

UNITED STATES PATENT OFFICE

2,477,336

COATED COTTON FABRIC

Alfred J. Jennings, Bridgeport, Conn., assignor to
E. I. du Pont de Nemours & Company, Wil-
mington, Del., a corporation of Delaware

No Drawing. Application July 14, 1944,
Serial No. 545,007

3 Claims. (Cl. 117-143)

1

This invention relates to a method for preventing deterioration of cotton fabrics from exposure to steam and more particularly to coated cotton fabrics which will withstand prolonged exposures to steam without impairment in the strength of the fabric.

Cotton fabric is seriously damaged with respect to its tensile strength when exposed to steam even for relatively short periods. This failure presents a serious industrial problem because of the frequent expensive and sometimes complicated replacements required. Such industrial installations as the plywood curing bag material employed in the process of manufacturing molded plywood described in U. S. Patent 2,276,004 are representative of the conditions of exposure to steam which are encountered by coated fabrics. In this utility the cotton fabric bags are usually coated with rubber or preferably a synthetic rubber compound such as neoprene, Buna or butyl rubber. Under normal operating conditions of the patented process, the fabric bag material is exposed to a mixture of steam and air at 75 pounds pressure and a temperature of 250-300° F. or steam at 40 pounds (287° F.), and it has been found that under such conditions, the fabric rapidly becomes "tenderized" and is easily torn, affording a useful life of as little as 10 cycles of 20 to 40 minutes each. In the present invention this important defect in currently available coated fabrics has been overcome and the life of such fabrics which are subjected to exposure to steam has been greatly prolonged.

This invention has as a primary object the provision of a cotton fabric which is highly resistant to steam for extended periods. Another object is the provision of a process for preventing deteriorating effects of steam on cotton fabric which is simple and economical. Another object is the provision of a process which will render coated cotton fabrics resistant to the deleterious effects of steam without adversely affecting the pliability and general durability of the fabric. Another object is the provision of an agent for rendering cotton fabrics resistant to steam which may be applied directly to the cotton fabric or incorporated in the coating for the fabric. Another object is the provision of a method for counteracting "tenderizing" of fabrics on exposure to steam which also functions as a curing means for rubber and synthetic rubbers in coating compositions containing these polymers. A further object is the provision of an organic coating composition which when applied to cotton fabrics prevents "tenderizing" of the fabric

2

when exposed to steam. Other objects will appear as the description of the invention proceeds.

These objects are accomplished in accordance with the present invention by treating the cotton fabric with a compound of magnesium or calcium or in the preferred embodiment of the invention, the oxide of the metal is introduced into an organic polymer containing coating composition as a protective agent, and this composition is then applied to the surface of a cotton fabric. When the coating composition contains a rubber or synthetic rubber, the oxide also functions as a curing agent.

The following examples are given by way of illustration only and no limitations are intended thereby except as indicated in the appended claims.

Example 1

	Per cent by weight
Neoprene (polymerized chloroprene) -----	50.0
Magnesium oxide -----	5.0
Wood rosin -----	5.0
Soft carbon black -----	26.0
Phenyl - alpha - naphthylamine (anti-oxidant) -----	1.5
Castor oil -----	2.5
Litharge -----	10.0
	100.0

The composition was prepared by charging the ingredients into a heavy duty kneading machine such as a Banbury mixer in accordance with procedures well known in the art and masticating until a homogeneous mass was secured.

This composition was applied to a fabric base consisting of cotton duck having the following characteristics:

	Warp	Fill
Count -----	90	x 49
Ply -----	2	x 3
Tensile strength (pounds per square inch) -----	185	175
Weight—9.5 ounces per 36" yard.		

The application of the coating was carried out in two steps. The fabric was first impregnated by spreading on its surface by means of a conventional double roll impregnator, a 30% solids solution of the composition in xylol and then passing it through a convection drying tunnel to give a total coating weight of about 3 ounces per square yard. A top coat of the composition at 50% solids solution in xylol was then applied on the surface by means of a conventional knife spreader in

3

multiple successive coats to give a total thickness (including the impregnant coating) of about .026 inch. If the fabric is to be used for plywood curing bags as in the present instance, one side of the fabric should be coated to have approximately twice as much coating as the other.

After removing the volatile xylol by passing the coated fabric through a convection drying tunnel following the application of each coat, the surfaces were dusted with talc, wound on a drum with Holland cloth liner and cured at 260° F. for 5 hours.

The excellent protective effects afforded by the magnesium oxide was demonstrated by results of tensile strength tests in a Scott tensile tester after exposures of the coated fabric to 4 cycles of 24 hours each at 80 pounds steam pressure, followed by 24 hours in air at room temperature after each steam exposure. The tensile strength of the coated fabric after this test was 188 pounds per square inch warp and 141 pounds per square inch fill showing that the original strength of the fabric had not been appreciably impaired.

However, when zinc oxide was substituted in the composition of Example 1 and applied to the same type of fabric using the same procedures, the tensile strength of the fabric after the 4 cycle steam exposure was so low that it could not be tested in the Scott tensile tester.

Example 2

	Per cent by weight
Butyl rubber (polymerized isobutylene) ..	50.00
Calcium oxide	2.00
Stearic acid	1.50
Medium thermal carbon black	44.75
Selenac* (rubber accelerator)	0.38
Mercapto benzothiazole (MBT)	0.25
Tetramethyl-thiuram-disulfide (Thiuram M)	0.37
Sulfur	0.75
	100.00

* Selenac is a rubber accelerator consisting of selenium diethyl dithiocarbamate.

The composition was prepared in accordance with the procedure described for Example 1.

An impregnating coating prepared by dissolving this composition in gasoline to 30% solids was applied to a cotton duck fabric as described in Example 1 to a coating weight of 1 ounce per square yard on each side of the fabric.

A top coating was prepared by dissolving the composition of Example 2 in gasoline to 40% solids and applied over the impregnant, first coating on one side with 5 coats and drying as in Example 1 to give a coating weight of about 3 ounces per square yard. The opposite side was coated with 2 coats of the same solution to a coating weight of about 2 ounces, dried as before and then calender-coated with 1 coat of the unthinned Example 2 composition to a total thickness of about .026 inch. The coated fabric was then cured as in Example 1.

The results of steam exposure tests carried out in accordance with the procedure of Example 1 gave a tensile strength of 175 pounds per square inch warp and 149 pounds per square inch fill showing that calcium oxide likewise functions as a protective agent against the deteriorating effects of exposure of cotton fabric to steam. If zinc oxide is substituted for the calcium oxide in this example and fabric coated with such compositions and exposed to steam for the same num-

4

ber of cycles, the fabric becomes so "tenderized" that no determinations of tensile strength can be made on the Scott tensile tester.

Example 3

	Per cent by weight
Buna S (butadiene-vinyl benzene co-polymer)	50.00
Brown factice (sulfonated vegetable oil) ..	7.50
Stearic acid	0.50
Semi-reinforcing carbon black	37.62
Magnesium oxide	2.50
2 mercapto-thiazoline (rubber accelerator) ..	0.63
Polyvinyl butyraldehyde-aniline condensation product (accelerator)	0.25
Sulfur	1.00
	100.00

An impregnating coating and a top coating composition was prepared from this example by dissolving in gasoline as in Example 2. Cotton duck fabric was impregnated by coating on both sides with one coat to a weight of 1.5 ounces with the impregnating solution (30% solids) by a conventional double roll impregnator and subsequently dried in a festooning chamber for 45 minutes at 175° F.

One side of the fabric was then coated with 5 coats of the top-coating composition (Example 3 dissolved in gasoline to 40% solids) to a weight of 3 ounces per square yard and after drying in a convection tunnel was dusted with talc. The opposite side of the fabric was coated with 2 coats of the same composition to a weight of about 1.5 ounces per square yard and dried as before and then calender-coated with one coat of Example 3 (unthinned) to a total thickness of about .026 inch. The coating was dusted with talc and the coating on both sides cured in a festooning type oven for 2½ hours at 260° F.

The coated fabric was exposed to steam in accordance with the 4 cycle procedure of Example 1 and after such treatment when tested in a Scott tensile tester was found to have a strength of 165 pounds per square inch warp and 150 pounds per square inch fill indicating that in this instance also there was no appreciable loss in fabric strength and that the magnesium oxide functioned as a protective agent against the destructive effects of exposure to steam. A substitution of zinc oxide for the magnesium oxide in this composition as in the case of Examples 1 and 2 likewise failed to counteract the tendency of steam to tenderize the fabric.

Example 4

Cotton duck fabric was pretreated with magnesium oxide as in Example 4 and then coated with the following composition:

	Per cent by weight
Neoprene (chloroprene polymer)	53.0
Zinc oxide	5.4
Soft carbon black	26.6
Antioxidant (phenyl alpha - naphthyl - amine)	1.6
Petroleum process oil (low aniline point) ..	2.7
Litharge	10.7
	100.0

This composition was prepared by charging the materials into a heavy duty kneading machine such as a Banbury mixer and masticating until a homogeneous mass was secured. It was then cut with xylol to about 30% solids solution

and applied to the magnesium treated fabric by means of a conventional knife spreader machine to give a total coating thickness after evaporation of the volatile solvent of about 10 mils. The coating was drum cured by heating at 260° F. for 5 hours.

After exposure to open steam for 70 hours at 80 pounds pressure (324° F.) a sample when tested in the Scott tensile tester gave a warp strength of 163 pounds per square inch and fill strength of 148 pounds per square inch. A sample of fabric coated with the same composition but omitting the magnesium oxide pretreatment and similarly exposed gave a warp strength of but 36 pounds per square inch and a fill strength of 36 pounds per square inch. These results also show the pronounced improvement in coated cotton fabric after stringent steam exposures accomplished by means of protective effects of the magnesium oxide.

Example 5

	Per cent by weight
Neoprene (chloroprene polymer) -----	53.2
Magnesium stearate -----	5.3
Soft carbon black -----	26.6
Antioxidant (phenyl alpha - naphthyl-amine) -----	1.6
Petroleum process oil (low aniline point) --	2.7
Litharge -----	10.6
	100.0

The composition was prepared by charging the materials into a heavy duty kneading machine such as a Banbury mixer and masticating until a homogeneous mass was secured. It was then cut with xylol to about 30% solids solution and applied to cotton duck fabric by means of a conventional knife spreader machine to give a total coating thickness after evaporation of the volatile solvent of about 10 mils. The coating was drum cured by heating at 260° F. for 5 hours.

After exposure to open steam for 72 hours at 80 pounds pressure, a sample of the coated fabric when tested in the Scott tensile tester gave a warp strength of 125 pounds per square inch and fill strength of 147 pounds per square inch. A sample of fabric coated with a similar composition but substituting zinc stearate for the magnesium stearate and similarly exposed gave a warp strength of but 26 pounds per square inch and a fill strength of 44 pounds per square inch.

In addition to the magnesium and calcium oxides shown in the examples, it has been found that magnesium carbonate and magnesium stearate offer similar protective effects. In general, the oxides and salts of organic acids of magnesium and calcium including such compounds as carbonates, stearates, palmitates, oleates, and ricinoleates of these metals offer the unique advantage in the prevention of "tenderizing" in cotton fabrics exposed to open steam. Exhaustive tests indicate that it is the metal radical in the compound which affords the remarkable improvements of the present invention. The magnesium compounds are particularly outstanding in their performance and represent the preferred protective agents. The oxides of iron, barium and lead exhibit a small improvement in the same direction but do not approach the desirable results secured with the oxides of magnesium and calcium. The oxides of zinc, chromium, manganese, antimony, cad-

mium and titanium have little or no protective action for the present purposes.

The protective agents of the invention which may be used singly or in admixture may be included in the compositions in amounts from about 1.0% to 10% or even up to 25% for some purposes of the total solids of the compositions although for most purposes amounts of from about 1.0% to 6% generally suffice.

When the protective agents are employed with rubber and synthetic rubber compositions for coating fabrics which are exposed to steam, it is preferable to introduce the agent directly into the composition since it then serves a bifunctional purpose of protecting the fabric against deterioration from steam exposure and also as a curing agent for the rubber or synthetic rubber polymer. Other conventional rubber curing agents which do not exhibit protective effects may also be employed in conjunction with the protective agents of the present invention. For example, it has been found that when magnesium oxide or calcium oxide is present in about 2-5% of the total composition, up to equal amounts of zinc oxide curing agent may be introduced without appreciably reducing the protective effect of the previously described agent. The protective properties appear to function within the proportion ranges indicated regardless of the presence of any additional non-protective type curing agent for the rubber or synthetic elastomer.

The protective agent may also be applied separately and directly to the cotton fabric base either by dusting the finely divided agent onto the surfaces of the fabric or from a suspension in a liquid volatile vehicle or from a suspension in a liquid volatile vehicle containing a small amount of binder and the film-forming coating composition then applied directly over the protective agent treated fabric.

In connection with the application of the protective agent directly to the fabric, it has been found that a profound effect against deterioration may be secured without the addition of any organic top coat but in general for best all-round durability and prolongation of the life of the fabric, the use of a top-coat containing an organic polymeric material is preferred.

The protective agents may be used generally with organic polymers such as natural rubber, the synthetic rubbers, including neoprene (polymerized chloroprene), "Butyl rubber" (a copolymer of a conjugated diolefin and isobutylene), Buna N (acrylonitrile-butadiene copolymer), Buna S (butadiene-vinyl benzene copolymer), synthetic resins such as alkyd, vinyl, urea-formaldehyde, and melamine-formaldehyde resins, and in addition nylon (linear polyamides), cellulose esters, mixed esters and ethers are suitable binders. The improvement, however, is of particular importance when used with rubber and synthetic rubber coatings where the protective agents have a dual function including a curing effect on the rubber or synthetic rubber polymer.

The invention is of general utility in connection with cotton fabrics which are exposed to steam, a commercially important installation consisting of curing bags employed in the manufacture of molded plywood where previously available rubber or synthetic rubber coated cotton fabric employed for this purpose failed in a short period due to "tenderizing" effects of the fabric base resulting from deterioration of the cotton fiber on frequent exposures to steam. In commercial practice involving this utility it has been found

that the improved coated fabric will withstand up to 60 "cooks" as compared to previously available fabrics when used for this purpose which invariably failed at less than 20 "cooks." It is also useful in connection with steam hose wrappings.

The primary advantage of the present invention resides in the remarkable improvement in the treated cotton fabric in resisting the destructive effects of prolonged exposure to steam. The vastly superior properties of the new product may be obtained by applying the protective agent directly to the fabric or it may preferably be incorporated in an organic polymer containing coating composition where the deteriorating effects of exposure to steam are likewise counteracted. In coating compositions containing rubber or synthetic rubber polymers, the protective agent serves a dual function since in addition to protective effects, it likewise provides curing action on this type of polymer. The use of the protective agent in accordance with the invention does not detract from the pliability and general durability of the fabric. The useful life of the improved cotton fabric is greatly prolonged, thus contributing to the economics of processes where exposure to steam is required. The new process for treating cotton fabrics is simple and inexpensive, thus extending its commercial utility for practical operations.

It is apparent that many widely different embodiments of the invention may be made without departing from the spirit and scope thereof; and, therefore, it is not intended to be limited except as indicated in the appended claims.

I claim:

1. A coated cotton fabric which is capable of withstanding steam under pressure up to 70 pounds per square inch which comprises a woven cotton fabric base and a tough durable adherent zinc oxide free coating thereon comprising material in major amount selected from the group consisting of natural rubber, polymerized chloro-

prene, copolymers of isobutylene and a conjugated diolefin, copolymers of butadiene and vinyl benzene, and copolymers of butadiene and acrylonitrile; and from 1 to 25% based on the weight of the total solids of total combined (A) Vulcanizing agent and (B) curing and protective material said material being selected from the group consisting of calcium oxide and magnesium oxide, the said oxide being the sole curing agent the balance of the composition having substantially zero curing effect on the said rubber.

2. The product of claim 1 in which the protective agent is present in amount between 1% and 10% of the total solids of the coating.

3. A coated cotton duck which is highly resistant to the destructive effects of steam under pressure having a coating thereon consisting of the following composition by weight:

	Per cent
Polymerized chloroprene	50.0
Magnesium oxide	5.0
Wood rosin	5.0
Soft carbon black	26.0
Phenyl-alpha-naphthylamine	1.5
Castor oil	2.5
Litharge	10.0
	100.0

ALFRED J. JENNINGS.

REFERENCES CITED

The following references are of record in the file of this patent:

UNITED STATES PATENTS

Number	Name	Date
2,087,878	Wedge	July 20, 1937
2,208,619	Armor et al.	July 23, 1940
2,213,954	Crawford	Sept. 10, 1940
2,229,579	Manchester	Jan. 21, 1941
2,305,412	Frolich et al.	Dec. 15, 1942
2,323,387	Edelstein	July 3, 1943