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PROCESS FOR GLAZING FABRIC

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This invention relates to the production of a smooth, lustrous finish on fabric, which is retained on washing or dry cleaning.

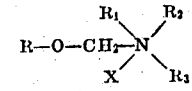
The conventional glazing of cloth is performed with starches and gums which are readily removed by washing. The use of certain heat-hardening resins has been proposed for this purpose, but they frequently cause difficulties from embrittlement, loss of strength of fabric, development of odor on ageing, lack of stability of resin during processing, depolymerization on overheating, and lack of uniformity due to variations in conditions of drying. It is difficult with few heat-hardening resins which have been found even partially suitable for mechanical glazing to obtain a full degree of luster and smoothness and to operate the process with the flexibility essential for adapting it to the equipment of different mills and for producing the variations in lustrous finishes desired for different types of textile materials.

It is the object of this invention to provide a reliable and convenient method for producing on fabrics a glaze which is resistant to spotting and to loss of luster when cleaned. It is also an object to provide a process of glazing fabrics, which can be practised with commonly available equipment, and which possesses the flexibility required for variations in finish on different sorts of fabrics. It is a further object to impart to fabrics a lustrous finish which is free from the disadvantages of previously available glazed fabrics.

I have found that a highly desirable glazed finish may be imparted to cloth or fabric by applying a water-soluble or alkali-soluble cellulose ether and a reactive, decomposable, quaternary ammonium salt, drying the treated fabric, calendering the treated cloth or fabric so as to glaze the surface, and at an appropriate stage of the process reacting the cellulose ether and quaternary ammonium compound by heating to yield an insoluble coating. The heating may be accomplished during the calendering, following the calendering or at both times.

The cellulose ethers which may be used include water-soluble forms of ethyl cellulose, methyl cellulose, hydroxyethyl cellulose and the like, and also akali-soluble forms of cellulose ethers, such as the alkyl, hydroxy-alkyl, or carboxy-alkyl ethers of cellulose, these latter cellulose ethers being of a low degree of alkylation.

In order to obtain a permanently glazed finish with a cellulose ether, it is necessary to use a suitable agent to fix or set the finishing material. With cellulose ethers this is very conveniently done with a quaternary ammonium compound which is sufficiently reactive with the cellulose ether to insolubilize the finish in practical mill operations and which does not tender the fabric or require excessive times or temperatures for processing. The quaternary ammonium salts, which are effective, have the general formula

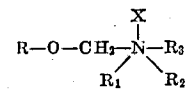


wherein R is an aliphatic hydrocarbon group, X is a salt-forming anion (such as chloride, bromide, ethyl sulfate, acetate, etc.), and R_1, R_2, and R_3 are selected to satisfy the remaining valences of the nitrogen atom from the class consisting of trivalent unsaturated hydrocarbon radicals which jointly with the nitrogen form a heterocycle, saturated divalent hydrocarbon and divalent ether radicals which jointly with the nitrogen form a heterocycle, and the monovalent radicals, aliphatic-oxy methyl, aralkyl, and aliphatic and alicyclic hydrocarbon. Examples of the divalent groups are those found attached to nitrogen in piperidine and morpholine. The trivalent group may be illustrated by the hydrocarbon chain of pyridine.

Typical compounds which are useful in reacting with the water-soluble cellulose ethers include:

Oleyloxymethyl pyridinium sulfite
Cetyloxymethyl pyridinium sulfite
Dodecyloxymethyl pyridinium acetate
Dodecyloxymethyl-methyl piperidinium chloride
Ethyloxymethyl diethyl benzyl ammonium chloride
Isobutyloxymethyl dimethyl β-methallyl ammonium chloride
Isooctyloxymethyl dimethyl ammonium bromide
Octyloxymethyl diethyl allyl ammonium chloride
Dodecyloxymethyl dimethyl benzyl ammonium chloride
Cetyloxymethyl dimethyl benzyl ammonium chloride
Octyloxymethyl trimethyl ammonium chloride
Dodecyloxymethyl butyloxymethyl dimethyl ammonium chloride
Octyloxymethyl dimethyl benzyl ammonium chloride

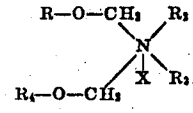
One highly desirable class of quaternary ammonium compounds for the purposes of this invention is represented by the general formula



wherein R represents an aliphatic hydrocarbon group, R_1 and R_2 represent lower alkyl groups, X represents a salt-forming anion, and R_3 represents a member of the group consisting of benzyl, allyl, methallyl, and crotyl radicals. These compounds react more rapidly and at lower temperatures than other quaternary compounds containing one oxymethyl group. They are more

stable in solution than pyridinium compounds and more effective, although the latter are reactive when heated to sufficiently high temperatures.

Particularly effective are the quaternary ammonium compounds containing two aliphatic oxymethyl groups, such as dodecyloxymethyl octyloxymethyl dimethyl ammonium chloride, cetyloxymethyl butyloxymethyl morpholinium chloride, and analogous compounds, the preparation of which is described in co-pending application Serial No. 244,197, filed December 6, 1938, by L. H. Bock which on June 18, 1940 issued as U. S. Patent No. 2,204,653. These compounds are effective at lower temperatures than those containing a single alkyl oxymethyl group and are, therefore, even more safetly and easily used. These compounds are of the general formula



wherein R is an aliphatic hydrocarbon group, R_4 an alkyl group, R_2 and R_3 are lower alkyl groups when taken individually or when taken jointly with the nitrogen are divalent hydrocarbon radicals or divalent ether radicals.

When the quaternary ammonium compound contains short chains, the glazed finish tends to be somewhat crisp and stiff, as is sometimes desired. Longer chained substituents give softer finishes. With hyrocarbon chains of fourteen or more carbon atoms attached to the oxymethyl group water-repellent effects may be obtained. For the average glazed fabric a balance of economy and of satisfactory glaze is obtained when an aliphatic oxymethyl group containing six to ten carbon atoms is used.

While the production of lustrous finishes requires the essential steps, outlined above, of applying to fabric a cellulose ether and a suitable quaternary ammonium compound, drying and calendering the treated fabric, and reacting the ether and fixing agent, variations of this general procedure permit the process to be adapted to a particular fabric, to the particular type of lustrous finish desired, and to the equipment and production methods of different textile mills. It is usually desirable to apply the cellulose ether and fixing agent in a single bath. This is not essential, however, as the ether may be applied in one step and the quaternary ammonium compound in a subsequent step. This latter procedure is necessary when alkali-soluble cellulose is used, in which case the fabric is impregnated with a heavy solution or paste of the ether, dried, passed through a dilute acid bath (one containing less than 5% sulfuric acid is satisfactory), thoroughly rinsed, freed from excess water by squeezing or partial drying, and then impregnated with a bath containing the desired quaternary ammonium derivative.

Since the quaternary compounds are usually acid as manufactured and since acid may be formed in setting the cellulose ether, the solutions or pastes for impregnating fabric should be adjusted to a pH close to 7 in order to avoid tendering fabric. Best results are obtained at a pH of 6.6 to 6.8. The presence of salts of weak acids or weak bases provides buffer action and also gives catalytic action for combining ether and ammonium compound. The optimum setting of the cellulose ether has been observed when the finished fabric by extraction in distilled water gave a pH of 4 to 5. For adjusting the solution and supplying catalytic salts there may be used acetic acid, sodium acetate, sodium bicarbonate, borax, ammonium chloride, ammonium acetate, trimethyl benzyl ammonium ethyl sulfate, dimethyl dibenzyl ammonium chloride, etc.

The finishing preparation should contain from 1 to 8% of a cellulose ether, the particular amount required depending on the nature of fabric and of ether, and from 3 to 20% of quaternary ammonium compound.

Application of the solutions or pastes to the fabric may be performed by padding on a quetch, by "slop padding" (that is, with padder rolls, the lower of which dips into the solution), by backfilling, or by other means of coating, including printing. The cellulose ether is dispersed or dissolved in water to yield a viscous solution or a paste, to which the quaternary compound may be added if desired. The treated fabric is then dried or partially dried by any suitable means, such as a loop drier, can drier, or tenter, taking care that the temperatures of the drying apparatus are low enough to leave the finish in a plastic state (150° F.–240° F.) Whether the fabric is completely dried or partly dried, it is usually desirable to frame it to width during or following this drying step. If the drying is controlled so as to leave the fabric slightly damp, the fabric may be run directly to the calender. But since it is not easy to control the residual moisture, it is better to dry the goods completely and then dampen them before the calendering operation by any of the well known methods, such as spraying, steaming, etc.

Calendering is done with heavy-set rolls and/or with friction rolls. The calendering operations may be repeated if desired. The degree of gloss may be controlled by the temperature, extent, and type of calendering, and also by the moisture content of the fabric. With temperatures of 260–400° F. for the calendar banks, the reaction of cellulose ether and quaternary ammonium compound is started in this operation and for some fabrics a satisfactory degree of permanency of finish is produced at this point. For the best fixation and in cases where the temperature of the calender rolls is not sufficiently high or the time of calendering is short, a final cure in a loop drier, closed tenter or equivalent equipment at 290° F. or higher is advisable. Final curing may also be accomplished with radiant heat as from electric lamps and reflectors. Treatment for three to ten minutes or more at this point at such temperatures ensures excellent retention of the lustrous finish on cleaning. As a final step, when desired, the glazed fabric may be washed to remove salts and decomposition products and then dried on cans or in a tenter. Calendering at this point is optional.

Typical procedures are given in the following examples to illustrate the application of a cellulose ether and quaternary ammonium compound in lustrous finishes.

Example 1

A bleached 68 x 72 cotton percale was padded on a lightly set quetch with solution containing 3% water-soluble ethyl cellulose and 8% octyloxymethyl dimethyl benzyl ammonium chloride. A small amount, approximately one-tenth per cent., of emulsified paraffin wax was added to the treating bath to give better action on the calender. The treated fabric was dried at finished width on a tenter running at about 220° F., then

passed through a hydraulic calender operating at 290° F. and 30 tons pressure on the bearings, and through a steam-heated friction calender operating at a ratio of 2.5:1 and at a temperature of 280° F. The fabric was finally heat-treated for five minutes in a loop drier at 300° F.

The cloth was glossy and smooth on the finished face with a suitable hand for a glazed fabric.

Example 2

An 80 x 80, mercerized, red naphthol dyed cotton fabric was impregnated on a Tommy Dodd backfilling machine with a solution containing four per cent. water-soluble ethyl cellulose and five per cent. cetyloxymethyl dimethyl benzyl ammonium chloride. The fabric was partially dried, framed to width, and passed through a calender at 300° F. and 30 tons pressure. A further heat treatment was given in an oven at 300° F. for five minutes.

The finished cloth was smooth, lustrous, full and well filled. On washing at 160° F. with soap and soda the gloss, fullness and filling were retained.

Example 3

An 80 x 80 unmercerized cotton print, bearing naphthol and indigosol colors, was slop-padded with a solution containing 6% hydroxyethyl cellulose and 8% capryloxymethyl dimethyl benzyl ammonium chloride. The fabric was frame-dried, dampened, and glazed with a gas-heated friction calender operating at 300-350° F. and cured at 300° F. for five minutes in a closed tenter.

The face of the cloth was lustrous and smooth, the colors brilliant, and the patten unimpaired.

Example 4

An 80 x 80, unmercerized percale was padded on a quetch with an alkali-soluble ethyl cellulose and dried to a canary yellow by passing through a hot calender and under carefully controlled conditions passed through a bath of three per cent. sulfuric acid, well washed and squeezed as dry as possible. The fabric was then padded through a solution containing approximately 20% butyloxymethyl dimethyl benzyl ammonium chloride, buffered with a small amount of ammonium chloride and sodium acetate to a pH between 6.5 and 7. The fabric was then loop-dried to approximately 12% moisture content, framed to width and hydraulic calendered at 290° F. Final curing was accomplished in a loop drier at 300° F.

A smooth, moderately glossy, somewhat paper-like finish resulted.

Example 5

A red naphthol dyed, 64 x 60, mercerized nainsook was padded on a quetch with a light set through a solution containing 4% of ethyl cellulose and 5% of octyloxymethyl dimethyl benzyl ammonium chloride. The cloth was frame-dried, lightly sprayed with water and calendered, first with a hot hydraulic calender with a heavy set and then with a friction calender operating at over 300° F. Then, the cloth was padded on a quetch with a heavy set through a 25% solution of octyloxymethyl dimethyl benzyl ammonium chloride solution, buffered with sodium bicarbonate at a pH of 6.8. The cloth was again dried, calendered as before, and given a final cure at 300° F. for ten minutes. The product was soft but firm and glossy. The luster was not destroyed by cleaning with hot soap and water.

A similar result was obtained with the same type of fabric by padding it through a solution containing 3% ethyl cellulose and 9% octyloxymethyl dimethyl benzyl ammonium chloride and omitting the second padding with the solution of the quaternary ammonium compound and the second drying and calendering steps. The resulting fabric was lustrous and smooth, but not as soft as the first piece.

Example 6

A 48 x 48, unmercerized, cotton print cloth, dyed with Naphthol AS and Fast Scarlet R salt, was padded under the liquor with a solution containing 2% water-soluble ethyl cellulose and 12.5% cetyloxymethyl dimethyl benzyl ammonium chloride, passed over six cans for partial drying and passed through a tenter-frame for completion of the drying. The fabric was lightly dampened with a fine water spray and glazed with a friction calender at 400° F. Final curing was performed for three minutes in a traveling apron drier at 310° F. The fabric was then open-soaped at 160° F. with a solution containing 0.5% soap and 0.25% soda ash and rinsed with water. It was then can-dried and calendered to relieve traces of cracking of the face.

Example 7

An 80 x 80, unmercerized, vat printed percale was padded in a solution containing 2% of 40% formaldehyde solution, 0.1% ammonium chloride, 1.8% ethyl cellulose, 17.5% octyloxymethyl dimethyl benzyl ammonium chloride and small amount of a high-melting wax (carnauba). This fabric was then frame-dried to width, dampened with steam, and glazed with a friction calender operating at 400° F. The fabric was then cured for five minutes in a net drier at 300° F.

The finish possessed high luster and was soft and pliable, with retention of luster after washing.

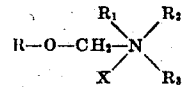
The process of glazing herein disclosed may be applied to any material which will withstand impregnation in aqueous solutions and heating to approximately 300° F. for a short time. Typical materials which may be so treated include cotton, linen, and viscose rayon in the form of chintz, decorative fabrics, such as upholstery fabrics and window drapes, table linen, ribbons, braids, such as shoe-strings, yarns, etc. If designs are printed and cured on a fabric, unprinted areas may be rendered matte by washing, damask effects thus being easily produced. The process imparts gloss, luster, smoothness, filling, fullness, stiffness, and other desirable properties. By choice of materials and by minor modifications in the method of application, as herein described, a great variety of permanent, lustrous finishes becomes possible. Thus, the fabric may be made soft and pliable or firm and somewhat stiff, even and smooth or thready and with any degree of luster from high to low. Also local effects may be obtained.

The process has many advantages. Up to the present time successful glazing has required fine yarn in at least an 80 x 80 construction. The new process has been successfully applied to lower count fabrics and to fabrics with coarse yarns and to yarns themselves. The process is not limited to cotton fabrics. If the glaze as first obtained is not up to requirements in water-resistance and retention of luster on washing, the fab-

ric may be again padded through a solution of a suitable quaternary ammonium compound and heated again. The fabric finished by this process are stabilized against shrinkage, coming well within the 1% limit prescribed for non-shrinking fabrics. Tear strength is higher than for other finishes and yet the fabric may be torn straight from selvage to selvage. Breaking strength is higher than for untreated fabric, increases in both warp and filling as great as 50% having been measured. Strips do not fray easily and, because of this property, fabrics may be cut on the bias and used for bias binding. But the greatest practical advantage of the new process is its flexibility and adaptability in meeting demands for variations in lustrous or glazed finishes with commonly available equipment.

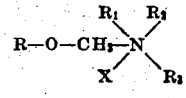
I claim:

1. The process of imparting to the surface of fabrics a wash-resistant, glazed finish which comprises applying to a fabric a cellulose derivative selected from a member of the class consisting of water-soluble cellulose ethers and alkali-soluble cellulose ethers, treating said cellulose derivative in the presence of the fabric with a quaternary ammonium salt having the formula—



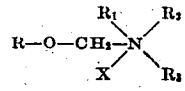
wherein R represents an aliphatic hydrocarbon group, X represents a salt-forming anion, and R_1, R_2, and R_3 are selected to satisfy the remaining valences of the nitrogen atom from the class consisting of trivalent unsaturated hydrocarbon radicals which jointly with the nitrogen form a heterocycle, saturated divalent hydrocarbon and ether radicals which jointly with the nitrogen form a heterocycle, and the monovalent radicals, aliphatic-oxy methyl, aralkyl, and aliphatic and alicyclic hydrocarbon, drying the fabric to which the cellulose derivative and quaternary ammonium salt have been applied, glazing it with a hot calender, and heating it to a temperature of at least 260° F. to react the cellulose derivative and quaternary ammonium salt.

2. The process of imparting to the surface of fabrics a wash-resistant, glazed finish which comprises applying to a fabric a cellulose derivative selected from a member of the class consisting of water-soluble cellulose ethers and alkali-soluble cellulose ethers, treating said cellulose derivative in the presence of the fabric with a quaternary ammonium salt having the formula—



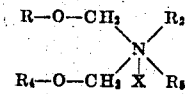
wherein R represents an aliphatic hydrocarbon group, X represents a salt-forming anion, and R_1, R_2, and R_3 are selected to satisfy the remaining valences of the nitrogen atom from the class consisting of trivalent unsaturated hydrocarbon radicals which jointly with the nitrogen form a heterocycle, saturated divalent hydrocarbon and ether radicals which jointly with the nitrogen form a heterocycle, and the monovalent radicals, aliphatic-oxy methyl, aralkyl, and aliphatic and alicyclic hydrocarbon, drying the fabric to which the cellulose derivative and quaternary ammonium salt have been applied, framing it to width, calendering it to produce a lustrous surface, and heating it to a temperature of at least 260° F. to react the cellulose derivative and quaternary ammonium salt.

3. The process of imparting to the surface of fabrics a wash-resistant, glazed finish which comprises applying to a fabric a water-soluble cellulose ether, treating said ether in the presence of the fabric with a quaternary ammonium salt having the formula—



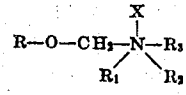
wherein R represents an aliphatic hydrocarbon group, X represents a salt-forming anion, and R_1, R_2, and R_3 are selected to satisfy the remaining valences of the nitrogen atom from the class consisting of trivalent unsaturated hydrocarbon radicals which jointly with the nitrogen form a heterocycle, saturated divalent hydrocarbon and ether radicals which jointly with the nitrogen form a heterocycle, and the monovalent radicals, aliphatic-oxy methyl, aralkyl, and aliphatic and alicyclic hydrocarbon, drying the fabric to which the cellulose ether and quaternary ammonium salt have been applied, glazing it with a hot calender, and heating it to a temperature of at least 260° F. to react the cellulose ether and quaternary ammonium salt.

4. The process of imparting to the surface of fabrics a wash-resistant, lustrous, glazed finish which comprises applying to a woven fabric a cellulose derivative selected from the class consisting of water-soluble cellulose ethers and alkali-soluble cellulose ethers, treating said cellulose derivatives in the presence of the fabric with a quaternary ammonium salt having the formula—



wherein R is an aliphatic hydrocarbon group, R_4 is an alkyl group, R_2 and R_5 are lower alkyl groups, and X is a salt-forming anion, drying the fabric to which the cellulose derivative and quaternary ammonium salt have been applied, glazing it with a hot calender, and heating it to a temperature of at least 260° F. to react the cellulose derivative and quaternary ammonium salt.

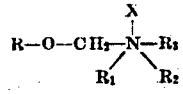
5. The process of imparting to the surface of fabrics a wash-resistant glazed finish which comprises applying to a fabric a cellulose derivative selected from the class consisting of water-soluble cellulose ethers and alkali-soluble cellulose ethers, treating said cellulose derivative in the presence of the fabric with a quaternary ammonium salt having the formula—



wherein R represents an aliphatic hydrocarbon group, R_1 and R_2 represent lower alkyl groups, X represents a salt-forming anion, and R_3 represents a benzyl radical, drying the fabric to which the cellulose derivative and quaternary ammonium salt have been applied, framing it to width, glazing it with a hot calender, and heating it to a temperature of at least 260° F. to react the cellulose derivative and quaternary ammonium salt.

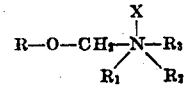
6. The process of imparting to the surface of fabrics a wash-resistant glazed finish which comprises applying to a fabric a water-soluble cellulose ether, treating said cellulose ether in the

presence of the fabric with a quaternary ammonium salt having the formula—



wherein R represents an aliphatic hydrocarbon group, R_1 and R_2 represent lower alkyl groups, X represents a salt-forming anion, and R_3 represents a benzyl radical, drying the fabric to which the cellulose ether and quaternary ammonium salt have been applied, framing it to width, glazing it with a hot calender, and heating it to a temperature of at least 260° F. to react the cellulose ether and quaternary ammonium salt.

7. The process of imparting to the surface of fabrics a wash-resistant, glazed finish which comprises applying to a fabric a solution containing a water-soluble cellulose ether and a quaternary ammonium salt having the formula—



wherein R represents an aliphatic hydrocarbon group, R_1 and R_2 represent lower alkyl groups, X represents a salt-forming anion, and R_3 represents a benzyl radical, drying the fabric to which the cellulose ether and quaternary ammonium salt have been applied, framing it to the desired finished width, glazing the dried and framed fabric with a hot calender, and heating it to a temperature between 260° F. and 400° F. for ten to three minutes.

8. The process of imparting to the surface of fabrics a lustrous, wash-resistant glazed finish which comprises applying to a fabric a solution containing a water-soluble cellulose ether and octyloxymethyl dimethyl benzyl ammonium chloride, drying the treated fabric, glazing it with a hot calender, and subjecting it to a temperature between 260° F. and 400° F. for three to ten minutes.

9. The process of claim 8 in which the water-soluble cellulose ether is an ethyl cellulose.

10. The process of imparting to the surface of fabrics a lustrous, wash-resistant glazed finish which comprises applying to a fabric a solution containing between 1 and 8% of a water-soluble cellulose ether and 3 to 20% of octyloxymethyl dimethyl benzyl ammonium chloride, drying the treated fabric, glazing it with a hot calender, and subjecting it to a temperature of at least 260° F. for at least three minutes.

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