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CHEMICAL PROCESS

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This invention relates to the treatment of tex-
tiles, particularly cellulosic fibers, yarns and fab-
rics, with sulfuric acid to obtain parchmentizing
effects; and more particularly to the treatment
of cotton fabrics with concentrated sulfuric acid
containing an agent which will inhibit the tender-
ing effect of the acid.

It is generally known that concentrated sul-
furic acid is destructive to textile materials,
nevertheless certain uses of sulfuric acids on tex-
tiles are so advantageous that endeavors have
been made to so treat textiles with sulfuric acid
as to obtain the desired effects but to avoid the
undesirable effects. Thus there is a series of pat-
ents which deal with the treatment of fabrics
with sulfuric acid in connection with a series of
mercerization treatments.

This invention has as an object the production
of permanent finishes on fabrics. A further ob-
ject is the production of permanent finishes on
yarns, textiles, fabrics, paper and the like. A
still further object is the production of parchmen-
tized effects on cellulosic fabrics. A still further
and particularly desirable object is the produc-
tion of organdy finishes on cotton fabrics. Other
objects will appear hereinafter.

These objects are accomplished by the follow-
ing invention wherein the fabric to be treated
is passed continuously through the parchmen-
tizing bath consisting of especially inhibited
strong sulfuric acid, thence through rolls or other
means for squeezing out the excess parchmen-
tizing reagent, then through washing and neutraliz-
ing baths, and finally over a calender or other
drying means. In this procedure the speed and
time of immersion of the goods in the parchmen-
tizing bath are adjusted with due regard to the
result desired and to the end that the goods are
transparentized to the desired degree and that
the parchmen-
tizing reagent is washed out before
the goods are tendered to an objectionable degree.

The novel feature of this process is the parch-
mentizing reagent which consists of strong sul-
furic acid of at least 65% concentration and pref-
erably from 80% to about 98%, having dissolved
therein an agent capable of inhibiting or at least
greatly retarding the normal degrading action of
strong sulfuric acid upon cellulose. This agent is
termed the cellulose degradation inhibitor. In
the claims the percentage composition of the acid
refers to the composition of the acid prior to
the introduction of the inhibitor.

It is known that strong sulfuric acid has a
marked degrading or disintegrating effect on cel-
lulose fibers, which in the case of acid of moder-
ate strength quickly produces a marked tender-
ing of the fiber and in the case of concentrated
sulfuric acid such as 96%, results in almost in-
stantaneous and complete destruction of the
fiber.

Now it has been discovered that the degrading
effect of the acid on the cellulose may be inhibited
or retarded by dissolving in the acid certain
agents which are termed inhibitors of cellulose
degradation. An inhibitor suitable for use in this
invention may be known by the fact that it meets
the following tendering resistance test. For
this test, 20 parts of the material to be tested is
dissolved in 100 pts. of 96% sulfuric acid, the
solution is brought to 20° C. and a sample of cot-
ton voile of known tensile strength is immersed
therein for a period of 30 seconds after which
the sample is withdrawn from the acid, washed to
neutrality, and dried by ironing. The sample is
now examined for parchmen-
tizing and tested for
loss or gain of tensile strength. If the sample is
found to be parchmen-
tized and to have increased
in tensile strength or to have lost less than 25%
of its original tensile strength, it is considered
that the agent tested is a satisfactory inhibitor
of degradation for use in this invention.

If a cotton fabric is immersed in 96% sulfuric
acid at room temperature, removed immediately
and rinsed very quickly with a large volume of wa-
ter, the fabric is disintegrated and destroyed.
However, if as much as 25 parts by weight of urea
is first added to each 100 parts of acid, then a
fabric may be treated with this solution up to 60
seconds without being appreciably tendered. If
fabrics so treated are washed free of acid, using
dilute alkali if desired, then ironed dry, a pleas-
ant, stiff, springy finish is obtained. When this
treatment is applied to cotton voile or lawn, an
organdy finish results. Not only is the cloth stiff
and springy, but it is also more transparent than
the original cloth. The effect is permanent to
washing. Instead of urea, certain other materials
may be used. These are listed below.

In carrying out the process in its preferred
form, the agent is first dissolved in concentrated
sulfuric acid with cooling if necessary to prevent
decomposition. The sulfuric acid may be from
about 65 and preferably from 85 to over 100 per
cent strength. From 10-30 parts by weight of
agent per 100 parts of acid is satisfactory. The
quantity of agent, as well as the concentration
of initial acid, may be varied somewhat depend-
ing on the time the textile material is to be in
contact with the solution, and depending on the
effect it is desired to produce. The parchmen-
tizing is slower and less pronounced with increasing
quantities of inhibitor. The time of contact of
the textile material with the solution will ordi-
narily be determined by the speed at which the
fabric sheet is run through the solution. From
10 to 30 seconds is usually sufficient, but under
suitable conditions the time of contact may be
60 seconds or more, without damage to the fabric.

The solution may be used at room temperature
or at temperatures below room temperature.

Lowering the temperature retards the parchmentizing effect. It will be seen that the degree of parchmentizing obtained can be varied at will over very wide limits by varying (1) the amount of inhibitor in the acid, (2) the amount of water in the acid, (3) the temperature of the solution, and (4) the time of contact of the cellulosic material with the solution.

The textile material, e. g., cotton fabric, is passed through the acid solution and then into water to remove the acid, then dried by ironing or calendering, or on a tenter frame which exerts a shearing action to minimize sticking together of the warp and filling threads at points of contact. The washing may be facilitated by using water first, followed by a dilute solution of a base such as ammonium hydroxide or sodium hydroxide. It is important that the greater part of the acid be washed out very rapidly in order to avoid local heating which would destroy the fabric. If a cellulosic material is treated in a solution of one of these agents and sulfuric acid, and then slowly immersed in water, the cellulosic material will be tendered or totally destroyed.

A novel fabric has been produced by treating cotton web, produced on a carding machine, with these inhibited sulfuric acid solutions. The fibers are stuck together at each point of contact, resulting in a strong sheet. This process affords a possibility of omitting the steps of spinning and weaving in the preparation of fabrics suitable for a variety of purposes.

Instead of using a single agent in the solution two or more may be used from the same or from different groups, or certain other agents may be added to give enhanced effects. A solution containing an amide and an ether, or an amide and an alcohol, or an amide and an amine, will give very satisfactory parchmentizing effects with practically no tendering of the cellulosic material. The use of additional agents also affords means of varying the effects obtained. Thus, an amide alone or an amide and an amine, or an amide and an alcohol, produce transparent organdy finishes, whereas an amide and a relatively large proportion of an ether produce an opaque parchmentized effect. Also, some agents give a more transparent finish than others. Thus, acetanilide and decamethylenediamine give opaque finishes.

If only limited areas of a fabric are treated by this process, a pattern effect may be obtained. Alternatively, certain areas of the fabric may be immunized against the sulfuric acid treatment and the fabric may then be treated over its entire surface with the acid to obtain pattern effects. Thus, areas printed with paraffin are not affected by the acid treatment, and this affords a means of immunizing against sulfuric acid.

Unfinished cotton fabrics can be treated by this process without any previous or subsequent mercerization and will yield, by a single treatment, a transparent organdy finish. The advantage of this process is obvious when it is considered that the present process of producing permanent organdy finishes normally involves three major operations: (1) mercerization, (2) acid treatment, and (3) mercerization; or (1) acid treatment, (2) mercerization, and (3) acid treatment. If some particular effect is desired, fabrics treated with concentrated sulfuric acid containing an inhibitor may be previously and/or subsequently mercerized, or mercerization may be carried out between two acid treatments, but when applying the process of this invention, the

mercerization step is not necessary to produce a good permanent organdy finish.

Instead of washing the fabrics in water after the acid treatment, they may first be washed in aqueous sulfuric acid (25 to 50 per cent H_2SO_4) and then washed in water. This reduces the cost of the treatment, since the dilute sulfuric acid produced, may be utilized in other operations.

While the use of inhibited sulfuric acid is of particular interest and value on cotton fabrics, for producing organdy effects, linen, and rayon may also be treated by this process with advantageous results. In this manner an organdy finish can be obtained on rayon voiles.

Fabrics or yarns treated by this process may be stretched during or after the acid treatment. The tenacity of cellulosic yarns is increased by stretching during or after the acid treatment.

In the above the invention has been considered in considerable detail, the principles of the invention being set forth minutely. The following examples still further illustrate the invention, but are purely illustrative and not limitative.

Example 1

To 100 g. of concentrated sulfuric acid (sp. gr. 1.84, 95.5-96.5 per cent H_2SO_4) was added 20 g. of urea, the acid being cooled during the addition of the urea. After the urea was completely dissolved, the solution was cooled to 20° C. A 68/76 unfinished cotton voile fabric was immersed in the solution for 30 seconds, removed, drained for a few seconds, then plunged into water at room temperature. After washing thoroughly in water it was rinsed in a 3 per cent solution of ammonium hydroxide, rinsed in water and dried by ironing. A stiff, springy finish of the type commonly called transparent organdy was obtained. The fabric was boiled for one hour in 0.5 per cent soap solution, rinsed and dried. The finish was not appreciably affected by this laundering.

If it is desired, the fabric may be mercerized with caustic in the usual way either before or after, or both before and after, the above acid treatment. Alternatively, two acid treatments of the type indicated may be given the fabric with mercerization between the two acid treatments.

Example 2

A 68/76 unfinished cotton voile fabric was immersed for 60 seconds in a solution of 20 g. thiourea in 100 g. 96% sulfuric acid, removed, drained for a few seconds, rinsed in water, then in dilute ammonium hydroxide, and again in water, and dried by ironing. A transparent organdy finish was obtained which was permanent to washing.

Example 3

A 68/76 unfinished cotton voile fabric was immersed for 20 seconds in a solution of 20 g. formamide in 100 g. 96% sulfuric acid. It was then removed, drained for a few seconds, quickly rinsed in water, dilute ammonium hydroxide and water, then dried by ironing. A permanent transparent organdy finish was obtained.

Example 4

A 68/76 unfinished cotton voile fabric was treated for 20 seconds with a solution of 20 g. of acetamide in 100 g. 96% sulfuric acid. The fabric was then removed, drained for a few seconds, washed with water, dilute ammonium hydroxide and water, and dried by ironing. A transparent organdy finish was obtained. This finish

was permanent to washing although the fabric was slightly tendered by the treatment.

Example 5

6 A 68/76 unfinished cotton voile fabric was immersed for 20 seconds in a solution made by adding 20 g. oxamide to 100 g. 96% sulfuric acid. The oxamide was only partially soluble in the acid. The fabric acquired a permanent transparent organdy finish but was slightly tendered.

Example 6

15 A 68/76 unfinished cotton voile fabric was treated for 20 seconds with a solution of 20 g. acetanilide in 100 g. 96% sulfuric acid. An opaque organdy finish was obtained and the fabric was only very slightly tendered by the treatment.

Example 7

20 A 68/76 unfinished cotton voile fabric was treated for 30 seconds in a solution of 25 g. of diethyl ether in 100 g. of 96% sulfuric acid. After the fabric was washed and dried it possessed a transparent organdy finish which was permanent to washing.

Example 8

30 An unfinished cotton voile fabric was treated for 10 seconds with a solution of 25 g. of ethyl alcohol in 100 g. 96% sulfuric acid, then washed in water, dilute ammonium hydroxide and water, and dried. A transparent organdy finish was obtained which was permanent to washing.

Example 9

35 An unfinished cotton voile fabric was treated for 20 seconds in a solution of 25 g. of acetone in 100 g. 96% sulfuric acid, washed and dried. A permanent organdy finish was obtained.

Example 10

40 An unfinished cotton voile fabric was treated for 10 seconds with a solution containing 20 g. of guanidine carbonate in 100 g. 96% sulfuric acid. A pleasing permanent organdy finish was obtained.

Example 11

50 An unfinished cotton voile fabric was treated for 10 seconds with a solution containing 25 g. of phenyl hydrazine in 100 g. 96% sulfuric acid. A permanent organdy finish was obtained. The fabric was slightly discolored.

Example 12

55 A 68/76 unfinished cotton voile fabric was treated for 10 seconds in a solution of 20 g. of urea and 5 g. of diethyl ether in 100 g. of 96% sulfuric acid. After the fabric was washed and dried, it possessed a transparent organdy finish which was permanent to washing. The tensile strength of the fabric was slightly greater than that of a fabric treated in a solution containing only urea and sulfuric acid.

Example 13

70 A 68/76 unfinished cotton voile fabric was treated for 20 seconds with a solution of 5 g. urea and 20 g. diethyl ether in 100 g. 96% sulfuric acid. The acid was washed out with water and dilute ammonium hydroxide and the fabric dried by ironing. An opaque organdy finish was obtained, the treated fabric being stiff and springy but no more transparent than the untreated fabric.

Example 14

A 68/76 unfinished cotton voile fabric was treated for 10 seconds with a solution of 20 g. urea and 5 g. ethyl alcohol in 100 g. 96% sulfuric acid. The fabric was washed free of acid and dried by ironing. A transparent organdy finish of good tensile strength was obtained.

Example 15

A 68/76 unfinished cotton voile fabric was treated for 20 seconds with a solution containing 5 g. of urea and 20 g. of ethyl alcohol in 100 g. 96% sulfuric acid. A transparent organdy finish of high tensile strength was obtained.

Example 16

A 68/76 unfinished cotton voile fabric was treated for 20 seconds with a solution containing 25 g. of urea and 10 g. of water in 100 g. 96% sulphuric acid. After washing free of acid and ironing, a very stiff, transparent finish was obtained.

Example 17

A 68/76 unfinished cotton voile fabric was treated for 5 seconds with a solution containing 25 g. of urea and 10 g. of water in 100 g. 96% sulfuric acid. After washing free of acid and ironing, the fabric possessed a stiff, transparent organdy finish much less stiff than the fabric obtained in Example 16. The fabric was not appreciably tendered.

Example 18

A 68/76 unfinished cotton voile fabric was treated for 10 seconds in a solution containing 25 g. of urea and 100 g. of 100 per cent sulfuric acid. The fabric was washed free of acid and dried by ironing. A permanent transparent organdy finish was obtained.

Example 19

A viscose rayon crepe fabric was treated for 10 seconds with a solution containing 25 g. of urea in 100 g. 96% sulfuric acid. The fabric was then washed free of acid and dried by ironing. The fabric was stiffer and more transparent than the original fabric and was only very slightly tendered.

This process can be applied to cotton, linen, viscose rayon or other regenerated cellulose fabrics, threads or fibers. Although the chief purpose of the invention is to produce organdy finishes on cotton voile fabrics, permanent starched effects may be obtained on heavier fabrics. The invention is applicable to all types of cellulosic fabrics, but the results are particularly desirable with textile materials such as cotton, viscose rayon, etc. The process is also applicable, as shown in the following example, to the preparation of parchment paper.

Example 20

An unsized sulfite paper weighing 20 lbs. per 500 sheets measuring 25"×38" was treated for 30 seconds in a solution of 20 g. of urea dissolved in 100 g. of 96% sulfuric acid, then washed in water until free of acid. The paper was dried by ironing. As a result of this treatment the paper was parchmentized. It was denser, harder, and more transparent than the untreated paper. The treated paper has a much greater wet strength as revealed by a qualitative test in which both treated and untreated paper were wet and broken in the hand. The resistance of the wet paper to rubbing was also greatly increased by the treat-

ment. The grease resistance of the treated and untreated paper was tested by placing the papers over white blotting paper and placing a drop of dyed turpentine on each in one test and a drop of a dyed cotton seed oil on each in a second test, and noting the time required for the oil to pass thru the paper. Both the turpentine and vegetable oil penetrated the untreated paper instantly. The turpentine penetrated the treated paper in about one minute but the cotton seed oil had not penetrated after a quarter of an hour. When written upon with ink the treated paper gave a smooth even line whereas the untreated paper gave an irregular line due to the blotting effect.

The sulfuric acid used in the parchmentizing reagent may vary from about 80 to 100% H_2SO_4 or even higher and is preferably from about 90 to 98% H_2SO_4 . When the inhibitor of cellulose degradation is an amide, for example, urea, useful results may be obtained with acid concentrations as low as 65% H_2SO_4 . The concentration of the inhibitor in the acid may vary within rather wide limits depending on the inhibitor itself and on the other conditions present. Under some conditions ratios as low as 5 parts inhibitor to 100 parts acid may be useful and ratios as high as 35 parts per 100 of acid may also be used provided the inhibitor is sufficiently soluble in the acid. The process is most desirably operated at normal temperature but the temperature may be varied somewhat above or below this point as desired.

In view of the number of variable factors, it is impossible to set forth in a patent application every possible combination of conditions which may be successfully operated. Given the requirement that a given quality of finish is to be produced on a given fabric using a given inhibitor of degradation, it is essential that the variables be adjusted so as to coordinate the reaction rate and the time of treatment so that a finish is obtained having the required properties. The various factors affecting speed of reaction are temperature, concentration of acid, and the concentration of the inhibitor in the acid. The reaction rate may be increased by increasing the temperature, or by varying the concentration of the acid, or by decreasing the concentration of the inhibitor in the acid. In general the speed of the parchmentizing reaction is increased as the concentration of the acid approaches the point of maximum activity which is about 92%. Since the process is in general operated continuously, it is obvious that the time of contact may be adjusted by varying the speed of travel or by varying the length of travel in the parchmentizing bath.

When the situation is such that the time of contact may be varied at will, the inhibitor to be used, for example, urea, is dissolved in the acid, the concentration of the acid and of the inhibitor in the acid being maintained at any desired point within the limits which have been set and within the limit of solubility of the inhibitor in the acid, the parchmentizing bath is brought to the temperature at which it is desired to operate, preferably around normal, and the fabric is passed continuously through this acid bath, the speed of travel being adjusted so that the degree of parchmentizing is satisfactory and that the reagent is washed out of the fabric before the fabric is tendered to an objectionable degree. If desired, the optimum time of contact in the parchmentizing bath may be readily determined merely by immersing a small sample of the fabric in the parchmentizing reagent and varying the time of immersion until

the required effect is produced after washing out the reagent and drying.

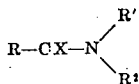
In the case where the time of treatment is limited, it may be necessary to adjust the speed of reaction between the cellulose and the reagent so that the reaction is completed within the time limit. For example, when it is desired to complete the reaction in shorter time, the reaction rate must be correspondingly increased and this result may be effected by increasing the temperature, or by decreasing the concentration of the inhibitor in the acid or by varying the concentration of the acid. When it is desired to reduce the reaction rate so as to permit a longer time of contact, this may be brought about by decreasing temperature or by varying the concentration of the acid or of the concentration of the inhibitor in the acid.

The process is not limited to any particular type of apparatus. The nature of the reagents will necessitate apparatus which is not corroded by concentrated sulfuric acid. With proper precautions, iron apparatus may be satisfactory. The treatment of the fabric may consist in passing it through the acid solution containing the inhibitor at such a rate that it will be thoroughly wet out by the acid, through squeeze rolls to remove excess acid, then into water at room temperature or below. It is essential that the water treatment should be such that the acid is diluted very rapidly. This will avoid having local areas become hot enough to cause tendering of the fabric. Subsequently, the fabric should be washed entirely free of acid using dilute alkali baths if desired, and then dried, either on a tenter frame or on hot rolls or calenders. The fabric may be given any other treatment desired for any particular finish. Thus, it may be tenter sheared to break down the bond at the intersection of warp and filling yarns, thereby preventing the finished organdy from having a "boardy" feel. For the most part the treatment in accordance with the present invention may be carried out in ordinary textile plant equipment.

The fabric may be treated over its entire surface or only on certain areas, so as to give pattern effects. For this treatment, the acid containing the inhibitor could be printed on by ordinary printing methods, using corrosion-resistant printing rolls. The treatment may be carried out by protecting certain areas with unreactive materials, such as paraffin, and then treating the entire fabric. In this way, the protected areas are not affected, and a pattern effect may also be obtained.

The process of the present invention has been illustrated with the use of amides which are the preferred class of inhibitors or negative catalysts of the present invention. In addition, the use of ethers, alcohols, ketones, amines and mixtures of ethers and amides have been exemplified. As explained earlier in this specification, suitable inhibitors are soluble in 96% H_2SO_4 and may be known by the fact that they react positively in the tendering resistance test. Those inhibitors which are of low molecular weight are particularly desirable. Thus urea, thiourea, formamide and ethanol-formamide are excellent inhibitors. Acetamide and acetanilide are effective but not to the extent of those just mentioned. Oxamide and decamethylene-diamine formamide may be used, as well as the amides of formic acid with forked chain amines wherein the alkyl group of the amines contains at least six carbon atoms and a forked chain.

These low molecular weight amido compounds are defined by the formula



wherein R may be hydrogen, an amido (NH₂) group or an alkyl group, R' and R'' are hydrogen or hydrocarbon radicals and X may be oxygen, sulfur or an imino group. These compounds are particularly effective when the compound contains 10% or more of nitrogen. In addition to those already mentioned guanidine, diphenylguanidine, urea-formaldehyde resins and aminoguanidine bicarbonate are effective as inhibitors to varying extents.

In addition to the amido compounds, above discussed, which form perhaps the most preferred class of inhibitors, amino compounds, e. g. decamethylene diamine, hexamethylene diamine, ethylene diamine, hexamethylene tetramine, 1, 3-diaminopropanol-2, dibutylamine, and triethanolamine are effective as modifiers for concentrated sulfuric acid, i. e. sulfuric acid of at least 80% concentration.

Low molecular weight alcohols soluble in concentrated sulfuric acid have also been found to be effective inhibitors for use with sulfuric acid of at least 80% concentration, including glycerol, methanol, ethanol, butanol, ethylene glycol, isopropanol and 1, 3-diaminopropanol-2.

Similarly, ethers such as diethyl ether and diethylene glycol have been found useful in inhibiting the tendering of cellulosic materials by sulfuric acid.

Acetone, methyl ethyl ketone and dipropyl ketone are effective in the order given, acetone being the preferred ketone.

In general the inhibitory effect decreases with increased molecular weight in a given series. The low molecular weight amides are more effective and are also more soluble, particularly the amides and amido derivatives of monocarboxylic acids.

Materials which give viscous solutions are less desirable than those which give limpid solutions, presumably because of the difference of ease of washing off the acid and also because of the slow wetting of the fabrics by these solutions.

While acids of concentration of approximately 80% and higher show the advantages of the use of amido derivatives in the most noteworthy manner, the use of amides is also advantageous with acids heretofore used as parchmentizing acids, for example, acids as low as 65% H₂SO₄ as shown in the following example. At the other end of the concentration range, acids of 100% strength and over, for example, 103% (thus containing free SO₃) may be employed with good results.

Example 21

A 68/76 unfinished cotton voile fabric was treated for 20 seconds in a solution of 40 gms. urea in 800 gms. of 70% sulfuric acid. The fabric was then washed and dried by ironing. This treatment resulted in a stiff springy finished but slightly harsh for an organdy.

Example 22

A 68/76 unfinished cotton voile fabric was treated for 20 seconds in a solution of 50 gms. urea in 800 gms. of 70% acid. After washing and ironing, the fabric possessed a springy organdy finish. The fabric was not noticeably tendered.

It is at the higher ranges of concentration,

for example, at 85% and above that the advantages of this invention are most striking. This is all the more surprising since materials such as formaldehyde which have hithertofore been recommended for use with acids of moderate concentration are ineffective with the concentrated acid.

Voil fabrics were treated for 20 seconds each in acids of the following compositions and tensile strengths determined with the results noted. The tensile strength shown is the force in pounds necessary to break a one inch strip of the cloth.

	Pounds
1. Untreated cloth	7.5
2. Cloth treated with solution of 20 gms. urea in 100 gms. 96% sulfuric acid	6.7
3. Cloth treated with solution of 20 gms. urea in 110 gms. of 87% sulfuric acid	9.3
4. Cloth treated with solution of 30 gms. urea* in 105 gms. of 91% sulfuric acid	10.2
5. Cloth treated with 96% sulfuric acid	0.0

*This amount of urea was not completely soluble in the acid at room temperature and so was not all in solution.

In addition to use in the treatment of fabrics, solutions of these inhibitors in sulfuric acid may be used for parchmentizing paper and treating regenerated cellulosic films, and as regeneration baths in the spinning of viscose yarn, or for subsequent treatment of viscose yarn, for example, to secure higher tenacity.

The parchmentized effects obtained by the present invention are not necessarily superior to the effects obtained by prior art methods but these effects can be obtained more consistently and with less spoilage of goods than is obtained by prior art processes. Thus in one of the recognized prior art processes for producing organdy finishes, there is first a mercerization in 20-25 per cent sodium hydroxide, followed by a treatment at a low temperature and at an inconveniently high speed, with sulfuric acid of about 66% concentration. The high speed is necessary to avoid ruining the fabric. Following the acid treatment the cloth is given another mercerization. On the other hand the process of this invention involves in its simplest form only a single acid treatment carried out at room temperature, in order to produce a satisfactory organdy finish. Furthermore the process of the present invention allows of a very great variation in the effects obtained, since the various factors such as time and temperature of treatment, and agents used can be varied at will over rather wide limits according to the effects desired.

The above description and examples are intended to be illustrative only. Only modification of or variation therefrom which conforms to the spirit of the invention is intended to be included within the scope of the claims.

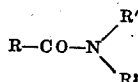
I claim:

1. Process which comprises immersing a cotton fabric at 20° C. for 30 seconds in a solution of 20 g. urea in 100 g. of 96% sulfuric acid, removing from said solution, washing with water and then with 3% ammonia solution and drying by ironing.

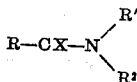
2. Process which comprises treating cotton fabric with sulfuric acid of at least 85% concentration containing 5-35 parts by weight of urea per 100 parts of H₂SO₄.

3. Process which comprises treating cotton fabric with concentrated sulfuric acid of at least 65% strength containing urea.

4. Process which comprises treating cellulosic textile materials with sulfuric acid of at least 65% strength containing urea.
5. Process which comprises treating cellulosic textile materials with sulfuric acid of at least 85% concentration containing in solution therein 5-35 parts by weight of urea per 100 parts of concentrated sulfuric acid.
6. Process which comprises treating cellulosic material with sulfuric acid of at least 65% concentration containing dissolved therein a low molecular weight amide.
7. Process which comprises treating cellulosic textile material with a solution in sulfuric acid of at least 65% strength of an amide of the formula



- wherein R, R' and R² are hydrogen or organic radicals containing up to six carbon atoms, the nitrogen content of said amide being at least 10%.
8. Process which comprises treating a cellulosic fabric with a parchmmentizing reagent until a stiff, springy, at least partly transparentized finish is obtained, thereupon discontinuing the treatment, and promptly washing out the reagent, said parchmmentizing reagent comprising sulfuric acid of at least 80% concentration having dissolved therein per 100 parts acid from 5-35 parts of a cellulose degradation inhibitor, said inhibitor comprising a low molecular weight amide which is soluble in 96% sulfuric acid at 20° C. to the amount of at least 20 parts per 100 of acid and which is further characterized by the fact that it reacts positively in the tendering resistance test, said test comprising, the step of dissolving 20 parts inhibitor in 100 parts 96% sulfuric acid, bringing the solution to 20° C., immersing therein a sample of cotton voile for a period of 30 seconds, thereupon removing the sample from the bath, and promptly washing out the acid and drying, a positive test being indicated by the fact that the sample is found to be at least partly parchmmentized and to have lost not more than 25% of its original tensile strength.
9. As a treating agent for cellulosic materials, a concentrated sulfuric acid solution of urea.
10. As a treating agent for cellulosic materials, a solution, in sulfuric acid of at least 65% concentration, of urea.
11. As a treating agent for cellulosic materials a solution, in sulfuric acid of at least 85% concentration, of an amido compound of the formula



wherein R is hydrogen, an amino group or an alkyl group, R' and R² are hydrogen or hydrocarbon radicals and X is oxygen sulfur or an imino group.

12. Process which comprises treating a cellulosic fabric with a parchmmentizing reagent until a stiff, springy, at least partly transparentized finish is obtained, thereupon discontinuing the treatment, and promptly washing out the reagent, said parchmmentizing reagent comprising sulfuric acid of at least 80% concentration having dissolved therein per 100 parts acid from 5-35 parts of a cellulose degradation inhibitor, said inhibitor comprising a low molecular weight amide which is soluble in 96% sulfuric acid at 20° C. to the amount of at least 20 parts per 100 of acid.

13. Process which comprises treating cellulosic textile materials with sulfuric acid of at least 65% concentration containing thiourea in solution therein.

14. Process which comprises treating cellulosic textile materials with sulfuric acid of at least 65% concentration containing in solution therein 5-35 parts of thiourea per 100 parts of concentrated sulfuric acid.

15. Process which comprises treating cellulosic textile materials with sulfuric acid of at least 65% concentration containing foramide in solution therein.

16. Process which comprises treating cellulosic textile materials with sulfuric acid of at least 65% concentration containing in solution therein 5-35 parts of foramide per 100 parts of concentrated sulfuric acid.

17. Process which comprises treating cellulosic textile materials with sulfuric acid of at least 65% concentration containing therein as a cellulose degradation inhibitor a low molecular weight amide soluble in 96% sulfuric acid at 20° C. to the extent of at least 20 parts per 100 parts of acid and having its amido group attached to a carbon atom doubly bonded to a non-metallic member of the sixth group of the periodic table having a molecular weight between 16 and 32.

18. Process for producing an organdy effect which comprises treating a thin cellulosic fabric with sulfuric acid of at least 65% concentration containing dissolved therein a low molecular weight amide.

19. Process for producing an organdy effect which comprises treating a thin cotton textile material with sulfuric acid of at least 85% concentration containing in solution therein 5-35 parts by weight of urea per 100 parts of concentrated sulfuric acid.

20. Process for producing an organdy effect which comprises treating a thin cotton textile material with concentrated sulfuric acid of at least 65% strength containing urea.

CERTIFICATE OF CORRECTION.

Patent No. 2,174,534.

October 3, 1939.

JOSEPH HARREL SHIPP.

It is hereby certified that error appears in the printed specification of the above numbered patent requiring correction as follows: Page 5, first column, line 65, for the word "finished" read finish; page 6, second column, lines 28 and 33, claims 15 and 16 respectively, for "foramide" read formamide; and that the said Letters Patent should be read with this correction therein that the same may conform to the record of the case in the Patent Office.

Signed and sealed this 12th day of March, A. D. 1940.

(Seal)

Henry Van Arsdale,
Acting Commissioner of Patents.