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L. A. FLUCK ET AL
ACID RESISTANT CELLULOSIC MATERIAL AND
PROCESS FOR PRODUCING SAME

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2 Sheets-Sheet 1

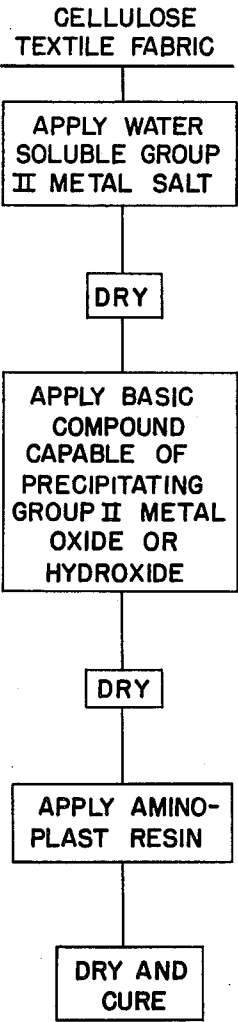


Fig-1

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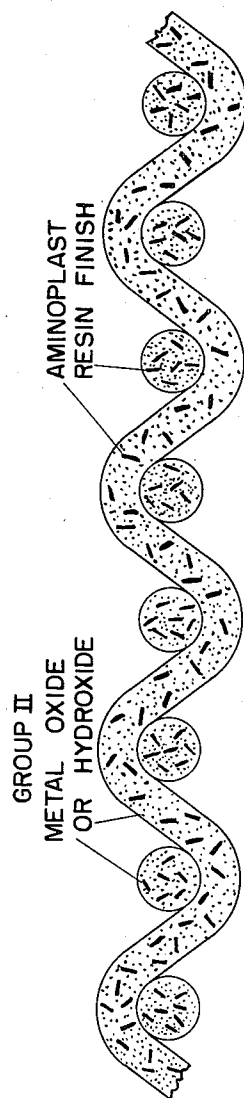


Fig-2

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ACID RESISTANT CELLULOSIC MATERIAL AND PROCESS FOR PRODUCING SAME

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The present invention relates to a novel process for rendering cellulosic materials acid resistant and to the acid resistant cellulosic materials produced thereby. More particularly, the present invention relates to a process whereby cellulosic material of the type subject to serious and repeated acid degradation is rendered resistant to such degradation. Still more particularly, the present invention relates to a process for producing acid-resistant decatizing blankets which are composed essentially of cellulosic material.

In the finishing of fibrous materials, such as certain rayon or woolen fabric, it is required to subject them to a steaming operation while the fabric is under tension in both warp and filling directions. This is customarily done in a so-called decatizing or decatizing machine. This machine consists principally of a perforated central hub or drum, about 60 to 65 inches wide, which is attached to a long endless belt or blanket of the order of from between 30 to 60 yards long, usually made of cellulosic materials.

Alternatively, the blanket may be in a single piece and one end is rolled up on a batch roll as the blanket is removed from the decatizing machine. When a fabric is to be inserted into the machine for steaming, the direction of the decatizer is reversed and the fabric to be treated is threaded between two layers of the decatizing blanket and rolled onto the machine. The tension on the layers of decatizing blanket may be controlled individually so that the fabric may be held in both warpwise and filling-wise directions, and when the piece to be steamed has been threaded onto the drum, steam is turned into the inside of the drum and passes through the perforated cylinder and through the goods, thus setting the fabric to give the desired finish. Thereafter, the treated fabric is dried. In short, it is a treatment similar to that produced by a Hoffman steam press customarily used in the pressing of garments, except of course, the decatizing machine can handle long lengths of fabric up to 60 or more yards in length and from 30 to 60 inches or more in width.

During the past few years, the textile finishing industry and particularly the woolen and worsted industry have attempted to manufacture fabrics that do not shrink after the fabric has been converted into suitings, etc. Another improvement has been the production of blankets for beds which will not felt or become scratchy during the usual laundering or dry cleaning processes. This requirement has been met quite successfully by various finishing treatments, one of which is the so-called chlorination process for wool fibrous materials.

One of the disadvantages of such a treatment is that some of the chlorine in the treating bath is retained by the wool fiber and during the decatizing operation the hot steam releases some of the chlorine as hydrochloric acid, which has a severely degrading effect on the decatizing blanket, necessitating frequent replacement at exorbitant expense. Even after this chlorinated fabric is made into suitings, the dry cleaner and presser has the same problem, although to a somewhat lesser extent.

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Therefore, it is an object of the present invention to provide a process to render cellulosic textile materials resistant to acid degradation.

It is a further object of the present invention to provide a process whereby cellulosic decatizing blankets or felt of the type employed on Hoffman presses and the like are rendered resistant to acid degradation and therefore have appreciably longer working life with a resulting reduction in operating cost for such machines.

It is still a further object to provide such cellulosic materials which, in addition to having a longer working life than those heretofore known, have the desirable soft hand required of such blanket and blanket type materials.

These and other objects and advantages will become apparent from the following description of the present invention set forth in detail hereinbelow.

This invention is further illustrated by the accompanying drawing, in which:

Figure 1 is a flow sheet illustrating a preferred process in accordance with this invention, and

Figure 2 is a cross section of a piece of textile material treated in accordance with this invention. As will appear, these figures are self-explanatory.

According to the present invention, a process is provided for treating textile fabrics containing cellulosic material to render the same resistant to acid degradation, which includes the application of an aminoplast resin thereto, which comprises applying to said fabric at least 10% based on the weight of the fabric of a water-soluble group II metal salt. Thereafter, the thus treated fabric is dried and the water-soluble salt applied thereto is precipitated with a basic compound capable of forming a water-insoluble group II metal compound selected from the group consisting of oxides and hydroxides. Thereafter, the so treated fabric is dried.

Suitable water-soluble group II metal salts within the meaning of the present invention are the water-soluble salt of magnesium, barium, calcium, strontium, zinc and cadmium and include the water-soluble halides and in particular, the chlorides, the sulfates, acetates, and the like. The water-soluble alkaline earth salts are greatly preferred.

These salts may be applied to the fabric as aqueous solutions employing conventional mill equipment as, for example, by padding, spraying, dipping, immersion and the like. Preferably, these materials are padded onto the textile fabric from a pad bath in amounts of between 10 and about 40% by weight and, preferably, in amounts of from between 15 and 30% by weight based on the weight of the textile fabric. A particularly preferred water-soluble alkaline earth salt is magnesium chloride.

The basic compound employed to precipitate the water-insoluble alkaline earth compound selected from the group consisting of oxides and hydroxides is preferably water-soluble and employed as an aqueous solution and in an amount sufficient to substantially neutralize the water-soluble alkaline earth salt. That is, it is preferred that a stoichiometrically equivalent amount of a basic compound be employed. If amounts slightly less than the stoichiometric equivalent are employed, no significant harm results and the effectiveness of the process is not greatly diminished. As a matter of fact, in certain instances where the water-soluble alkaline earth salt is a recognized aminoplast curing catalyst, it may sometimes be preferred to employ the basic compounds in amounts slightly less than the stoichiometric equivalent. What has been said with regard to amounts of the basic compounds, which are slightly less than the stoichiometric equivalent of the water-soluble alkaline earth salt applies for the most part to slight excesses of the basic compounds. Large excesses, however, are undesirable in that large amounts of the curing catalyst must necessarily be employed in order to

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effect the cure on the acid side of the aminoplast resin. In addition, these large excesses do not significantly improve the acid resistant characteristic of the treated fabric and result in deleterious dusting, which greatly limits the use of the treated fabric.

Examples of suitable basic compounds within the meaning of the present invention are alkaline compounds as, for example, ammonium hydroxide, potassium hydroxide, sodium hydroxide, calcium carbonate, and the like.

These materials may be employed either singly or in combination and are basic compounds of the type which, when reacted with water-soluble alkaline earth salts, produce water-insoluble alkaline earth compounds, selected from the group consisting of oxides and hydroxides and mixtures thereof.

After the textile fabric has been treated with basic compounds, it is then dried. Depending upon the extent of drying will depend the final composition of the water-insoluble alkaline earth compound. When drying is substantially complete, the resultant alkaline earth compound consists essentially of the alkaline earth oxide. If the extent of drying is reduced, the resulting alkaline earth compound usually contains amounts of hydroxide in addition to some oxide.

The aminoplast resin may be applied at three different stages of the present process and still produce acceptable results. These are (1) simultaneously with the water-soluble alkaline earth salt, (2) simultaneously with the basic, neutralizing or precipitating compound, or (3) independently of (1) and (2) and after the fabric has had (2) applied and dried.

Of these three, the last is greatly preferred in that it provides greater resistance to acid degradation than either of the others. Regardless of when the aminoplast resin is applied, it is applied in amounts of from between 1.5% and 30% resin solids based on the weight of the fabric and preferably amounts of from between 2% and 10% resin solids based on the weight of the fabric.

Preferably, the aminoplast resin is a polymethylol melamine resin. These resins may be prepared by reacting relative amounts of 1 mole of melamine with 1 to 6 or more moles of formaldehyde on the alkaline side to produce methylol melamines containing from 1 to 6 combined methylol groups. Thereafter, the methylol melamines may be further reacted with alcohols, as for example, saturated aliphatic alcohols containing from 1 to 4 carbon atoms in amounts of from between 1 to 6 and more moles of alcohol whereby alkylated methylol compounds having from 1 to 6 moles of combined alcohol may be produced.

Particular species of the methylated methylol melamine type compounds are the trimethylated trimethylol melamine, the trimethylated pentamethylol melamine, and the like. Methylol melamine compounds are greatly preferred in that they impart excellent hand, dimensional stability, and other desirable characteristics. In lieu of the methylol melamine type of resinous compound, guanamine-formaldehyde reaction products including certain substituted guanamine-formaldehyde reaction products may be employed. Urea-formaldehyde resins, methylated urea-formaldehyde resins, and ethylene urea, its homologues and their formaldehyde reaction products may be employed. These and other suitable aminoplast resins may be employed singly or in combination with one another.

If the fabric has not been treated with resin, but has been treated with an alkaline earth salt and is to be dried, or with the basic compound and is to be dried, temperatures of the order of about 180 to 250° F. should normally be employed. Temperatures in excess of 250° F., for the most part, should be avoided in that they are not required and they tend to adversely affect the tensile strength of the cellulosic material. Temperatures lower than 180° F. may be employed; however, longer periods of time are required without resulting benefit.

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When the aminoplast resin has been padded onto the cellulosic textile fabric, preferably the fabric is dried, as for example, at temperatures of the order of from between 200 and 275° F. until the fabric is substantially dry to the touch and thereafter the resin is cured, as by employing temperatures of the order of 280° F. to 450° F. for from 5 minutes to ¼ minute, respectively. If preferred, the treated fabric may be dried and cured in a single operation at temperatures of from 280° F. to 450° F. for from 6 minutes to ½ minute, respectively.

It is sometimes desirable to include a compatible wetting agent in the aqueous solution of the water-soluble alkaline earth salt or salt and aminoplast solution in order to achieve greater penetration of the fabric by the soluble salt. Wetting agents may be employed in amounts of from between 0.05 to 1.25% based on the weight of the solution and preferably in amounts from between 0.08 to 0.20%. Suitable wetting agents include Deceresol wetting agent NI Conc., the reaction product of nonyl phenol and ethylene oxide; Triton X-100, the reaction product of octyl phenol and ethylene oxide; Leonyl-O, the reaction product of oleyl alcohol and ethylene oxide; Renex-30, the reaction product of tridecanol and ethylene oxide. While these are examples of non-ionic wetting agents, any wetting agent compatible with the solutions referred to above are contemplated.

In order that the present invention may be more fully understood, the following examples are given primarily by way of illustration. No details therein should be construed as limitations on the present invention except as they appear in the appended claims. All parts and percentages are by weight unless otherwise indicated.

EXAMPLE 1

An 80" x 80" bleached unmercerized cotton cloth was padded through a solution of magnesium chloride containing 30% magnesium chloride and 0.1% Deceresol wetting agent NI, and then through squeeze rollers adjusted to provide a 120% wet pick-up. Thereafter, the cotton cloth so treated was dried at 100° C. The fabric was then padded through a 14% solution of ammonium hydroxide employing 2 passes and was again dried at 100° C.

The dried fabric was then padded through a pad bath composition, containing 5% of trimethylated trimethylol melamine, 3% of a reaction mixture of trimethylated methylol melamine and methylol stearamide, a non-ionic surface active agent, and 2.1% of anhydrous magnesium chloride. Thereafter, the treated textile fabric was dried and cured for 4 minutes at 350° F.

A piece of 80" x 80" cotton which was treated according to the above procedure, a similar piece which was so treated and then washed were tested for acid resistance. The wash referred to above was accomplished by passing the cloth through a 0.07% solution of synthetic detergent at room temperature, and then twice through water at room temperature. Thereafter the fabric was squeezed and dried at 225° F. for 2 minutes.

The test for acid resistance was as follows:

The respective pieces of 80" x 80" cotton were padded through a 5% hydrochloric acid solution, after which they were subjected to a squeeze roller adjusted to give an 80% wet pick-up. Thereafter, they were placed on a Hoffman press, steamed for 5 minutes, and then pressed dry. Strip tensile strength measurements were then made on the warp. The results were as follows:

Table I

Treatment	Dusting	Percent Loss by HCl Degradation
Untreated.....		93.6
Treated as described.....	slight.....	0
Treated and then process washed.....	slight.....	11.6

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It will be observed that the untreated piece lost 93.6% of its tensile strength by the above testing procedure, whereas the treated piece lost 0% by a similar test procedure. It is further noted that even after washing, the tensile strength loss was only 11.6%.

EXAMPLE 2

An 80" x 80" bleached unmercerized cotton cloth was padded through a solution containing 30% anhydrous magnesium chloride and 0.1% of Deceresol wetting agent NI and then through squeeze rollers adjusted to provide a 120% wet pick-up. Thereafter, the material so treated was dried. This fabric was then padded through a 5% solution of trimethylated trimethylol melamine having incorporated therein 10% ammonium hydroxide. The treated fabric was then dried and cured for 4 minutes at 350° F. When further processed as in Example 1 to determine the effect of acid degradation, it was found that the treated material lost 36.8% of the strength against 93.6% for the untreated piece.

EXAMPLE 3

An 80" x 80" piece of bleached unmercerized cotton was padded through a 5% solution of trimethylated trimethylol melamine containing, in addition, 30% of anhydrous magnesium chloride based on the total weight of the pad bath solution and then through squeeze rollers adjusted to provide a 120% wet pick-up. The treated piece was then dried and cured for 4 minutes at 350° F. This fabric was then padded twice through a 14% ammonium hydroxide bath, and thereafter dried. The piece was then tested for acid degradation by the procedure described above in Example 1 with the following results:

Table II

Treatment	Dusting	Percent Loss by HCl Degradation
Untreated.....	No.....	93.6
The piece which was treated with melamine resin, magnesium chloride, and neutralized with ammonia, but not process washed.....	Slight.....	19.7

It will be observed from the above table that the present process results in acceptable resistance to acid degradation but provides less acid resistance than is achieved by the process of Example 1.

EXAMPLE 4

A piece of 80" x 80" bleached unmercerized cotton was padded through an aqueous solution containing 15% anhydrous magnesium chloride, containing 0.1% Deceresol wetting agent NI and then through squeeze rollers adjusted to provide a 110% wet pick-up. Thereafter, the fabric was dried at 225° F. and the dried fabric was then padded through a 14% solution of ammonium hydroxide, two passes being run, and thereafter, dried again at 225° F. Thereafter, the dried fabric was padded through a pad bath composition containing 5% trimethylated methylol melamine, 3% of a composition consisting of the reaction of trimethylated methylol melamine and methylol stearamide, and a non-ionic surface active agent and 2.1% based on the weight of resin solids of anhydrous magnesium chloride. Then, the fabric so treated was dried and cured for 4 minutes at 350° F. A sample of the fabric was subjected to the acid degradation test set forth in Example 1, and compared with an untreated sample. The results of this comparison are set forth in the chart hereinbelow.

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Table III

	Dusting	Percent Loss by HCl Degradation
Untreated.....	No.....	93.6
Treated.....	No.....	6.2

Another advantage or feature of the present invention is that by employing the process steps as they are set forth in the above examples, subsequent dusting of the alkaline earth oxide or hydroxide is reduced to a minimum; that is, they are reduced to a point where commercially they are not a significant disadvantage.

A further advantage of the present invention is that the various process steps employed herein are readily adaptable to conventional mill equipment.

While the present process has been described as being particularly applicable to cellulosic textile materials, this should not be construed as necessarily meaning fabric composed wholly of cellulosic materials in that the present invention is applicable to blends of cellulosic fibers and other textile fiber materials such as certain synthetic fibers and the like. It is preferred, however, that the present process be employed in conjunction with material containing 50% and more of cellulosic fiber.

In addition to being particularly useful in the treatment of decatizing blankets and fabric employed to cover steam presses and the like, it is highly suited for processing filters, cloth material and the like, to render the same resistant to acid degradation.

What is claimed is:

1. A process for treating textile material containing at least 50% of cellulosic material to render the same resistant to acid degradation which includes the application of an aminoplast resin thereto, comprising applying to said fabric at least 10% based on the weight of the fabric of a water-soluble group II metal salt, drying said material, and precipitating said salt with a basic compound capable of forming water-insoluble group II metal compound selected from the groups consisting of oxides and hydroxides.

2. A process for treating textile material containing at least 50% of cellulosic material to render the same resistant to acid degradation which includes the application of a melamine-formaldehyde reaction product thereto, comprising applying to said fabric from an aqueous bath between 10% and about 40% based on the weight of the fabric of a water-soluble alkaline earth salt, drying said material and precipitating substantially all of said salt with an aqueous solution of a basic compound capable of forming a water-insoluble alkaline earth compound selected from the group consisting of oxides and hydroxides and thereafter drying said fabric.

3. A process according to claim 2 characterized by the alkaline earth salt being magnesium chloride and the basic compound being ammonium hydroxide.

4. A process according to claim 3 characterized by from 0.05 to 1.25% based on the weight of the bath of a non-ionic surface active agent being present in the aqueous bath containing the water-soluble alkaline earth salt.

5. A process for treating cellulosic textile fabric to render the same resistant to acid degradation comprising applying to said fabric at least 10% based on the weight of the fabric of a water-soluble alkaline earth salt, drying said treated material, precipitating said salt with a basic compound, capable of forming a water-insoluble alkaline earth compound selected from the groups consisting of oxides and hydroxides, drying said treated fabric, applying thereto a water-soluble aminoplast resin and thereafter drying said fabric and curing said resin to a water-insoluble state.

6. A process according to claim 5 characterized by

the application of from between 10 and about 40% based on the weight of the fabric of a water-insoluble alkaline earth salt and by the application of from between 1.5 and 30% based on the weight of the fabric of the water-soluble aminoplast resin.

7. A process according to claim 6 characterized by the water-soluble alkaline earth salt being magnesium chloride, the basic compound being ammonium hydroxide and the aminoplast resin being a melamine-formaldehyde reaction product.

8. A process for treating cellulosic textile fabric to render the same resistant to acid degradation which comprises applying to said fabric at least 10% based on the weight of the fabric of a water-soluble alkaline earth salt, drying said treated material and precipitating said salt by treating said fabric with an aqueous solution containing a water-soluble aminoplast resin and a basic compound capable of forming a water-insoluble alkaline earth compound selected from the group consisting of oxide and hydroxides and, thereafter, drying said treated fabric and curing said aminoplast resin to a water-insoluble state.

9. A process according to claim 8 characterized by the application of from between 10 and about 40% of a water-soluble alkaline earth salt from between 1.5 and 30% of a water-soluble melamine-formaldehyde reaction product and by the basic compound being ammonium hydroxide.

10. A process for treating cellulosic textile fabric to render the same resistant to acid degradation which comprises treating said material with an aqueous solution of an aminoplast resin containing at least 10% based on

the weight of the fabric of a water-soluble alkaline earth salt, drying said treated fabric and curing said water-soluble resin to a water-insoluble state, precipitating said salt with a basic compound capable of forming a water-insoluble alkaline earth compound selected from the group consisting of oxides and hydroxides and, thereafter, drying the said treated fabric.

11. A process according to claim 10 characterized by the aminoplast resin being a water-soluble melamine-formaldehyde reaction product, by the water-soluble alkaline earth salt being magnesium chloride and by the basic compound being ammonium hydroxide.

12. A process according to claim 11 characterized by the application of the melamine-formaldehyde reaction product being in an amount from between 1.5 and 30% by the magnesium chloride being applied in amounts between 10 and 40% both based on the weight of the fabric.

13. A textile fabric produced according to the process of claim 1.

14. A textile fabric produced according to the process of claim 8.

15. A textile fabric produced according to the process of claim 11.

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