WOOL CHLORINATION PROCESS

No Drawing. Application November 6, 1953, Serial No. 390,740

9 Claims. (Cl. 8—128)

The present invention relates to processes of chlorinating wool. This application is a continuation-in-part of our co-pending application, Serial No. 27,364, filed May 15, 1946, for Wool Chlorination and Process, now abandoned.

A further purpose is to maintain the pH range by buffering, preferably by alkaline sodium borate.

A further purpose is to effect the absorption of an acid compound on the wool, to regulate such acidity to a desired amount, and then to bring the wool into contact with an alkaline bath containing a combined available chlorine, preferably as calcium or sodium hypochlorite, in which gradual and controlled chlorination will take place as the hypochlorite encounters the acidity in the wool.

For many years it has been the practice to chlorinate wool for the purpose of altering the structure either chemically or physically, of the fibers so as to reduce shrinkage caused by felting or to reduce the differential coefficient of friction of the fibers or the tendency of the fibers to interlock with one another.

Chlorination has no effect on shrinkage due to fiber and fabric relaxation. The essential effect is believed to be one of altering the surface characteristics of the scale structure of the fiber, so that the fibers exhibit a reduced differential coefficient of friction. Chlorinating of wool has also been applied for other purposes, although the primary purpose has usually been to reduce shrinkage.

In the prior art practice of chlorinating wool, several difficulties have been encountered which have interfered with the success of the operation or made the process troublesome. One of the primary prior art troubles has been through yellowing of the wool, and one of the important advantages of the present process is that yellowing is minimized or wholly eliminated.

In the prior art practice in some cases the scale destruction has been so extensive that the wool has been structurally impaired, so that the fibers have been actually weakened and embrittled to such an extent that the resilience is reduced. The present process permits effective chlorination without any such undesirable effect on the fibers.

In many prior practices the wool has been seriously damaged, as evidenced by marked increase in alkali solubility, whereas in accordance with the present invention no appreciable damage is caused and only a slight increase in alkali solubility results from the chlorination.

In order to overcome some of these difficulties, prior art practices have in many cases been modified so as to protect the fiber, and such modifications have prevented effective elimination of felting. By the present invention the shrinkage of fine wool can be reduced to below five per cent, and the shrinkage of coarse wool can be reduced to below two percent, reliably and uniformly.

In the prior art chlorination, much difficulty has been caused by the hardness produced in the hand of the wool.

The process of the present invention has no undesirable effect on the softness and natural feel of the fiber.

Great difficulty has been encountered in the prior art in obtaining uniform results. The present invention greatly increases the uniformity, to such an extent in fact that blends of coarse and fine wool may be processed together with highly satisfactory results. The present process, furthermore, can be applied uniformly to blends of wool with other fibers, such as cotton, rayon, nylon and the like, such other fibers going through the process unchanged and without harm.

An important feature of the present invention is that the various types of wool, including the extremely coarse and extremely fine wool fibers, absorb acid to approximately the same extent, and such absorbed acid can be used to control the chlorination in an alkali bath.

Further purposes appear in the specification and in the claims.

When reference is made herein to wool, it is intended to refer to animal textile fibers of the character of wool, whether sold as wool, lama, alpaca, animal hair or the like.

Not only is the shrink-resisting treatment of the present invention uniform and controllable, but the results obtained are reproducible. Furthermore, the treatment of the present invention, unlike some of those employed in the prior art, withstands laundering.

One difficulty with the prior art chlorinating techniques has been that in many cases if applied to undyed wool they affect the evenness of dyeing, and if applied to dyed...
wool they cause marked changes in color. The process of the present invention has little or no effect on evenness of dyeing if applied to undyed wool, and can be applied on properly selected dyestuffs if applied to dyed wool.

From a commercial standpoint, the process of the invention is desirable because it operates at relatively high speed, and makes efficient use of chloride.

In carrying out the process, it is immaterial whether the wool is wholly acid or alkaline or a mixture of the two.

The chlorination may be applied in the yarn, raw stock, sliver, roving, top, fiber or fabric. The process may be applied preferably before scouring, but it may also be applied prior to scouring if desired. Likewise, the wool may be dyed before or after chlorination, although of course, if the dyeing is accomplished before chlorination, the dye must be fast to chlorine. To obtain good whites, the chlorination should preferably be applied before peroxide bleaching.

The process of the invention can be applied after carbonizing, in which case a smaller amount of acid in the wool can be employed to control the chlorinating process. Likewise the process of the invention can be applied batchwise or continuously as desired.

All percentages herein are based on the total dry weight of the goods being treated, unless otherwise indicated.

The goods or material being treated are first treated with acid to cause the wool to come into the requisite condition to control the chlorination. The acid may be applied by spraying or otherwise, but the goods will preferably be dipped or soaked in the acid.

The acid content may be adjusted by partial neutralization, so as to bring the quantity of acid to the desired level.

If the pure acid is a liquid, it may be applied as such liquid, or it may be applied in a water solution.

The acid content retained in the wool when it enters the chlorinating bath should be at least 0.3 (preferably 0.4) milliequivalent per gram of clean air dried wool fiber.

For a given process it is best not to have the acid content in excess of 0.7 (preferably not in excess of 0.6) milliequivalent per gram as aforesaid. This is particularly suitable for time ranges of 1 to 10 minutes, preferably 1 to 5 minutes at a pH of 7.2 to 9 as later explained. Particularly for continuous operation and particularly for short times (2 seconds to one minute and especially less than 20 seconds) the acid in the wool may be built up to much higher levels, as high as 2 milliequivalents per gram as aforesaid. Also acid contents as low as 0.3 milliequivalent per gram may be used for such short times if desired, and for best results the acid retained should exceed 0.7 milliequivalent per gram. It is not desirable to use acid contents in excess of 2 milliequivalents per gram as aforesaid or even with the shortest duration (down to a minimum of two seconds).

It will be understood that the upper limit of acid content is set by the desire to avoid damage to the wool, and that if the temperature is kept down below 100° F., preferably at sub-atmospheric temperatures, large quantities of acid can be tolerated for short times.

In any case it will be evident that the acid content in the wool is sufficient so that it is not fully neutralized by the alkaline chlorinating bath and the wool still contains acid unneutralized at the end of the chlorination.

The preferred acid for use for acidulating is sulphuric acid in the form of sodium bisulphate. Other acids may be used. If sodium bisulphite is not desired, potassium bisulphite, or other alkali metal bisulphite, sulphuric acid or a mixture of sulphuric and oxalic acids, sulphurous and sulphonic acids or the like may be used. Instead of the above, oxalic, hydrochloric, phosphoric, formic, propionic, glacial acetic, chloroacetic, trichloroacetic, or other acid may be used. The desired acidity may be employed for the purpose of the present invention phosphoric acid acts as a monobasic acid.

A wetting agent, such as an alkyl aryl sulphonate may desirably be employed to accelerate the treatment of the wool with acid, but the process can readily be carried out on without such wetting agent. Where the wetting agent is used, it may conveniently be employed in a proportion of about 1% of the weight of the acid; thus if sodium bisulphate is used, the composition may be 98% sodium bisulphate and 2% alkyl aryl sulphonate (these percentages are of the dry composition).

The temperature of the acid solution can vary, the preferred temperature being about 130° F., although lower temperatures down to or below room temperature and higher temperatures for example up to the boiling temperature of water can be used.

Of course it will be understood that where the wool is being carbonized and carbonizing acid is in the wool, much higher temperatures will be used for short times in connection with the baking in carbonizing, and the acid content in the wool will often be of the order of 4 to 6% of residual sulphuric acid, or in general 2 to 10 percent of acid.

The effect of the treatment of the wool with the acid as just noted causes the wool to take up a large quantity of acid and if the acid quantity is excessive for the control of purposes desired the acid can be partially neutralized before chlorination. Also, of course, acid content should be removed from the wool by squeezing or draining before chlorinating. For the short time process particularly, operating continuously at times less than one minute, the water content in the wool leaving the squeeze rolls from the acid treatment at the beginning of chlorination should not exceed 100 percent on the weight of the wool.

The partial neutralization of the wool with alkali before chlorination, while retaining at least 0.5 milliequivalents of the wool acid in the wool, particularly in the batch process as giving increased uniformity and reliability by eliminating the delay and uncertainty due to presence on the wool of acid which is not combined with the fiber but mixed with the fiber and free in the wool and which otherwise is likely to react with and waste hypochlorite.

The alkali which is used in case of partial neutralization of the wool before it enters the chlorinating bath will preferably be sodium metaphosphate, but other alkalis may be used, such as borax, potassium metaphosphate or other alkali borates (preferably of alkali metals), sodium carbonate, potassium carbonate or the like.

Considering now the chlorination bath in detail, this bath will be alkaline at the beginning of chlorination and will remain alkaline throughout the process, this standing that the wool is acid even at the end of chlorination. The pH of the chlorinating bath will at all times be in excess of 7.2. It is not desirable to use a pH in the chlorinating bath in excess of 11.5. In the batch process the pH in the chlorinating bath is very desirably kept below 9, as the time of chlorination is preferably 1 to 10 minutes and most desirably 1 to 5 minutes, and in this time some yellowing may occur if the pH of the chlorinating bath is in excess of 9. In the case of continuous operation, with times not in excess of one minute and preferably between 2 and 20 seconds the pH of the chlorinating bath will have a pH of 7.2 to 11.5 and preferably 8 to 11.5.

While the pH may drop during chlorination, it will not drop below about 7.5 and certainly not below 6.0.

The pH in the chlorinating bath should be kept within the desirable range in any suitable manner. An automatic pH control and feeder may be used to add alkali as required to maintain the limits. The limitation may also be maintained by buffering, using any suitable buffer, preferably sodium metaborate or other alkali metal metaborate. Where a buffer is used in the batch process it will preferably hold the pH between 7.2 and 9.

The temperature of the chlorinating water bath will be maintained between 32 and 100° F., and preferably between 70 and 80° F. The percentage of available chlorine should be between 0.075 and 2% of the weight of the solution.

In the batch process 0.075 to 0.4% available chlorine on the weight of the solution is preferably used and preferably the quantity of the desired 0.5 to 1.5% of available chlorine on the weight of the solution is preferably used. Higher chlorine content may be used for finer wool.

The chlorinating solution consists of a hypochlorite. In the batch process calcium hypochlorite which should contain at least 70% available chlorine on the weight of the hypochlorite is preferably used. For the continuous process sodium hypochlorite or potassium hypochlorite are preferable.

Where the batch process is being used, the preferred treatment to make up a partial neutralizing solution such as sodium metaborate dissolved in warm water and run the goods in it for at least 30 minutes then add a
stabilizing solution of sodium metaborate in warm water and agitate the goods in it at least once on the turntable and finally add the solution of chlorinating agent with additional sodium metaborate and run the goods for the short period of one minute, which should not exceed ten minutes and preferably be one to five minutes. The actual chlorination is completed in about two minutes, using acid contents of 0.3 to 0.7 milliequivalent per gram of air dried clean wool fiber and preferably 0.4 to 0.6 milliequivalent per gram and using pH ranges of 7.2 to 9.

The time may be as short as 2 seconds, especially with acid contents of 0.7 to 2.0 milliequivalents per gram of air dry clean wool fiber in the continuous process and impermissibly with pH ranges of 7.2 to 11.5, preferably 8 to 11.5. For best results the continuous process will be run from 2 seconds to 20 seconds. Chlorinating beyond ten minutes is undesirable due to yellowing. The chlorination should be stopped at the end of the time. If the chlorinating agent is used up, it will be sufficient to run the goods through squeeze rolls in the continuous process, but in most cases the chlorination will be stopped by using an antichlor such as sodium bisulphite, although any other suitable antichlor can be used.

The goods at the end of chlorination and before adding the antichlor are slightly acidic and the acidity in the wool is never fully neutralized in the process. Thus the process is characterized by chlorination of wool containing retained acid in an alkaline bath, thus avoiding the extremely rapid chlorine release and greater wool damage due to chlorinating in an acid bath. The control of the bath process is the chlorination of the wool, which is regulated to the desired level before chlorination. As long as the chlorinating bath has a pH within the desired range, the time the pH can vary somewhat in that range without affecting the result providing the acidity in the wool is the same in each case. Control can be secured by the acidity in the wool without regard to the exact pH of the chlorinating bath within the range.

Thus the process produces a rapid chlorination effect and the chlorate being released as the hypochlorite reacts with the wool. As soon as some chlorination has occurred in the wool fiber, especially in the batch process, the potential for further chlorination at that point is lowered by reduction in the acid contained in the fiber at the point where chlorination has occurred.

If the goods are to be subsequently dyed or bleached they should be neutralized and secured according to any accepted procedure. The satisfactory method is to run for twenty minutes in a bath at 120 °F, containing a synthetic detergent (fatty alcohol) and 2% by weight of acid. This treatment will leave the goods clean and slightly alkaline which is the minimum condition for starting to dye chlorinated wool or for bleaching.

The neutralizing and securing bath should be dropped and followed by one warm rinse before dyeing or bleaching.

In some cases it is necessary (in grain knit hosiery or piece goods) and in other cases it is preferable (on light blues, pinks, and other like shades where levelness is a problem even on untreated wool) to chlorinate after dyeing. This can be done provided the dyes are fast to chlorine. No special precautions need be taken except for thorough rinsing after dyeing.

The equipment used for the process should be stainless steel, Monel metal, wood, rubber, plastic or a similar material not to chlorine. No particular change in the process is required with variations in the type of equipment. The amounts of the chemicals used and the preferred temperatures remain unchanged. In some machines however, the time of processing should be changed for best results and other precautions should be taken to assure maximum uniformity.

**EXAMPLE I**

The following is a typical example of chlorination in a batch process: One hundred pounds of goods containing 90% wool are to be chlorinated, to be subsequently dyed and bleached. For the proper handling of the goods in the equipment used 240 U.S. gallons or 2,000 pounds. Dividing the volume of the tank by the weight of the goods, it is determined that the bath ratio is 20. Since the goods are to be subsequently dyed but not bleached, the tables used as set forth immediately after these examples are Table 1-D to Table 4-D.

In Table 1-D, the left hand column is headed "Bath ratio," and reading down to "20" you then read horizontally to the column headed "90% wool in goods." The quantity required of sodium bisulfate (98%) plus alkali aryl sulfonate (2%) for 20% of the dry weight of the goods. The quantity of sodium metaborate for partial neutralization for all bath ratios is shown by Table 2-D for 20% wool in the goods is 3% of sodium metaborate and Table 3-D for the calcium hypochlorite, and Table 4-D for the sodium bisulfite read similar to Table 1-D, indicating that 4% of calcium hypochlorite and 2% sodium bisulfite solubility.

With sulphuric acid (including sodium bisulfate) the pH may vary from 1 to 4 in the acidulating bath, although a pH from 3 to 4 is not preferable because of the length of treatment time required. A pH below 1 is not recommended because of the tremendous quantity of acid required, and the likelihood of undesirable effects on the later processing.

The quantity of acid will, of course, vary with the particular acid. Where sodium acid sulphate is used, the quantity of this compound plus 2% wetting agent is given in Table 1-D for goods subsequently to be dyed or left natural, and in Table 1-B for goods which are subsequently to be peroxide bleached. The quantity of acid may vary widely, and even when it is desired to keep close to the conditions specified in the uniformity of results, the acid plus wetting agent may vary either way by 20% of such percentage (provided the acid retention is kept within the limits set).

A bath is prepared at 130 °F, the acid (plus wetting agent, if used), previously dissolved and diluted is added to the bath, the goods entered and run or agitated for about 20 minutes. The bath is then 95% off and the goods are drained well. The goods should not be rinsed at this point.

The excess of acidity is preferably next neutralized in a cold water bath (about 70 °F, 90 ° F - 95 ° F), adding sodium metaborate (previously dissolved), in the amount specified in Table 2-D for goods subsequently to be dyed or left natural, and in Table 2-B for goods subsequently to be peroxide bleached. The machine is then run for approximately thirty minutes. By the end of this treatment a sufficient degree of equilibrium has been attained between the acid in the wool and the added sodium metaborate to give the desired degree of acidity.

Next preferably 1% of buffer (preferably sodium metaborate) is added (in water solution) to the first alkali bath, and the goods run for one to five minutes.

Next, the chlorination solution (prepared as described below) is added to this bath and goods run agitated for one to five minutes.

Next, the antichlor, sodium bisulphite (previously dissolved in warm water), is added to this bath, the amount being used as indicated by Table 4-D or Table 4-B (depending on the subsequent processing of the goods). The antichlor should be agitated or run with the goods for about ten minutes, after which the entire bath is dropped and two warm rinses preferably at 90° to 100° F. are applied.

The preferred manner of making up the chlorinating solution is to dissolve in warm water two percent of sodium metaborate buffer on the dry weight of the goods, and separately dissolve in warm water the amount of calcium hypochlorite indicated by Table 3-D or Table 4-B depending on whether the goods are to be dyed or left natural on the one hand and to be peroxide bleached. The calcium hypochlorite is preferably stirred slowly into ten to twenty times its own weight of water with constant stirring. The calcium hypochlorite does not entirely dissolve, but the solution becomes turbid. The sodium metaborate and calcium hypochlorite solutions are then combined by pouring the former into the latter while stirring.

It is desirable to take special precautions on goods which are subsequently to be peroxide bleached. Peroxide bleaching imparts a certain amount of shrink resistance to the fiber, and therefore it is desirable to give a milder treatment than would be the case if the goods were subsequently to be dyed or which is to receive no further treatment. Accordingly when the wool is subsequently to be peroxide bleached, Table 1-B to Table 4-B should be followed, which specify somewhat higher
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concentration of alkali in the pretreatment bath immediately prior to chlorination, and a somewhat lower content of hypochlorite in the chlorinating bath, as well as a somewhat lower content of anticlor.

No special change in the bleaching procedure need be made.

By referring to the tables, it will be seen that on the weight of the goods the sodium bisulphite varies from 7.8 to 10% for the acid bath, for the neutralization and buffering, the sodium metaborate varies from 4¹/₂ to 10%, the calcium hypochlorite varies from 1¾ to 4½% while in the anticlor the sodium bisulphite varies from 2% to 4½% and the calcium metaborate, from 0% to 2%. Where the goods are subsequently to be dyed or left natural, the sodium bisulphite in the acid bath varies from 7% to 34.4% and calcium hypochlorite varies from 1% to 7½%, the calcium hypochlorite varies from 2% to 4½%.

Where the goods are subsequently to be bleached, the sodium bisulphite varies from 7.8% to 34.4% in the acid bath, in the neutralizing and chlorinating bath the total sodium metaborate varies from 0% to 1½% and the calcium hypochlorite varies from 1% to 2½%, while the anticlor varies from 1% to 2%.

EXAMPLE II

In the batch process, the entire procedure may be carried out in a single bath, rather than using two separate baths as in Example I. Ten percent of the acid composition (8% of sodium bisulphite and 2% of wetting agent) is made up in a bath using a 20 to 1 bath ratio. The goods are run in this bath at 100°F. for forty-five minutes. Then 2% of sodium metaborate is added and the bath run for five minutes. Following this the chlorination is run on as in Example I employing 5% calcium hypochlorite and 2% of sodium metaborate, and following with the anticlor as in Example I. It will be understood that in this one bath method the proportions may be varied as indicated in the tables for different goods compositions, provided recognition is given to the fact that the acid is not being drained from the goods or removed from the bath but must be neutralized prior to the chlorination.

EXAMPLE III

The procedure of Example I is followed out except that the acidulation is accomplished in a bath containing 17% oxalic acid, based on the weight of goods being treated.

EXAMPLE IV

The procedure for continuous chlorinating according to the invention, was applied to cloth in the piece consisting of a wool-nylon shirting woven from yarn made from single ply 45% fleece and/or old wool, not lower in grade than 62's U. S. standard, from 10 to 15% first quality staple nylon, of about 1½ inches staple length, and not more than 40% of wool wools not lower in grade than 62's U. S. standard. The cloth was carbonized in the usual manner, running it through a sulphuric acid bath at 2° Baume maintained at 70° to 80°F., and was then carried through a usual carbonizing oven, having a temperature of 180° F. in the first chamber, 200° F. in the second and third chambers, and 260° F. in the fourth and fifth chambers. The running time in the carbonizing bath, was two minutes, and the running time in the baking oven was 2½ minutes per chamber.

After carbonizing, the acid content in the wool was 1.225 milliequivalents per gram of air-dried clean wool fiber. Good results were obtained even when the wool containing the acid was held for a couple of days before chlorinating.

The cloth containing the above acid content, was run into a chlorinating bath which had a pH of 10.0 maintained plus or minus 0.2 by metered additions of sodium hydroxide. The chlorinating bath contained 0.5% available chlorine. The chlorinating bath was maintained at a temperature of 75° to 80°F. by cooling coils under the control of a temperature controller.

The chlorinating was carried out in a continuous padding machine with a time exposure of the cloth to the chlorinating bath between eight and ten seconds. The chlorinating consumption was 0.027 pound per pound of fabric. After chlorinating, the cloth was immediately run through squeeze rolls, and then immersed in a cold running water rinse, and then run to a continuous washer chamber where it was neutralized by a 1% solution of sodium bicarbonate in water. The washing time was eight to ten minutes, and the neutralizing time following the washing was eight to ten minutes.

It was found that there was no residual chlorine content, and no anticlor was used in this particular instance, though an anticlor was used in other experiments.

After neutralizing, the neutralizing chemicals were washed out and a softener was applied.

**Tables 1-D to 4-D**

<table>
<thead>
<tr>
<th>PERCENT OF WOOL IN GOODS</th>
</tr>
</thead>
<tbody>
<tr>
<td>Bath Ratio</td>
</tr>
</tbody>
</table>
| **TABLE 1-D**
| Percentage of sodium bisulphate (98%) plus alkyl aryl sulphophthalein (2%) |
| --- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| 8 | 15 | 11 | 10 | 9 | 8 | 7 | 6 | 5 | 4 |
| 12 | 12 | 10 | 9 | 8 | 7 | 6 | 5 | 4 | 3 |
| 16 | 20 | 17 | 15 | 13 | 11 | 9 | 8 | 7 | 6 |
| 20 | 24 | 20 | 16 | 13 | 11 | 9 | 7 | 6 | 5 |
| 24 | 28 | 24 | 20 | 16 | 13 | 11 | 9 | 7 | 6 |
| 28 | 32 | 28 | 24 | 20 | 16 | 13 | 11 | 9 | 7 |
| **TABLE 2-D**
| Percentage of sodium metaborate for partial neutralization |
| --- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| 4 | 35 | 35 | 33 | 31 | 29 | 27 | 25 | 23 | 21 |
| **TABLE 3-D**
| Percentage of calcium hypochlorite |
| --- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| 8 | 35 | 35 | 33 | 31 | 29 | 27 | 25 | 23 | 21 |
| 12 | 45 | 45 | 43 | 41 | 39 | 37 | 35 | 33 | 31 |
| 16 | 55 | 55 | 53 | 51 | 49 | 47 | 45 | 43 | 41 |
| 20 | 65 | 65 | 63 | 61 | 59 | 57 | 55 | 53 | 51 |
| 24 | 75 | 75 | 73 | 71 | 69 | 67 | 65 | 63 | 61 |
| **TABLE 4-D**
| Percentage of sodium bisulphite |
| --- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| 8 | 2 | 2 | 2 | 2 | 2 | 2 | 2 | 2 | 2 |
| 12 | 3 | 3 | 2 | 2 | 2 | 2 | 2 | 2 | 2 |
| 16 | 4 | 4 | 3 | 3 | 2 | 2 | 2 | 2 | 2 |
| 20 | 5 | 5 | 4 | 4 | 3 | 3 | 2 | 2 | 2 |
| 24 | 6 | 6 | 5 | 5 | 4 | 4 | 3 | 3 | 2 |
| 28 | 7 | 7 | 6 | 6 | 5 | 5 | 4 | 4 | 3 |
| **TABLE 1-B**
| PERCENT WOOL IN GOODS |
| Bath Ratio | 100 | 90 | 80 | 70 | 60 | 50 | 40 | 30 | 20 |
| Percentage of sodium bisulphate (98%) plus alkyl aryl sulphophthalein (2%) |
| --- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| 8 | 15 | 11 | 10 | 9 | 8 | 7 | 6 | 5 | 4 |
| 12 | 12 | 10 | 9 | 8 | 7 | 6 | 5 | 4 | 3 |
| 16 | 20 | 17 | 15 | 13 | 11 | 9 | 8 | 7 | 6 |
| 20 | 24 | 20 | 16 | 13 | 11 | 9 | 7 | 6 | 5 |
| 24 | 28 | 24 | 20 | 16 | 13 | 11 | 9 | 7 | 6 |
| 28 | 32 | 28 | 24 | 20 | 16 | 13 | 11 | 9 | 7 |
The neutralizing bath was maintained at a temperature from 80° to 90° F. and consisted of a 1% sodium bicarbonate solution by weight. The cloth was retained in the neutralizing bath for two minutes in each case. The cloth was then rinsed three times for a total of fifteen minutes, to remove all soluble chemicals.

Following the chlorination, the cloth was given four, thirty minute washes with 0.5% soap at 140° F. followed by three, ten minute rinses at 140° F. in a Najor washer and the felting shrinkage was measured for both the warp and the filling. The average value in each instance for four measurements, is given in Table 5. In some cases, the dimensions of the cloth actually increased, as indicated by the negative values. The increase in alkali solubility was also determined with respect to controls. It will be evident that the felting shrinkage values and the alkali solubilities are within satisfactory commercial limits.

**TABLE 2-B**

<table>
<thead>
<tr>
<th>Percentage of sodium metaborate for partial neutralization</th>
<th>7</th>
<th>6%</th>
<th>5%</th>
<th>4%</th>
<th>3%</th>
<th>2.5%</th>
<th>2%</th>
<th>1.5%</th>
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<td>12</td>
<td>15</td>
<td>20</td>
<td>24</td>
<td>30</td>
<td>35</td>
<td>40</td>
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</table>

**TABLE 3-B**

<table>
<thead>
<tr>
<th>Percentage of sodium metaborate</th>
<th>8</th>
<th>12</th>
<th>15</th>
<th>20</th>
<th>24</th>
<th>30</th>
<th>35</th>
<th>40</th>
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</thead>
<tbody>
<tr>
<td>All</td>
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<td>20</td>
<td>24</td>
<td>30</td>
<td>35</td>
<td>40</td>
</tr>
</tbody>
</table>

**EXAMPLE VI**

Tests were conducted on wool-rayon fabrics, of which the following is typical. An 8 ounce fabric (8 ounces per linear yard, 57 inches wide) woven from yarns containing 60% wool fibers and 40% viscose rayon fibers, was passed into a solution of 6% by weight of formic acid in water, and then through squeeze rolls to a pick up of 6% by weight of formic acid in the cloth. The acidulated cloth was then passed wet into a chlorinating bath containing hypochlorite, having a concentration of available chlorine of 0.5% for a time of from eight to ten seconds. The chlorinating bath had a pH of 9 maintained during the chlorination. The temperature of the chlorinating bath was maintained at from 70° to 80° F. The material was passed through squeeze rolls from the chlorinating bath, and then into a neutralizing bath containing 1% sodium bicarbonate in water at 80° to 90° F. Following the neutralizing bath, the material was scoured and finished. The alkali solubility was increased about 10% with the procedure, and the felting shrinkage after four standard washes, was 3% in the warp, and less than 2% in the filling.

It will be understood that for other acids, alkaline and chlorinating agents, the percentages will be varied to produce the particular acidities and chlorine availability values desired.

The present invention has been successfully applied to the chlorinating of a wide variety of wools under varying conditions of blends and mixtures with other fibers, and has been found to impart uniform chlorination and reliable non-felting properties, without harm to the physical properties, such as structure and resilience of the fibers, and without appreciable yellowing or other undesirable effect.

It is possible to employ the same chlorinating bath or a chlorinating bath of the same composition to produce different degrees of chlorination by changing the acidity of the wool within the range specified, the extent of the chlorination increasing with the acidity and reducing with reduction of the acidity. It will be understood that such control of the chlorination is possible notwithstanding wide variation in the pH in the chlorinating bath within the limits set forth.

In view of our invention and disclosure variation and modifications to meet individual whim or particular need will doubtless become evident to others skilled in the art, to obtain all or part of the benefits of our invention without copying the process and composition shown, and we, therefore, claim all such inventions they fall within the reasonable spirit and scope of our claims.

Having thus described our invention what we claim as new and desire to secure by Letters Patent is:

1. The process of chlorinating wool, which comprises treating the wool with acid in solution, retaining in the wool acid amounting to 0.3 to 2 milliequivalents per gram of air-dried clean wool and treating the wool with a chlorinating agent in water solution while maintaining a pH range in the solution of 7.2 to 11.5, for a time of 2 seconds to 10 minutes, at a temperature of 32 to 100° F. acidity in the wool being retained throughout the chlorination.

2. The process of claim 1, wherein the acid retained in the wool is sulphuric acid.

3. The process of chlorinating wool in a batch process, which comprises treating the wool with acid in solution,
retaining in the wool acid amounting to 0.3 to 0.7 milliequivalent per gram of air-dried clean wool fiber, and treating the wool with a chlorinating agent in water solution, while maintaining a pH range in the solution of 7.2 to 9, for a time of 1 to 10 minutes, at a temperature of 32 to 100 °F., acidity in the wool being retained throughout the chlorination.

4. The process of claim 3, wherein the acid retained in the wool is from 0.4 to 0.6 milliequivalent.

5. The process of claim 3, wherein the chlorinating agent is buffered by alkaline sodium borate.

6. The process of claim 3, wherein the acid is from 7.8 to 34.4 percent of sodium bisulphate on the weight of the goods in water solution, and the chlorinating solution contains from 4.5 to 10 percent on the weight of the goods of sodium metaborate and from 1.75 to 6.5 percent on the weight of the goods of calcium hypochlorite having an excess of 70 percent of available chlorine calculated on the weight of calcium hypochlorite.

7. The process of claim 3, wherein the amount of acid retained in the wool is not in excess of 0.6 milliequivalent and the chlorinating solution contains from 1.75 to 6.5 percent of calcium hypochlorite on the weight of the goods having in excess of 70 percent of available chlorine on the weight of the calcium hypochlorite.

8. The process of chlorinating wool in a continuous process, which comprises treating the wool with acid in solution, retaining in the wool acid amounting to 0.3 to 2 milliequivalents per gram of air-dried clean wool fiber, and treating the wool with a chlorinating agent in water solution containing from 0.2 to 2% available chlorine on the weight of the solution, while maintaining a pH range in the solution of 7.2 to 11.5, for a time of 2 seconds to 1 minute, at a temperature of 32 to 100 °F., acidity in the wool being retained throughout the chlorination.

9. The process of claim 8, wherein the amount of acid retained in the wool is from 0.7 to 2 milliequivalents and the pH during chlorination is maintained in the range between 8 and 11.5.

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