This invention relates to the production of a finish on fabrics composed of cellulose and regenerated cellulose and mixtures thereof, and the primary object of the invention is to produce a durable to water finish which has the luster hereinafter described, which is crease, spot and soil-resistant, in which the individuality of the fibers and yarns is retained and the flexibility and elasticity of the fibers increased whereby the hand and drape of the fabric is superior to the original, in which the feel of the fabric is soft or "mellow" (not dry to the touch), in which the fabric "breathe" well (substantially retains its porosity), is resistant to fraying, and in which the fabric is stabilized against shrinkage, with substantial retention of the original tear strength.

As will be hereinafter set forth in detail, in the practice of our invention we employ a mechanical finishing machine and thermosetting resin-forming materials, which are applied in the water soluble state and polymerized into the water insoluble state, by heat. Such materials in relatively large concentrations have been employed to crease-proof fabrics, but the finished fabric has a dull or lack-luster appearance. Such resin-forming materials have also been used in combination with mechanical finishing to produce a fabric having a durable relatively highly glossy luster, such, for example, as a chintz fabric.

In the latter procedure, the finishing machine, whether a calendering machine, a friction calender, a smooth or flat nip calender, a schreiner calender or an embossing calender, operates with relatively high pressure, say, for example, a total pressure of 20 tons, 25 tons, 40 tons, or even higher, in some cases. In each case certain of the rolls of the calender are ordinarily heated. Due to the heat some polymerization of the resin-forming material occurs in the machine. Due to the pressure and the friction (which is intentionally present in the friction calender and unavoidably present to some extent in the other calenders) on the fabric in passage of the fabric through the machine, the polymerized material on the fabric is caused to flow and is given a polishing action. This flowing and polishing is enhanced by the presence of water, in which connection it is the custom to only partially dry the fabric before passage through the machine, the free moisture (about 10% or more over natural moisture content) in the fabric acting as a plasticizer of any polymerized material present. Due to the flowing and the polishing action, surface deposits of polymerized material are ironed into a continuous coating and are, as it were, "shined up" so that the resultant luster optically appears as a highly glossy surface luster. In the presence of such free moisture, which is also a plasticizer of cellulose, the pressure of the machine itself produces a relatively glossy finish luster which, as described, is enhanced by the flowing and polishing of surface deposits thereon. The fibers and yarns of the fabric lose in individuality and therefore suffer a substantial reduction in their flexibility and elasticity so that the finished product, after washing and drying, has been stiffened and therefore has lost in hand and feel, and the crease-resistance is relatively low. The flowing and polishing also tends to fill up or close interstices.

In the production of the finish of this invention we aim on the one hand to avoid the dull lack-luster appearance obtained in crease-proofing, and on the other hand to avoid the loss in individuality, flexibility and elasticity of the fibers and reduce the closing of interstices hereinefore described in connection with highly glossy finishes, while obtaining a luster which optically appears as an interior deep-seated luster or glow rather than a glossy surface luster and while obtaining quite substantial crease-resistance and the other properties of the finish hereinefore first described.

Generally speaking, in the practice of our invention we first prepare a water solution of the resin-forming materials, containing, in the case of cotton and the like, a permanent softener, and also containing a delayed action catalyst of the character to be hereinafter described, the resin-forming materials constituting from 5% to 20% of the solution by weight, with the softener constituting but a very minor part, from 1% to as high as 4% by weight of the solution, and with the catalyst constituting from 3% to 5% by weight of the solid resin-forming material.

The solution is applied to the fabric in any well known way as by impregnation, spraying, or the like, with a solution pick-up from approximately 70% to 90% on the weight of the fabric in its natural dry state. The fabric is now dried
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by running it through an atmosphere heated to from substantially 200° F. to 350° F. for a period sufficiently long to remove substantially all free moisture, and then cooling by passing over cooling cans, leaving behind substantially only the natural moisture at room temperature. In other words, a cotton fabric has a natural moisture content of from substantially 4% to substantially 6%, and rayon from substantially 7% to 10%.

The fabric is now run through the mechanical finishing machine either one or more times. In the case of melamine-formaldehyde, urea-formaldehyde and dicyandiamide-formaldehyde resins, the delayed action catalyst is of the type which has a pH on the dried fabric of from substantially 6 pH to substantially 8 pH as determined by indicator solutions, but which will develop high acidity at the relatively elevated temperatures hereinafter to be more particularly pointed out in connection with the polymerization of the resin-forming materials to the water-insoluble state. In other words, the catalyst gives for all practical purposes substantial neutrality or very low acidity on the fabric, when it has a temperature of substantially 212° F., which it will have for a few seconds at the time the warm moisture has just been driven off. The cooling is to drop the temperature of the fabric quickly to room temperature or below (say from 60° F. to 100° F.) under which conditions the polymerization is kept at a negligible minimum. (Out of excess precaution, one may also add ammonium hydroxide to the solution to maintain neutrality during drying and until the ammonia is driven off.)

The dried cool fabric is now run through the finishing machine, which is operated at such a temperature that the temperature of the fabric will always be at or below 200° F., as will further appear. (We prefer a range of temperature of the fabric of from 100° F. to 175° F.) By maintaining the temperature of the fabric in its passage through the heated calender below 200° F., it will be seen that the catalyst will remain substantially neutral and the pH will not fall below 6 pH, at which pH the rate of polymerization will be so low that for all practical purposes little polymerization can occur during passage through the calender.

After passage through the calender, the fabric is run through an oven in which the temperature and the time of residence are sufficiently high to cause the catalyst to develop marked acidity, say a pH of 4 or 5, whereupon the catalyst will catalyze the reaction and the resin-forming material will be polymerized and become water-insoluble. The fabric is now washed to remove unreacted materials and dried, when it is in the finished state.

It will be seen from the foregoing that at the time of the imparting of the mechanical finish there is little polymerization of the resin-forming material, the rate of polymerization being relatively exceedingly slow under the conditions; and, no substantially polymerized material being present, the luster imparted is that which is obtained essentially by the pressure and polishing action of the machine on the cellulose fibers and yarns themselves. In other words, the machine physically deforms the fibers and yarns only to the extent that they can be deformed by the machine. The luster imparted by the deformation and polishing of the fibers and yarns is thus of subdued character due to the absence of any appreciable free moisture or other plasticizer of cellulose. Since polymerization of the resin materials to the insoluble stage is so slow that only at a time when there is no pressure on the fabric, there is no flowing or polishing of the resin formed, in consequence of which the luster of the finished product, i.e., the washed and dried fabric, is only that which is obtained in the case of machine on the fibers and yarns themselves, which luster is seen through the surface resin and appears not as a glossy surface luster but as an interior or deep-seated luster or glow. The fibers substantially retain their original individuality instead of being imbedded in a hard brittle resinous mass as in the case of regular highly glossy glazed chintz. This individuality, together with the increased flexibility caused by resin deposition, gives the finished fabric a hand, drape and flexibility superior to the original. The size of the interstices has been to a large extent retained and the fabric has a good porosity when compared with the original. The finished fabric has far more crease-resistance than does durable chintz, for example, and has a crease-resistance approaching that obtained in the ordinary procedures followed in producing crease-resistant cotton. The finish is durable to repeated washings and dry-cleanings and has the other properties hereinbefore set forth. Since the hard resinous material produced in the polymerization in the oven is formed when there is no pressure, the softener used in the case of cotton is not the most effective to act, as were, as a lubricant whereby the original tear strength is substantially retained. In this respect the fact that the catalyst becomes actively acid in the oven, also tends to the retention of tear strength. In the case of regenerated cellulose a softener is not needed to retain strength.

For thermosetting resin-forming materials we may employ any acid-catalyzable materials, such, for example, as urea-formaldehyde, thiourea-formaldehyde, dicyandiamide - formaldehyde mixtures thereof, melamine-formaldehyde, mixtures of melamine and urea or thiourea dicyandiamide formaldehyde, and in fact any of the acid catalyzable resin-forming materials used in the textile art for producing crease-resistance. For the formaldehyde, glyoxal may be substituted. The resin-forming materials may be employed in the unreacted state or may be partially condensed to the so-called "A" stage in which they are still water soluble.

For the catalyst we prefer to use carbamide hydrochloride. This may be substituted by any of the following which are the equivalents for the carrying out of the process, namely, phenyl biguanide hydrochloride; monoguanil urea phosphate; di-monoethanolamine hydrogen phosphate; octadecloxymethyl pyridinium chloride; and similar products such as "Catalyst AC" (an organic nitrogen chloride); and for the permanent softener when used we prefer to use octadecloxymethyl pyridinium chloride for which any of the following may be substituted as equivalents, namely, for the permanent softener we use the so-called substantive cation active type such as Sapamine KW (trimethyl ammonium methyl sulfate of monostearilmetaphenylenediiline), Triton K-80 (tetra alkyl quaternary ammonium chloride), Ammonyx T (trivialbenzyl ammonium chloride), and numerous other similar products sold under such commercial names as "Soromine," a quaternary derivative of a
fat acid, "Aheovel," s-Di(1-(3-palmitamido-ethyl)) environ monoacetate, "Aerotex Softener H," a mixed ebonite and antioxy long chain hydrocar- 

derivative, and so forth. These softeners 

will withstand the heat of the process and re-

main permanently. (Octadecyloxymethyl pyri-

dinium chloride is both a catalyst and a softener.)

When producing the finish on the chasing 

calender, say, for example, a thirty-ton 5-bowl 
calender with three heated steel bowls and two 
cotton bowls, the steel bowls are heated to a 
temperature of from substantially 250° F. to 

300° F. In such a calender, 15 tons' pressure is 

applied to each ram. Calculated on the linear 

basis, with bowls 60” wide, the pressure is one-

half ton per inch on a 60’ fabric. The fabric 

is given a number of passes, preferably four, 

each through the nip of the 5-bowl calender, i.e., 

there will be four thicknesses or layers of the 

fabric in the nip between each pair of bowls at 

all times. Under these conditions the tempera-

ture of the fabric will remain well below 175° F. 
The number of passes may be increased or de-

creased, so long as the temperature achieved by 

the fabric is not above 175° F. 

After the chasing calender, the fabric is run 

through an oven having an atmosphere heated 

to a temperature of from substantially 250° F. to 

substantially 350° F. The time in the oven is 

from substantially 8 minutes to substantially 

2 minutes. In the oven the resin-forming ma-

terials are polymerized and become water in-

soluble. 

In the case of a plain or flat nip calender, 

with bows rotating at the same peripheral speed, 

the heated bows may be safely heated to a 
temperature from 250° F. to 350° F., at customary 

operating speeds which are usually in the neigh-

borhood of 150 yards per minute. These calen-

ders are usually of the multi-bowl type having 

from three to seven bowls, although two-bowl and 
nine-bowl machines are known. There are, fo-

r instance, seven- to nine-bowl calenders. The heated 

bowl in this case may be operated at a tempera-

ture of from substantially 300° F. to substan-

tially 350° F. at the customary speeds of oper-

ation, which are usually in the neighborhood of 

60 yards per minute. In this case the fabric 

is desirably passed through these calenders 

once only, in order to provide nearly the same 

temperature of the heated bowls such as to ensure 

that the fabric does not achieve a temperature in 

excess of 175° F. Where the number of passes is 

smaller, the fabric may be run through two or 

three times, providing again that the tem-

perature of the heated bowls is not sufficient to 

cause the fabric to achieve a temperature above 

175° F. 

In the case of a shrinker or an embosser, the 

fabric can of course be only run through once. 

Due to a temperature on the heated bowl may be 

relatively somewhat higher, but in no case should 

be such as to cause the fabric to achieve a tem-

perature of substantially above 175° F. 

In such machines a temperature as high as 350° F. may 

be safely obtained at customary operating speeds, which are usually in the neighborhood of 

15 yards per minute.

In the case of the friction glazer, the fabric 

is passed through the nip but once or twice in-

stead of the three or more times employed in 

the former. A durable finish may be obtained 

in this case by using a 5-bowl calender. The soften-

ers heretofore listed are also lubricants so they 

may be used in the double capacity in the case 

of cottons. In the case of rayon, they act as 
lubricants. In the latter case, non-permanent 

softeners or lubricants can be used, such as sul-

fonated castor oil, sulfonated hydrocarbon (Avi-
tone A) and polymerized ethylene oxide (car-

boxwaxes) may be used, and this material is large-

ly removed during the final washing.

Example I

1. Solid resin 5%

2. Cotton

3. Chase—high pressure—low temperature

4. Cure—short time—high temperature

5. Dry—high temperature

40.0 lbs. Aerotex M-3 (dimethyl trimethyl 

melamine-formaldehyde 80% solid resin) 

20.0 lbs. Aerotex #450 (dimethyl urea-

formaldehyde resin 50% solid)

10.0 lbs. Triton K-60

4.0 lbs. monoguanyl urea phosphate

1.0 lb. ammonium hydroxide (28%) to neu-

tralize 

Water to make 100 gallons.

A cotton fabric was impregnated with the above 
solution and squeezed until a solution "pick-up" of 

70% was obtained.

This cloth was then dried in an atmosphere of 

350° F. until approximately 6% moisture (natu-

ral) remained in the fabric which was then 

quickly cooled to room temperature substan-

tially 85° F.

The fabric was then chase calendered in a 

5-bowl calender machine using 50 ton total pressure and 

250° F. on the steel bowls. The speed of the 

machine was 170 yards a minute.

The fabric was then cured in an oven for 2 

minutes at 350° F. and finally washed and dried.

The resulting fabric had a deep-seated mellow 

glow and a soft dry-erapy hand and was more re-

sistant to creasing than the original fabric.

Example II

1. Solid content high 20%

2. Rayon

3. Chase—low pressure—high temperature

4. Cure—long time—low temperature

5. Dry—low temperature

46.0 lbs. methylol urea-formaldehyde resin 

(50%)

20.0 lbs. Aerotex #801 (dimethylol urea-

formaldehyde resin 109% solid)

35.0 lbs. Sapamine K. W.

20.0 lbs. catalyst A. C.

1.0 lb. ammonium hydroxide (28%) to neu-

tralize 

Water to make 150 gallons.

A spun rayon fabric was impregnated with the 

above solution, squeezed until 80% by weight of 

the solution was retained by the fabric which 

was then dried in a drying unit operating at a 

temperature of 250° F. until approximately a total 

of 8% moisture (natural) remained in the fabric. 

It was then quickly cooled to approximately 65° F. 

by passing the fabric over cooling cans.

The sized, dried, and cooled fabric was passed 

once through all the nips of a 5-bowl chasing 

calender operating at 20 ton total pressure and a 

temperature of 300° F., the speed of the calender 

being 160 yds. a minute.

The fabric after curing 3 minutes at 250° F. in 

an oven was washed to remove soluble materials 

and then dried.

The resulting fabric had a deep-seated mellow 

lustrous finish and a soft, dry-erapy crease-re-

sistant hand.
Example III

1. Solid 12%  
2. Cotton  
3. Chase—medium pressure—medium temperature  
4. Cure—medium time—medium temperature  
5. Dry—medium temperature  

100.0 lbs. Resloam HP (dil and trimethyl melamine-formaldehyde resin)  
30.0 lbs. Acovel G  
2.0 lbs. di-monoethanolamine hydrogen phosphate  
1.0 lb. ammonium hydroxide (28%) to neutralize  

Water to make 100 gallons.

A cotton printed broadcloth fabric was impregnated with the above solution and squeezed to remove excess solution. It was then carefully dried in a tenter frame at 300°F to natural moisture and then quickly cooled to substantially 70°F.  
The fabric was then chase calendared by running it once through a 5-bowl calender operating at 30 tons and 275°F. The cloth was then cured 5 minutes at 300°F, and finally washed and dried.  

A beautiful deep-seated lustrous flexible fabric was obtained with increased crease-resistance and good strength.

Example IV

1. Rayon  
2. Glass  

60.0 lbs. Aerotex M-3  
100.0 lbs. methyl urea-formaldehyde resin (50%)  
20.0 lbs. Avitone A  
8.0 lbs. carboxide hydrochloride  
1.0 lb. ammonium hydroxide (28%) to neutralize  

Water to make 100 gallons.

A spun dyed rayon fabric, 40", 40/40, 3.50 yds./lb., was impregnated with the above solution, squeezed, dried, and cooled as described in Example III.  

This fabric was glazed once by passing it through the glazing calender operating at 40 tons, 325°F temperature and a friction ratio of 2:1.  

The resulting fabric was cured 6 minutes at 285°F, and then washed and dried.  

The resulting fabric had a good deep-seated luster and a linen-like flexible hand and good crease-resistance.

Example V

80.0 lbs. Aerotex M-3  
60.0 lbs. Aerotex #450  
20.0 lbs. Avitone A  
10.0 lbs. catalyst A. C.  
1.0 lb. ammonium hydroxide (28%) to neutralize  

Water to make 100 gallons.

A 45", 39/32, 2.25 yds./lb. spun rayon (75%) cotton (25%) fabric was impregnated, squeezed, dried, and cooled as described in Example III.  

The fabric was then run once through a 5-bowl chase calender at 40 tons pressure and 250°F, followed by curing 3 minutes at 275°F. This was followed by washing and drying.  

The resulting fabric had a linen-like hand and appearance. In addition the fabric was very flexible and decidedly resistant to creasing.

Example VI

1. Solid 11%  
2. Glaze—high pressure—high temperature—high friction  

30.0 lbs. Aerotex M-3  
50.0 lbs. dicyandiamide-formaldehyde resin (50%)  
30.0 lbs. Triton K-60  
6.0 lbs. carboxide hydrochloride  
1.0 lb. ammonium hydroxide (28%) to neutralize  

Water to make 100 gallons.

A printed, 39", 80/92, 3.50 yds./lb., cotton fabric was sized and dried and cooled in the previously described manner in Example III. It was then passed twice through a glazing calender at 50 ton total pressure at a temperature of 350°F and with a friction ratio of 2:2:1.  

The fabric was then cured, washed, and dried in the usual manner.  

A deep-seated lustrous, flexible, full fabric of good crease-resistance was obtained.

Example VII

1. Solid high 20%  
2. Rayon  
3. Flat nip—high pressure—low temperature  
4. Cure—medium  
5. Dry  

460.0 lbs. methyl urea-formaldehyde (50%)  
28.0 lbs. Rhonite #513 (dimethyl urea-formaldehyde resin 50%)  
20.0 lbs. Glyoxal  
20.0 lbs. catalyst A. C.  
1.0 lb. ammonium hydroxide (28%) to neutralize  

Water to make 150 gallons.

A spun rayon fabric was impregnated and squeezed with the above solution and then carefully dried to natural moisture and cooled as described in Example III.  

This fabric was given one run through a regular 5-bowl flat nip calender operating at 40 tons and 350°F, followed by curing 5 minutes at 300°F.  

After washing and drying, a deep-seated low luster crease-resistant fabric of a good full hand was obtained.

Example VIII

1. Cotton  
2. Schreiner  

80.0 lbs. Aerotex M-3  
60.0 lbs. Aerotex #450  
10.0 lbs. Sapsamine K. W.  
10.0 lbs. catalyst A. C.  
1.0 lb. ammonium hydroxide (28%) to neutralize  

Water to make 100 gallons.

A sateen dyed cotton fabric was sized, dried, and cooled as previously described in Example III and then passed once through a schreiner calender operating at 40 tons pressure and 300°F on the engraved steel bowl.  

The fabric was then cured at 350°F for 2 minutes, washed in open width with minimum warp tension and then dried.  

The resulting fabric had a beautiful deep-seated mellow glow or sheen and a flexible lively hand.

Example IX

1. Cotton  
2. Embossing
A plain weave dyed cotton fabric was treated as described in Example VIII except that the fabric was passed through an embossing calender instead of the schreiner. The results were the same, except that there was also an embossed effect.

We claim:

1. The process of finishing fabrics of cellulose and regenerated cellulose and mixtures thereof which comprises impregnating the fabric with an aqueous solution of water-soluble acid-catalyzable thermosetting textile resins of the aldehyde type in concentration of from substantially 5% to substantially 20% by weight of the solution and carbazide hydrochloride as a catalyst, in minor part by weight, with a solution pick-up of from substantially 70% to substantially 90% by weight of the fabric in the dry state, drying the fabric at temperatures between about 200° F. and about 350° F. until it is substantially devoid of free moisture, the drying being at a temperature and for a time such that the pH of the dried fabric is not below 6 pH as determined by indicator solutions, promptly after such drying, cooling the fabric to substantially room temperature, passing the dried, cooled fabric through a pressure mechanical finishing calender heated to a temperature such that the fabric is heated to a temperature of between from substantially 100° F. to substantially 200° F. in its passage therethrough, and passing the fabric through an atmosphere heated to from substantially 250° F. to substantially 350° F. for a residence time of from substantially 8 minutes at the lower temperature to substantially 2 minutes at the higher temperature.

2. The process of finishing fabrics of cellulose and regenerated cellulose and mixtures thereof which comprises impregnating the fabric with an aqueous solution of water-soluble acid-catalyzable thermosetting textile resins of the aldehyde type in concentration of from substantially 5% to substantially 20% by weight of the solution, octadecyloxymethyl pyridinium chloride as a softener and carbazide hydrochloride as a catalyst, each in minor part by weight, with a solution pick-up of from substantially 70% to substantially 90% by weight on the fabric in the dry state, drying the fabric at temperatures from about 200° F. to about 350° F. until it is substantially devoid of free moisture, the drying being at a temperature and for a time such that the pH of the dried fabric is not below 6 pH as determined by indicator solutions, promptly after such drying, cooling the fabric substantially to room temperature, passing the dried, cooled fabric through a pressure mechanical finishing calender heated to a temperature such that the fabric is heated to a temperature of from substantially 100° F. to substantially 200° F. in its passage therethrough, and passing the fabric through an atmosphere heated to from substantially 250° F. to substantially 350° F. for a residence time of from substantially 8 minutes at the lower temperature to substantially 2 minutes at the higher temperature.

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