

UNITED STATES PATENT OFFICE

2,689,194

FINISHING PROCESS AND FABRIC

William W. Russell, East Providence, and Herman C. Allen, Saylesville, R. I., assignors to Sayles Finishing Plants, Inc., Saylesville, R. I., a corporation of Rhode Island

No Drawing. Application February 18, 1952,
Serial No. 272,263

19 Claims. (Cl. 117-62)

1

This invention is concerned with a finishing process for textile fabrics, especially cotton fabrics, and with the fabrics so produced. More particularly the invention relates to a process for producing highly permanent, lustrous, glazed and embossed effects upon such fabrics.

Heretofore the art has sought to produce effects of the type here attained by impregnating the fabric with a thermosetting resin and then calendering or glazing the fabric at elevated temperatures, the resin normally becoming cured or set as an incident of the calendering or glazing. The finishes so produced have a number of defects. Thus, when the resin employed contains nitrogen, as is generally the case, e. g., urea, thiourea, melamine, etc., the finish is at least to some extent chlorine retentive. As a consequence, when the fabric is laundered and no anti-chlor rinse is used, the fabric retains chlorine taken from any bleaching reagents which may have been present, with the result that on later ironing, the fabric becomes yellowed and weakened. The yellowing may be noticeable, even when the fabric is dyed, unless the shade is quite dark. It is virtually impossible, using one of the nitrogen-containing resins, to produce a finished white fabric which will remain white. In any case, the permanency to washing and/or dry cleaning of the fabric may be unsatisfactory.

The present invention has as an object to provide a process giving satisfactory finishes without the use of resins or through the use of resins in reduced amounts, whereby the indicated adverse effects are either eliminated or substantially reduced. Another object is to provide a finishing treatment which does not result in any serious weakening or deterioration of the component yarns of the treated fabric. Still another object is to give added permanence to finishes imparted by calendering.

The process herein is distinguished, inter alia, in that before the fabric is mechanically treated as determined by the desired finish, there is deposited in and on the yarns thereof a readily deformable cellulosic material. Following the mechanical treatment, the fabric is impregnated with formaldehyde and/or a formaldehyde donor, i. e., a formaldehyde-yielding substance, and with a material adapted to catalyze the reaction of cellulose and formaldehyde, the impregnated fabric being later heated as necessary to effect the indicated reaction.

When there is used in the practice of the process a cellulosic substance from which cellulose is readily precipitated or regenerated, as is pre-

2

ferred, the fabric is subjected at some stage to a treatment adapted to bring about conversion of the substance or its residuum to cellulose.

It will be understood that the nature of the mechanical treatment varies depending upon the surface characteristics sought. In general, such calendering operations as friction and chase calendering are contemplated, along with embossing, schreiner, glazing, beetling, and the like, all of which are designated in this description and in the claims as "calendering."

It has been found that the deposited cellulosic material, which remains permanently or substantially permanently on the fabric, enables attainment of a better, more durable calendered or embossed finish with relatively little mechanical damage to the yarns of the treated fabric. In fact, by reason of the presence of the deposited cellulosic material, the yarns in some cases actually may have a greater tensile strength after the calendering treatment than before.

In carrying out the process as practiced according to one of two preferred schemes, the parent fabric is initially impregnated with an aqueous solution of cellulose xanthate and is thereafter subjected to the action of an acid, sulfuric acid, for example, to regenerate the cellulose from the xanthate. Following this operation, the fabric is washed and preferably while still moistened at least to a slight extent is subjected to the mechanical treatment, most usually a calendering operation. Thereafter the fabric, either in the wet or dry state, is impregnated with formaldehyde and a catalyst, ammonium thiocyanate for instance, and passed through a hot air curing chamber in which it is maintained at a temperature of the order of 350° F. for a few minutes to bring about reaction between the cellulose and the formaldehyde. Although the nature of such reaction is not definitely known, that there is a definite reaction is conclusively established by the fact that the affected cellulose is insoluble or at least has a reduced solubility in copper-ammonia solution.

Using the other of the two preferred schemes, the procedure is as above described except that not until after the mechanical treatment is the fabric treated in an acid bath to convert the deformed cellulose xanthate to regenerated cellulose. Alternatively, instead of decomposing residual cellulose xanthate prior to curing, the treatment in the acid bath may be delayed until after the curing operation.

Instead of solutions or dispersions of cellulose xanthate, other solutions or dispersions from

which cellulose can be regenerated or precipitated, so as to become a permanent or substantially permanent part of the fabric, may be employed in the practice of the invention—for example, cellulose in cuprammonium solution, or in certain quaternary solvents. In addition there are certain cellulose compounds, dispersible in and precipitable as such from aqueous, or aqueous-alkaline media, which can be similarly employed, e. g. cellulose ethers of a suitable degree of etherification, such as the ethyl or propyl ethers, and hydroxy ethers such as the ethyl hydroxy ether of cellulose. The terms “dispersion” and “dispersed” as used in the specification and claims are intended to include all of the foregoing, whether they be true solutions or colloidal dispersions. Those cellulosic materials generally applied in the arts in solution in organic solvents are unsuited for the purposes of the invention. As exemplary of this class of materials, nitrocellulose may be cited.

Formaldehyde donors which may be employed in place of formaldehyde itself in the practice of the invention include hexamethylenetetramine, certain formals, paraform and the like. Also there may be used resins synthesized in part from formaldehyde which, under the conditions of the heating and curing steps of the process, are capable of providing formaldehyde for reaction with the cellulose. These formaldehyde-resins are therefore formaldehyde donors and such resins are prepared, as is well known in the art, by reacting formaldehyde with suitable materials, for example, urea, thiourea, a ketone such as acetone, or melamine or numerous other materials. Particularly preferred for this purpose are water-soluble precondensates or low polymers of methylolurea, methylol melamine and the like. It is recognized that such resins are of the class noted in the forepart hereof as tending through chlorine retention to cause discoloring of the fabric from ironing. However, in the present case the resins can be employed in relatively small amounts so that such undesirable effects are mitigated. The principal advantage residing in the use of formaldehyde-resins as formaldehyde donors goes to the fact that the finish, in addition to being highly durable, is rendered more resistant to creasing. For this effect it is obviously necessary that the resin be used in sufficient quantity that all of it is not exhausted in providing formaldehyde for reaction with the cellulose. We may, of course, employ such resins in an impregnating bath additionally comprising in any desired ratio either formaldehyde added as such or a formaldehyde donor other than a formaldehyde-resin. Also resins of a relative high state of polymerization may be used in conjunction with the soluble low polymers.

Many substances are known to catalyze the reaction of formaldehyde and cellulose. These are in general acidic substances or substances which become acidic at the elevated temperatures at which the reaction takes place, e. g., tartaric acid, lactic acid, boric acid, various sulfonic acids, oxalic acid, acetic acid, formic acid, salts such as ammonium thiocyanate, ammonium acid phosphate, ammonium chloride and the like. Catalytic properties have also been attributed to essentially neutral salts, especially inorganic salts, and to alkaline substances such as caustic soda, alkali carbonates and the like. A number of the foregoing substances also catalyze the polymerization of the formaldehyde-resins just discussed.

Most of the mechanical treatments here contemplated are carried out at an elevated temperature. For example, in calendering cotton fabric containing cellulose xanthate or cellulose regenerated from cellulose xanthate the calender rolls are generally maintained at a temperature of the order of 350° to 425° F. The pressure exerted by the rolls will vary depending on the fabric and the finish desired. Usually, however, the total nip pressure is between 10 and 60 tons. Rolling, chasing, or embossing calenders as well as friction calenders are, of course, applicable to the invention, preferred practices of which are illustrated by the following examples, which are not to be taken as in any way limitative of the scope of the invention.

Example 1

A bleached, cotton fabric counting 80 x 80, weighing 4.0 yards per pound, and having a grey width of 37 inches was impregnated in a mangle with an aqueous cellulose xanthate composition containing 3.0% cellulose (present as the cellulose in the xanthate), 2.5% sodium hydroxide and 2.0% of a substantive, non-ionic softener. In addition, the bath contained products of the xanthation reaction.

The pick-up from the bath was about 50%.

The impregnated fabric was run without drying into a bath of 10% sulfuric acid in which the xanthate was decomposed leaving regenerated cellulose in and on the fabric. The acid-treated fabric was thoroughly washed first with hot and then with cold water, squeezed off and partially dried by passing through a heated tenter frame at near grey width. The fabric as it emerged from the frame carried about 10 to 15% moisture and was immediately passed three times through a friction calender heated to 400° F. and exerting a total nip pressure of 55 tons. Following this operation, the calendered fabric was impregnated in a mangle with a 5% aqueous solution of formaldehyde containing 0.3% ammonium thiocyanate. The pick-up was about 50%. The formaldehyde-impregnated material was substantially dried by passing it through a tenter frame heated to about 260° F. Thereafter it was passed through a hot air curing chamber in which the fabric was maintained at 350° F. for two minutes. The cured fabric was subjected to the action of an aqueous scouring bath (containing 0.5% sulfonated higher fatty alcohol detergent, and 0.2% ammonia) and then thoroughly washed in hot and cold water, respectively. The framed finished fabric was characterized by a rather high gloss or glaze, yet it had a pleasingly soft and good tensile strength. Upon repeated washing the fabric retained its glossy finish and pleasing hand substantially unimpaired.

Example 2

A bleached cotton fabric counting 80 x 92, weighing 3.50 yards per pound, and having a grey width of 40 inches was impregnated in a mangle with an aqueous cellulose xanthate composition, made by dissolving cellulose xanthate (prepared from well aged alkali cellulose) in water, and then neutralizing with dilute acid to a pH of about 7 under conditions avoiding any permanent coagulation or precipitation of the cellulose xanthate. The composition had the following formulation, ignoring products of the xanthation

reaction and reaction products from the neutralization:

	Per cent by weight
Cellulose (present in the xanthate) -----	2.0
Substantive, non-ionic softener -----	2.0

The pick-up by the fabric from the bath was about 50%.

The impregnated fabric was conveyed through a heated tenter frame at near grey width. The fabric as it emerged from the frame carried about 10 to 15% moisture and was immediately passed through an embossing calender heated to about 400° F.

Following the calendering, the fabric was run into a bath of 10% sulfuric acid in which any remaining xanthate was decomposed, leaving mechanically deformed regenerated cellulose in and on the fabric. The acid-treated fabric was thoroughly washed first with hot and then with cold water, squeezed, and dried by passage through a heated tenter frame at near grey width. After this operation, the fabric was treated with a 5% aqueous solution of formaldehyde containing 0.3% ammonium thiocyanate. The pick-up from the formaldehyde bath was about 50%. The formaldehyde-impregnated material was substantially dried by passing it through a tenter frame heated to about 260° F. Thereafter it was passed through a hot air curing chamber in which the fabric was maintained at about 350° F. for two minutes. The cured fabric was scoured and washed and finished as in Example 1. The finished fabric had a well defined, three dimensional embossed pattern which was fast to washing.

Example 3

The treatment was the same as in Example 2 through the embossing operation. However, the embossed fabric, without any intervening operation was treated with a 5% aqueous solution of formaldehyde containing 0.3% ammonium thiocyanate and enough dihydrogen ammonium phosphate to neutralize any residual alkali in the cellulose xanthate. The pick-up from the modified formaldehyde bath was about 100%. The formaldehyde-impregnated material was substantially dried by passing it through a tenter frame heated to about 250° F. Thereafter, it was conveyed through a hot air curing chamber (340° F. for two minutes). The cured fabric was run into a bath of 10% sulfuric acid in which any remaining xanthate was decomposed leaving deformed, formalized regenerated cellulose in and on the fabric. Subsequently, the cured fabric was washed and then scoured, washed again, and finished as in Example 1. The finished fabric had properties in general similar to the fabric of Example 2.

Example 4

The treatment was the same as in Example 2, except that the fabric was impregnated using the cellulose xanthate bath of Example 1, instead of the cellulose xanthate bath of Example 2, and excepting, further, the fact that the xanthate-impregnated fabric was passed thrice through a friction calender instead of once through an embossing calender as in Example 2. The finished fabric had properties in general similar to the fabric of Example 1.

Example 5

In a modification of the process as illustrated by Example 1, the treatment may conform with such example through the calendering operation.

Thereafter, in lieu of impregnating the fabric as described in such example, the fabric may be impregnated by running the same through a bath containing 10% melamine-formaldehyde resin (water-soluble low polymer) and 0.3% isopropyl-amino-hydrochloride. On subsequent passage of the impregnated fabric through the hot air curing chamber (350° F. for two minutes), some formaldehyde is freed from the resin for reaction with the cellulose while the undecomposed portion polymerizes to confer crease resistance to the fabric.

Calendering or embossing the xanthate-impregnated fabric with the cellulose xanthate substantially undecomposed, or only partially decomposed gives added permanence to the calendered and embossed effects obtained. While it is desirable to substantially neutralize the cellulose xanthate, partially neutralized xanthate, or even unneutralized xanthate is operable. To neutralize the cellulose xanthate reaction mixture one may use for example dilute mineral acids, organic acids, or other acidic substances.

As is well known in the art, cellulose xanthate is readily prepared by reacting carbon bisulfide with suitably aged alkali cellulose and dispersing the reaction product in water, if necessary, with the aid of a small amount of caustic soda. The amount of regenerated cellulose, or cellulosic material added to the fabric may vary widely with the fabrics treated and the effects desired, but will usually be within the limits of a few tenths to several per cent—5 to 10 per cent or higher. After impregnation with cellulose xanthate, the fabric is preferably not dried if the regeneration or precipitation of the cellulose is to follow immediately. In any case the fabric is preferably not completely dried prior to calendering. The fabric, after the calendering step, may be dried at a high temperature, for example 350° F., but it is usually adequate to dry at normal temperatures, for example 250° F. prior to the formaldehyde impregnation. In the impregnation steps the liquid pick-up may vary widely but usually will be between the limits of somewhat less than 50 per cent up to 200 per cent or higher. The time and temperature required to bring about reaction of the formaldehyde and the cellulose and/or the cellulosic material chiefly depend on the strength of the catalyst, and the amount of catalyst employed. Usually, the concentration of the formaldehyde will lie within the range of from one to 15%, depending on the structure and composition of the fabric, the finish desired, and other considerations.

While any suitable softener can be employed at one or more stages of the process, it is preferred to use a substantive softener such as a non-ionic softener—for example, an alkaline dispersion of a substituted fatty amide, or an anionic softener especially for admixture with cellulose xanthate solution.

It is to be understood that the present invention may be applied not only to cotton fabrics, but also to fabrics of linen, cellulosic rayons, especially spun rayons, and to their mixtures with cotton.

Having thus described and illustrated our invention, what we claim is:

1. Method of finishing cellulosic textile fabrics comprising depositing in and on the yarns of the fabric from an aqueous medium a mechanically deformable cellulosic material dispersed in said medium, said cellulosic material being react-

able with formaldehyde, thereafter calendering the fabric to provide the desired finish while the fabric is free from acidic catalytic materials, thereafter impregnating the calendered fabric with at least one substance of the class consisting of formaldehyde and formaldehyde donors and with a catalyst for the reaction of formaldehyde and the cellulosic material, and finally heating the fabric to produce such reaction.

2. Method of finishing cellulosic textile fabrics comprising impregnating the fabric with cellulose xanthate from an aqueous medium, thereafter treating the impregnated fabric with a cellulose-regenerating agent, thereafter calendering the so-treated fabric to provide the desired finish while the fabric is free from acidic catalytic materials, thereafter impregnating the calendered fabric with formaldehyde and an acidic catalyst for the reaction of formaldehyde and cellulose and finally heating the fabric to a temperature and for a period of time effective to bring about such reaction.

3. Method of finishing cellulosic textile fabrics comprising impregnating the fabric with cellulose xanthate from an aqueous medium, thereafter calendering the so-treated fabric to provide the desired finish while the fabric is free from acidic catalytic materials, thereafter treating the calendered fabric with a cellulose-regenerating agent, thereafter impregnating the fabric with formaldehyde and an acidic catalyst for the reaction of formaldehyde and cellulose and finally heating the fabric to a temperature and for a period of time effective to bring about such reaction.

4. Method of finishing cellulosic textile fabrics comprising impregnating the fabric with cellulose xanthate from an aqueous medium, thereafter calendering the so-treated fabric to provide the desired finish while the fabric is free from acidic catalytic materials, thereafter impregnating the calendered fabric with formaldehyde and an acidic catalyst for the reaction of formaldehyde and cellulose, thereafter heating the fabric to a temperature and for a period of time effective to bring about the indicated reaction, and subsequently subjecting the fabric to treatment with a cellulose-regenerating agent.

5. Method of finishing cotton fabrics comprising impregnating the fabric with cellulose xanthate from an aqueous medium, thereafter treating the impregnated fabric with acid to regenerate cellulose from the xanthate, thereafter calendering the so-treated fabric to provide the desired finish while the fabric is free from acidic catalytic materials, the fabric as introduced to the calendering being in a moistened condition, thereafter impregnating the calendered fabric with formaldehyde and an acidic catalyst for the reaction of the formaldehyde and cellulose and finally heating the fabric to a temperature and for a period of time effective to bring about such reaction.

6. Method according to claim 5 where the calendering consists of a hot calendering operation.

7. Method of finishing cellulosic textile fabrics comprising impregnating the fabric with substantially neutral aqueous cellulose xanthate solution, thereafter calendering the so-treated fabric to provide the desired finish while the fabric is free from acidic catalytic materials, the fabric as introduced to the calendering being in a moistened condition, thereafter treating the calendered fabric with a cellulose-regenerating

agent, thereafter impregnating the fabric with formaldehyde and an acidic catalyst for the reaction of formaldehyde and cellulose and finally heating the fabric to a temperature and for a period of time effective to bring about such reaction.

8. Method according to claim 7 where the calendering consists of a hot calendering operation.

9. Method of finishing cotton textile fabrics comprising impregnating the fabric with a substantially neutral aqueous cellulose xanthate solution, thereafter calendering the so-treated fabric to provide the desired finish while the fabric is free from acidic catalytic materials, the fabric as introduced to the calendering being in a moistened condition, thereafter impregnating the calendered fabric with formaldehyde and an acidic catalyst for the reaction of formaldehyde and cellulose, thereafter heating the fabric to a temperature and for a period of time effective to bring about the indicated reaction, and subsequently subjecting the fabric to treatment with a cellulose-regenerating agent.

10. Method according to claim 9 where the calendering consists of a hot calendering operation.

11. Method of finishing a cellulosic textile fabric comprising depositing in and on the yarns of the fabric from an aqueous medium a mechanically deformable cellulosic material dispersed in said medium, said cellulosic material being reactable with formaldehyde, thereafter calendering the fabric to provide the desired finish while the fabric is free from acidic catalytic materials, thereafter impregnating the calendered fabric with a formaldehyde-resin and an acidic catalyst, and finally subjecting the impregnated fabric to heat whereby a portion of the resin becomes more completely polymerized to impart crease resistance to the fabric, the remaining portion providing formaldehyde for reaction with the cellulosic material.

12. Method according to claim 11 where the cellulosic material deposited on the fabric is cellulose xanthate and the fabric is treated at some stage to convert the cellulose xanthate to regenerated cellulose.

13. Method of finishing cellulosic textile fabrics comprising impregnating the fabric with cellulose xanthate from an aqueous medium, thereafter treating the impregnated fabric with a cellulose-regenerating agent, thereafter calendering the so-treated fabric to provide the desired finish while the fabric is free from acidic catalytic materials, thereafter impregnating the calendered fabric with a formaldehyde-resin and an acidic catalyst, and finally heating the fabric to a temperature and for a period of time effective to bring about polymerization of the resin and reaction of formaldehyde with the cellulose.

14. Method of finishing cotton fabrics comprising impregnating the fabric with cellulose xanthate from an aqueous medium, thereafter treating the impregnated fabric with acid to regenerate cellulose from the xanthate, thereafter calendering the so-treated fabric to provide the desired finish while the fabric is free from acidic catalytic materials, the fabric as introduced to the calendering being in a moistened condition, thereafter impregnating the calendered fabric with a formaldehyde-resin and an acidic catalyst, and finally heating the fabric to a temperature and for a period of time effective to bring about

polymerization of the resin and reaction of formaldehyde with the cellulose.

15. Method of finishing cellulosic textile fabrics comprising impregnating the fabric with substantially neutral aqueous cellulose xanthate solution, thereafter calendering the so-treated fabric to provide the desired finish while the fabric is free from acidic catalytic materials, the fabric as introduced to the calendering being in a moistened condition, thereafter treating the calendered fabric with a cellulose-regenerating agent, thereafter impregnating the fabric with a formaldehyde-resin and an acidic catalyst, and thereafter finally heating the fabric to a temperature and for a period of time effective to bring about polymerization of the resin and reaction of formaldehyde with the cellulose.

16. A textile fabric prepared by the method of claim 1.

17. A textile fabric prepared by the method of claim 2.

18. A textile fabric prepared by the method of claim 11.

19. A textile fabric prepared by the method of claim 13.

References Cited in the file of this patent

UNITED STATES PATENTS

Number	Name	Date
1,036,282	Lilienfeld	Aug. 20, 1912
2,104,748	Huex et al.	Jan. 11, 1938
2,119,150	Bowen et al.	May 31, 1938
2,314,277	Hurd	Mar. 16, 1943
2,378,186	Clark	June 12, 1945
2,411,818	Weiss	Nov. 26, 1946
2,504,857	MacIntyre	Apr. 18, 1950
2,603,576	Cook et al.	July 15, 1952

FOREIGN PATENTS

Number	Country	Date
634,634	Great Britain	Mar. 22, 1950