinous paints used in the awning industry which fabrics thus striped or colored will satisfy the said Specification. In practice, the duck is treated with a fire-resisting compound, according to the following disclosure, the fabric is then delivered to the awning striper who applies the conventional oleo resinous paint according to the usual procedure.

More fully defined the carrier or base, in accordance with the preferred practice of the invention, consists of a halogenated organic material, which is solid or semi-solid at normal room temperatures, is substantially insoluble in water, and of a waterproofing nature, and it is such that it will release at or near the combustion temperatures of the fabric or other cellulose being treated, a halogen containing gas which will immediately combine with certain of the other ingredients to be described producing one or more flameproofing or retardant compounds or gases which impart to this fabric a fire resistant quality whereby it meets the requirements of the above specification.

Chlorinated paraffin of approximately 40% chlorine content is considered most desirable for this purpose, chlorine compounds being more generally available at low prices than those of the other members of this group, it is, however, regarded as feasible to use fluorine or bromine derivatives of paraffin or of other suitable organic materials when available. The sources of chlorine and chlorine compounds are so much more generally distributed and chlorine is so much cheaper that reference to the other elements in this group is almost unnecessary. Various halogenated organic compounds may be used.

Examples of halogenated organic materials which may be used instead of halogenated paraffin are halogenated fish oil, halogenated cottonseed oil, halogenated cocoanut oil, halogenated soy bean oil, halogenated peanut oil, halogenated vinyl resins, halogenated linseed oil, halogenated asphalt and halogenated asphalt oils, etc., 30% to 50% chlorination being preferred.

Hereinafter "chlorinated" will be used for convenience, instead of "halogenated," the significance of the term having been discussed. The chlorinated naphthalenes and chlorinated diphenyls are found to be not as effective as chlorinated paraffin for the purposes of the invention. Halogenated or more particularly chlorinated rubber gives a stiff fabric which would not be desirable, but small quantities of chlorinated rubber used with other chlorinated materials as chlorinated paraffin to condition the fabric, preventing tackiness, give satisfactory results.

The chlorinated material in addition to the other purposes which it serves should be adapted to act as a binder or carrier for the other solids contained in the composition to form a fire extinguishing deposit in the fabric when applied to the fabric in sufficient quantities.

The solids which we have found satisfactory for this purpose include compounds of arsenic, antimony and bismuth. Metallic compounds known to be satisfactory for this purpose are Sb_2O_3 ; Sb_2S_3 ; As_2O_3 ; As_2S_3 ; Sb_2S_5 ; Bi_2O_3 ; Bi_2S_3 . The chlorides, sulphates and nitrates of these metals are less desirable on account of their tendency to attack the fabric. The antimony

oxides and sulphides are preferred. Arsenic trioxide is a cheap material which is poisonous but otherwise suitable. Bismuth compounds are at present too expensive for this purpose. The higher oxides as the pentoxide of these metals are less desirable on account of the excess of oxygen. The double salts such as the arsenate and the antimonates of lead are also usable.

Any compound of these metals which is not injurious to the fabric may be used provided that it is substantially water insoluble and will when intimately mixed in finely divided form with a halogenated organic material as chlorinated paraffin and applied to cotton duck, in proportions within the maximum limits in the above-weight table, render the fabric fire resistant according to the requirements stated as to Specification CCC-D-746.

The exact percentage of chlorine in the organic compound is ultimately controlled by the proportions necessary to a flameproofing composition which will, when applied to any particular fabric, satisfy the requirements of the above recited specification without unduly increasing the weight of the fabric. While 40% chlorination has been found satisfactory, higher percentages, up to 60% or even 70% may be used where freezing temperatures are not encountered and flexibility at low temperatures is not important, or use of plasticizers is feasible, and the degree of chlorination may be reduced with some loss of effectiveness, 20% being regarded as usable where increased weight of the deposit is not objectionable. The foregoing table gives the minimum increase attained by the invention and the maximum increase permitted. The formulas herein are found to comply with these conditions and to comply approximately with the table of weight increase which appears above. This composition is widely different from any painting composition as painting compositions are apt to be injurious to cotton fabric and have drying qualities which would make the fabric too stiff for use.

In the practice of the invention to produce a fire and water resistant fabric which retains substantially its full flexibility at and below freezing temperatures, it has been found most satisfactory to use as the chlorinated ingredient of this composition a chlorinated organic material, examples of which are 30% to 50% chlorinated paraffin wax or oil; 30% to 50% chlorinated fish oil; 30% to 50% chlorinated coconut oil; 30% to 50% chlorinated peanut oil; 30% to 50% chlorinated corn oil. Chlorinated naphthalene, on account of its waxiness at normal temperatures, is notably inferior to chlorinated paraffin. Chlorinated rubber may be used with these materials as a modifier to prevent tackiness, other materials suitable to this purpose being herein-after discussed.

The chlorinated compounds listed in this paragraph do not substantially reduce the flexibility of the impregnated fabric even at freezing and lower temperatures and are so highly chlorinated as to give the fireproofing required by Specification CCC-D-746 without increase of weight beyond that permitted by standard requirements of which said specification contains a statement.

In order to impregnate the fabric or fibers with the finely divided solids and the chlorinated materials, it is desirable to have a "solvent" for the chlorinated material. The term solvent is used herein to designate a solvent which suitably dis-

solves the chlorinated materials to be used. Examples are mineral spirits; coal tar solvents; carbon tetrachloride; xylene, aromatic solvents; ethyl acetate and certain proprietary solvents as Stoddard solvent, Solvesso #2, and Solvesso #3, are also usable. (Stoddard solvent is gasoline, and Solvesso #2 and #3 are mixtures of aromatic and straight chain hydrocarbons.) While the formulas specify 40% to 55% solvent, this is variable to suit the treatment desired.

In addition to the chlorinated organic materials the solvent therefor and the arsenic, antimony or bismuth compound, the treating composition requires a pigment which is selected to impart the desired color to the composition or to the treated fabric but which serves the more important function of protecting the chlorinated organic material from action of sunlight encountered in normal weather exposure. In this connection, it will be understood that these chlorinated compounds are subject to quick deterioration in sunlight accompanied by a release of hydrogen chloride and a consequent deterioration of the fabric by action of the hydrogen chloride thus released in active condition. Hence the protective action of the pigment is of great importance and the pigment must be adapted to cut off sufficient of the light to protect the chlorinated material. Any suitably opaque and sufficiently inert pigment will serve this purpose, substantial insolubility of the pigment in water being regarded as necessary to a waterproof fabric. The following are examples suitable to this use; sulphate of lead; zinc oxide; titanium oxide; antimony oxide; litharge; red lead; all of the various iron oxide pigments; white lead; chrome yellow; cadmium yellow; antimony sulphide; arsenic sulphide; ultra-marine blue; Prussian blue; chrome green; chromium oxide; lamp black; carbon black; and others. Appropriate compounds of arsenic, antimony and bismuth may be used as part or all of the pigment in addition to serving their fireproofing functions. The pigments as well as the filler should be substantially insoluble in the chlorinated material and in the solvent therefor as well as in water.

The composition also includes, in some instances, a filler or extender. Examples of suitable fillers are barium sulphate, barium carbonate, silica, calcium sulphate, clay, asbestos, and other relatively inert materials which increase the solid content of the composition.

The composition may contain a small quantity of modifier as chlorinated rubber, ethyl cellulose or a drying oil as tung oil, or soya bean oil. A very small percentage of the total including the solvent is sufficient where any is used. The purpose of the modifier is to alter the condition of the product to give a dry non-tacky flexible, soil resistant fabric. With the most highly chlorinated materials a tricresyl phosphate may be used.

Any considerable proportion of linseed or other drying oil or oxidizing oil in unchlorinated form is undesirable as reducing the fireproofing effect of the composition and it is further regarded as highly objectionable because considerable proportions of drying oils as unchlorinated linseed oil reduce the flexibility of the impregnated fabric below that required by the above Specification. A degree of flexibility closely approximating that of the untreated fabric is an essential in a large majority of uses in which the treated fabric is employed.

In practice the chlorinated material, the metallic compound of arsenic, antimony or bismuth, the filler and the pigment are ground together in a paint mill or otherwise suitably mixed and the resulting paste is then dispersed by agitation in a solvent which may contain a small quantity of modifier; the practice being variable to suit conditions and the method and order of mixing being no part of the invention. The resulting compound is then applied to the fabric by dipping or in any suitable manner. The solvent is then removed by evaporation at normal room temperatures or by heat. A single dipping or passing of the fabric through the compound is sufficient. The accomplishment of fireproofing in and by a single treatment is important in its contribution to low cost production of the treated fireproof fabric.

It may be regarded as feasible, though not at present preferred, instead of using a solvent to emulsify the paste above described by agitation in water forming a fluid which may be applied to the fabric by dipping, or brushing, etc.

The following formulas are intended to indicate, in a general way, the possibility of variation in ingredients and proportions of the composition.

MATERIALS

Percentage by weight

	Black	Green	Orange	Terra cotta	White	Yellow	White
	Per cent 24	Per cent 24	Per cent 24	Per cent 24	Per cent 24	Per cent 24	Per cent 24
Chlorinated organic material							
Black antimony sulphide (Sb_2S_3)	10	10		10			
Golden antimony sulphide (Sb_2S_3)			10				
Black iron oxide	10			10			
Red iron oxide							
C. P. chrome green		10					
C. P. chrome orange			10				
Antimony oxide (Sb_2O_3 , mainly)					10		
Titanium dioxide					10		
Solvent	50	50	50	50	50	50	50
Arsenic sulphide (As_2S_3 , or As_2S_5)						10	
Arsenic oxide (As_2O_3)							10
C. P. chrome yellow						10	
White lead							10
Modifier or plasticizer	6	6	6	6	6	6	6

The proportion of chlorine in the organic material in the above formula should be sufficient to combine with the antimony, arsenic, or bismuth compound present, to release sufficient non-inflammable gas to protect the fabric according to the above Specification. Approximately 30% to 70% chlorination is regarded as effective within the weight limits of the foregoing table, giving a minimum of 26% to 44%, and in some instances to 55%, increase in weight of the fabric, and lower degrees of chlorination, down to 20%, may be used with less effective fireproofing, approximately 40% chlorinated material having been found most satisfactory in practice.

In the following formulas containing approximately 25% of antimony, arsenic, or bismuth compound, there is an excess of the antimony or arsenic or bismuth compounds which thus serves the purposes of a pigment, protecting the chlorinated material from light also acting as a filler. Such formulas are effective but not at present regarded as desirable from a commercial standpoint.

It is noted that a deposit which only partially permeates the thickness of the fabric may be

uniformly distributed over the area of the fabric and fully effective.

Percentages are on a weight basis

	Per cent
Chlorinated paraffin containing 30% to 50% chlorine	25
Commercial grade antimony trioxide (Sb_2O_3)	25
Petroleum solvent (Stoddard solvent)	50

This composition will enable a #2, weight per yard, cotton duck, containing 30.8% gain of solid materials by the above formula and a #8, weight per yard, cotton duck containing 37.8% gain of solid materials by the same formula, to satisfy the fire test of CCC-D-746 after removal of the solvent.

	Per cent
Chlorinated paraffin containing 30% to 50% chlorine	25
Arsenic trisulphide— As_2S_3	25
Stoddard solvent	50

This composition will enable #2 cotton duck containing 38% gain of solid materials by this formula, and a #8 duck containing 41% gain by the same formula to satisfy the fire test of CCC-D-746, after removal of the solvent.

	Per cent
Chlorinated paraffin containing 30% to 50% chlorine	25
Antimony trisulphide— Sb_2S_3	25
Stoddard solvent	50

This composition will enable a #2 cotton duck containing 42.7% gain of solid materials by this formula, and a #8 duck containing 42% gain by the same formula to satisfy the fire test of CCC-D-746, after removal of the solvent.

	Per cent
Chlorinated paraffin containing 40% chlorine	20
Antimony oxide— Sb_2O_3	7
Zinc oxide	1
Calcium carbonate	3
Yellow iron oxide	6
Lamp black	1
Black iron oxide	2
Chlorinated rubber containing 67% chlorine	5
Xylol	55.00

When the solid materials of the above formulation are deposited on the following weights of

duck in the above proportions to the extent of the following gains over the untreated weight of duck, the respective weights of duck will then satisfy the fire test of CCC-D-746.

Weight of cotton duck	Percent gain over untreated weight after removal of solvent
	Percent
#2.....	26
#4.....	29
#6.....	32
#8.....	35
#10.....	40
#12.....	44
15 oz. Army.....	37
12 oz. Army.....	38
10 oz. Army.....	41
8 oz. Army.....	44

Chlorinated paraffin containing 40% chlorine	Per cent
Ethyl cellulose.....	23.50
Antimony oxide— Sb_2O_3	1.50
Zinc oxide.....	7.09
Calcium carbonate.....	1.01
Yellow iron oxide.....	3.00
Lamp black.....	6.00
Black iron oxide.....	0.90
Ethyl acetate.....	2.00
	55.00

Percent gain of solid materials of the above which will enable duck so treated to pass the fire test of CCC-D-746, after removal of the solvent is:

	Per cent
On #8 cotton duck.....	55
On #2 cotton duck.....	40

Chlorinated paraffin containing 40% chlorine	Per cent
Black antimony trisulphide— Sb_2S_3	30
Black iron oxide.....	12.5
Calcium carbonate.....	12.5
Stoddard solvent.....	5.0
	40.0

The solid materials of the above when deposited on duck in the above proportions and in the following amounts will render the duck capable of passing the fire test of CCC-D-746.

	Percent gain over untreated weight after removal of solvent
	Percent
On #8 cotton duck.....	53
On #2 cotton duck.....	45

Chlorinated paraffin containing 40% chlorine	Per cent
Chlorinated rubber containing 67% chlorine.....	21
Sb_2O_3 bismuth trioxide.....	9
Zinc oxide.....	25
Xylol.....	5
	40

The solid materials of the above when deposited on duck in the above proportions and in the following amounts will render the duck capable of passing the fire test of CCC-D-746.

	Percent gain over untreated weight after removal of solvent
	Percent
On #8 cotton duck.....	70
On #2 cotton duck.....	47.5

	Per cent
Chlorinated paraffin containing 40% chlorine.....	21
Chlorinated rubber containing 67% chlorine.....	9
Arsenic trisulphide— As_2S_3	25
Zinc oxide.....	5
Xylol.....	40

The solid materials of the above when deposited on duck in the above proportions, and in the following amounts will render the duck capable of passing the fire test of CCC-D-746.

	Percent gain over untreated weight after removal of solvent
	Percent
On #8 cotton duck.....	70
On #2 cotton duck.....	49

Chlorinated paraffin containing 40% chlorine	Per cent
Chlorinated rubber containing 67% chlorine.....	24
Golden antimony sulphurette— Sb_2S_5	6
Zinc oxide.....	25
Xylol.....	5
	40

When deposited on cotton duck in the proportions above the solid materials of this formula will enable the resultant fabric to pass the fire test of CCC-D-746, after removal of the solvent, when present in the following amounts.

	Percent gain over untreated weight after removal of solvent.
	Percent
#8 cotton duck.....	66.25
#2 cotton duck.....	46.25

Chlorinated paraffin containing 40% chlorine	Per cent
Chlorinated rubber containing 67% chlorine.....	21
Lead antimonate ($Pb_3(SbO_4)_2$).....	9
Zinc oxide.....	20
Calcium carbonate.....	5
Xylol.....	5
	40

Amount of solid materials of the above, in their given proportions, which will enable duck so treated to satisfy the flame resistant requirements of CCC-D-746, after removal of the solvent.

	Percent gain over untreated weight
	Percent
#8 duck.....	65.5

Chlorinated paraffin containing 40% chlorine	Per cent
Chlorinated rubber.....	21
Lead arsenate ($Pb_3(AsO_4)_2$).....	9
Zinc oxide.....	25
Calcium carbonate.....	5
Xylol.....	5
	40

	Percent gain over untreated weight after removal of solvent.
	Percent
#8 cotton duck.....	67.5
#2 cotton duck.....	48.6

When deposited on cotton duck in the above proportions the solid materials of this formula

will enable the resultant fabric to pass the fire test of CCC-D-746, after removal of the solvent, when present in the percentages stated. The last two formulas show that mixed metallic compounds of antimony, arsenic or bismuth may be used. As aforesaid various compounds may be used within the conditions and limitations previously stated.

	Per cent
Chlorinated paraffin containing 40% chlorine..	21
Chlorinated rubber containing 67% chlorine..	9
Arsenic trioxide—As ₂ O ₃	25
Xylol.....	40
Zinc oxide.....	5

Amount of solid materials of the above in their given proportions which will enable duck so treated to satisfy the flame resistance requirements of CCC-D-746, after removal of the solvent.

	Percent gain over untreated weight
	Percent
#8 cotton duck.....	70
#2 cotton duck.....	46

A composition which has been found satisfactory for the treatment of cellulosic fabrics for quick application in the field as by brushing and which imparts to the fabric when treated on both sides, standard fire, water and weather resistant properties, contains approximately 3% of chlorinated organic material of a nature which tends to condition the fabric preventing tackiness, chlorinated rubber being satisfactory, and 13% of other chlorinated organic material of a more flexible nature as chlorinated paraffin which may be of 40% chlorination, it being understood that the amount of chlorinated material and the degree of chlorination may be varied, one oppositely to the other, without considerable change of the amount of chlorine in the composition. In this formula, the proportions being by weight, the chlorinated material is about 55% or approximately one-half of the total solids, the other 45%, approximately the other one-half, totalling about 12% to 14% of the whole, being in finely divided form, uniformly mixed in the chlorinated material. These finely divided solids are substantially insoluble in water and in the solvent of the chlorinated material and in said material. They may comprise approximately 35% antimony oxide and 65% filler and pigment.

For the antimony oxide other compounds of antimony, arsenic or bismuth, which are substantially insoluble in water and are not injurious to the fabric may be substituted. The balance of these finely divided solids comprising about 7% or 8% of the entire composition is pigmental material and filler. The diluent and solvent, preferably comprises about 70%, i. e., about two-thirds and the solids about one-third of the whole composition.

It is also important that the composition shall dry by natural evaporation of the solvent and that the composition shall not contain any perceptible amount of unchlorinated drying or semi-drying oils as linseed oil, tung oil, fish oil, soya bean oil, etc., which dry by oxidation or by polymerization as with the aid of catalysts.

The pigments and fillers regarded as satisfactory include zinc oxide, calcium carbonate, hy-

drolyzed iron oxide, lamp black and black iron oxide.

The following formulas are found satisfactory where the use of a plasticizer is not objectionable or where low temperatures are not encountered.

	Black	Green	Orange	Terra cotta
	Percent	Percent	Percent	Percent
50% to 70% chlorinated paraffin.....	24	24	24	20
Tricresyl phosphate.....	6	6	6	5
Black antimony sulphide.....	6	6	6	7.5
Golden antimony sulphide.....	14	14	14	17.5
Black iron oxide.....	50	50	50	50
Red iron oxide.....				
C. P. chrome green.....				
C. P. chrome orange.....				
Solvent.....				

	White	Terra cotta
	Percent	Percent
50% to 70% chlorinated paraffin.....	24	24
Tricresyl phosphate.....	6	6
White arsenic (As ₂ O ₃).....	6	6
Red arsenic (As ₂ S ₃).....	14	14
White lead.....	50	50
Red iron oxide.....		
Solvent.....		

Formula using antimony oxide

	Per cent
50% to 70% chlorinated paraffin.....	24
Antimony oxide mainly (Sb ₂ O ₃) or other of the antimony, arsenic or bismuth compounds above disclosed.....	10
Pigment and filler.....	10
Plasticizer (optional).....	6
Solvent.....	50

According to the above formulas and the weight increase tables the impregnated fabric may be said to contain approximately 7% to 18% of its weight of chlorine and approximately 4% to 30% of its weight of metallic compound.

The product which may be regarded as the primary accomplishment of the invention is a fire, water and weather resistant fabric which retains substantially its full flexibility and also its tensile strength and particularly its fire resistant qualities for long periods exceeding the life of the untreated fabric. This treated fabric, according to the preferred practice of the invention, contains by coating and/or impregnation which may be accomplished in a single treatment, as by dipping, brushing, or spraying, a deposit of approximately one-fourth to three-fourths the weight of the fabric prior to treatment. The weight of the deposit is determined according to the weight and thickness and density of the fabric and the requirements to be met. The term untreated fabric as used herein refers to the fabric as it exists prior to treatment by impregnation.

These proportions are approximate and refer to the effective or essential weight of the deposit or percentage increase of the weight of the fabric to impart the standard resistive qualities to fabrics of different counts. The object of the invention is to impart to the fabric, with the minimum increase in weight and minimum expense standard fire, water and weather resistant qualities which are relatively permanent retaining the flexibility of the fabric and retaining or increasing its normal life. The percentages of increase given are with this object in view. The treatment may, however, be practiced to less advantage by increasing the weight of the deposit using the

same or similar compositions with or without a decrease in the fire resistant qualities. The practice of the fire resistant treatment with such changes is regarded as utilizing the teaching of the invention, as is also the reduction of the deposit or its effectiveness.

The deposit consists of the solids in the above formulas, the solvent which is generally computed at half the weight of the treating composition, being eliminated by evaporation from the fabric. To avoid repetition it is therefore noted that the formula for the treated fabric, i. e., the weight of each ingredient per yard, is directly deducible from the corresponding composition formula, using as factors the relative percentages of solids in the formulas after deducting the weight of the solvent, and applying to this, the percentage increase in weight per yard essential to the desired result according to the above table of weights and the weight per yard of the fabric can be determined.

An important accomplishment of the invention is the production preferably by a single treatment or a single step treatment, of a fireproof cotton or similar fabric which is standard in that it conforms to the prescribed tests for such material and retains its full flexibility, the fireproofing quality being attained without undue increase in weight and combined with resistance to action of water and weather whereby these combined qualities are retained for a long period.

We have thus described in various forms a treated fabric and a treating composition and a method of producing a treated fabric having standard fire, water and weather resistant qualities, the description being specific and in detail in order that the manner of operating, applying and using the invention may be understood. However, the specific terms herein are used descriptively rather than in a limiting sense, the scope of the invention being defined in the claims.

What we claim as new and desire to secure by Letters Patent is:

1. A composition for use in the impregnation of cellulosic fabrics and the like to give them fire, water and mildew resisting qualities, comprising a substantial proportion of an organic material selected from the group consisting of chlorinated paraffin, chlorinated paraffin oil, chlorinated fish oil, chlorinated vegetable oils, chlorinated rubber, chlorinated tung oil; the same being from about 20% to about 70% chlorinated and having water-proofing properties, and being adapted to decompose giving off chlorine containing gases at the combustion temperatures of said cellulosic materials, and a substantial proportion of metallic compound which is not injurious to cellulosic fibers and is selected from the group consisting of antimony oxide, antimony sulphide, arsenic sulphide, arsenic oxide, bismuth oxide and bismuth sulphide, a plasticizer, a volatile solvent and a quantity of opaque material sufficient to protect the chlorinated material from decomposition by light the composition having a high degree of penetration and being adapted to form a deposit of a flexible nature when dried whereby fabric treated with this composition will retain its full flexibility.

2. A compound for the treatment of cellulosic fabrics by impregnation to give them fire, water and mildew resisting properties which comprises approximately 25% of organic material which is from about 20% to about 70% chlorinated and has waterproofing properties and is adapted to give off chlorine containing gases at combustion

temperatures of said fabric, approximately one-fourth of this amount of antimony oxide, approximately 15% of black iron oxide, the remainder comprising a solvent adapted to be eliminated from the fabric by evaporation, the organic chlorinated materials retaining a high degree of flexibility after evaporation of the solvent and through an indefinitely long period of exposure.

3. A composition for use in the impregnation of fabrics and other cellulosic materials to give them fire, water and mildew resisting properties without appreciable reduction of the flexibility of the fabric, the same comprising substantial proportions of organic materials, which are from about 20% to about 70% chlorinated and having water-proofing properties, and substantial but lesser proportion of a compound selected from the group consisting of compounds of arsenic, antimony and bismuth which are substantially insoluble in water, which are also substantially insoluble in the solvent for the chlorinated material and are not injurious to the fabric, and which decompose at said combustion temperatures forming with the products given off by said chlorinated materials combustion retarding gases, and a proportion of opaque material sufficient to protect the chlorinated material from decomposition by light and a volatile solvent for the chlorinated material.

4. A composition for use in the impregnation of cellulosic fabrics and the like, to give them fire, water and mildew resisting qualities, comprising a substantial proportion of an organic material, the same being from about 20% to about 70% chlorinated and adapted to decompose giving off chlorine containing gases at the combustion temperature of said cellulosic materials, and a substantial proportion of metallic compound selected from the group consisting of compounds of arsenic, antimony and bismuth and which is not injurious to the cellulosic fibers and is substantially insoluble in water and in the solvent of said chlorinated material, and which decomposes at said combustion temperatures forming with said chlorine containing gases fire retarding gases, a volatile solvent for the chlorinated material and a quantity of opaque material sufficient to protect the chlorinated material from decomposition by light, the composition having a high degree of penetration and being adapted to form a deposit of a flexible nature when dried whereby the fabric treated with this composition retains substantially its full flexibility.

5. A cellulosic fabric having fire, water and mildew resisting properties to a high degree, the same containing a deposit of a substantial proportion of organic material which is from about 20% to about 70% chlorinated and has water-proofing properties and gives off chlorine containing gas at the combustion temperatures of said fabric, said chlorinated material containing mixed therein in finely divided form a metallic compound selected from the group consisting of compounds of arsenic, antimony and bismuth which are not injurious to the fabric and are substantially insoluble in water and in volatile solvents of said chlorinated material and which decompose at said temperatures forming with said gas a fire retarding gas, the deposit containing sufficient of opaque material to protect the chlorinated material from decomposition by light, evaporation and the fabric retaining substantially its full original flexibility and its full fireproofing properties after long exposure to the weather.

6. A composition for use in the impregnation of fabrics and other cellulosic materials to give them fire, water and mildew resisting properties without appreciable reduction of the flexibility of the fabric, the same comprising substantial proportions of organic materials, which are from about 20% to about 70% chlorinated and having waterproofing properties, and substantial but lesser proportion of a compound selected from the group consisting of antimony oxide, antimony sulphide, arsenic sulphide, arsenic oxide, bismuth oxide and bismuth sulphide, which are substantially insoluble in water, which are also substantially insoluble in the solvent for the chlorinated material and are not injurious to the fabric, and which decompose at said combustion temperatures forming with the products given off by said chlorinated materials combustion retarding gases, and a proportion of opaque material sufficient to protect the chlorinated material from decomposition by light and a volatile solvent for the chlorinated material.

7. A composition in accordance with claim 3 in which the organic material is from about 30% to about 50% chlorinated.

8. A composition in accordance with claim 3 in which the organic material is from about 50% to about 70% chlorinated, the composition further incorporating a plasticizer.

9. A composition in accordance with claim 3 in which the organic material comprises from about 20% to about 40% of the composition.

10. A cellulosic fabric impregnated in accordance with claim 4 and in which the chlorine of the impregnating compound comprises from about 7% to about 18% of the weight of the fabric.

11. A cellulosic fabric impregnated in accordance with claim 4 and in which the metal compound comprises from about 4% to about 30% of the weight of the fabric.

12. A composition in accordance with claim 3 in which at least a part of the opaque material comprises zinc oxide.

13. A composition in accordance with claim 3 in which the organic material is from about 30 to 50% chlorinated and in which at least a part of the opaque material comprises zinc oxide.

EDWIN C. CLAYTON.
LAWRENCE L. HEFFNER.

CERTIFICATE OF CORRECTION.

Patent No. 2,299,612.

October 20, 1942.

EDWIN C. CLAYTON, ET AL.

It is hereby certified that error appears in the printed specification of the above numbered patent requiring correction as follows: Page 7, second column, line 73, claim 5, strike out the word "evaporation"; page 8, second column, lines 9 and 14, claims 10 and 11, for "claim 4" read --claim 5--; and that the said Letters Patent should be read with this correction therein that the same may conform to the record of the case in the Patent Office.

Signed and sealed this 29th day of December, A. D. 1942.

(Seal)

Henry Van Arsdale,
Acting Commissioner of Patents.

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