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## PROCESS FOR IMPARTING SHRINK RESISTANCE TO WOOL

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This invention relates to a process for the treatment of wool, and more particularly to a process to impart shrink resistance to wool of all types.

It is customary to clean woolen clothing in the form of suits and dresses by dry cleaning, and to wash woolen socks and underwear in lukewarm water with great care, and dry them on hangers and forms in order to avoid shrinkage and loss of shape. The reason for this is the well-known tendency of wool fibers to shrink and felt during washing. Great monetary losses are incurred by the public as a result of the loss of utility of shrunken woolen goods. Numerous attempts have been made to treat wool so as to overcome its tendency to shrink, but such processes have heretofore been characterized by difficulties in controlling them, strict control being necessary to avoid damage to the wool. This is true of treatments with acidic as well as alkaline baths.

It is customary in the prior art processes to immerse the fabric in an aqueous or non-aqueous bath containing a suitable reagent with the acidity or alkalinity of the bath adjusted to obtain, at ambient temperature, or below, the desired rate of reaction as an immediate result of the immersion.

The principal disadvantage of this method is the difficulty of obtaining level treatment even at the expense of slow reaction rates. Unlevel treatment results in streaked dyeing, tendering, poor wear and laundering life. Slow reaction rates, i. e., long exposure periods, although yielding a somewhat more level treatment, result in an undesirable penetration of the wool fiber.

A major object of this invention is control of the processes for imparting shrink resistance to wool, so as to obtain the minimum reduction of area of the woolen goods after laundering, together with a minimum of damage to the wool fibers.

Another object of this invention is a reduction of the period of treatment of the woolen goods.

Another object is the production of goods which are evenly treated with the shrink resistance imparting agent.

Still another object of our invention is a process for imparting shrink resistance to wool which does not impair the evenness of subsequent dyeing operations.

Yet another object of our invention is the shortening of the treatment, whereby treatment is limited to the surface of the wool fibers without substantially changing the cortex of the wool fiber.

And another object of this invention is a wool

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treatment process, preferably with hypophalites, which permits the use of alkaline baths with higher hydroxyl-ion concentration and acidic baths with higher hydrogen-ion concentration, without increasing damage to the wool, than has been possible heretofore.

Another object is a continuous process for imparting shrink resistance to wool and worsted goods, and to mixed fabrics composed partly of wool.

Other objects will appear in the following description.

We have discovered that even, level treatment can be achieved with very high rates of reaction if the reagent is applied evenly to goods in open form as a solution, slurry, or paste, in a separate step wherein the conditions are such that substantially no reaction ensues, followed by a second step wherein the thus impregnated material is exposed to conditions selected to bring about the desired rate of reaction.

More specifically, the reagent, in water or other solvent, or as a slurry, or in paste form, is applied continuously and evenly to the fabric with a padder or other suitable means, or by immersion in a bath of the desired composition. To avoid substantial reaction prior to the second step, either the reagent is so compounded as to be substantially inert at ambient temperature, or the time lapse between the application of the reagent to the fabric and the second or exposure step is held very small. The impregnated fabric is then passed immediately and continuously through a heater where it is exposed to a degree of heat of the order of 125° F. or more; particularly good results were obtained at about 200° F. The actual temperature selected for the second or heating step depends on the type of wool, the particular shrink-resistance imparting agent selected, and on the actual room temperature existing at the time the process is carried out; for instance if the room temperature is high in a hot climate, the temperature of the second or heating step should be sufficiently high to provide the necessary temperature gradient between the first and second step of the process. Heating is effected either with air or neutral or acidified steam in accordance with the type of reagent used. The speed and the partial or full completion of the reaction can be regulated by adjusting the degree of heat and the length of exposure of the wool thereto. Preferably the heating step should not substantially exceed 15 minutes, in order to protect the goods, and may be substantially shorter than 15

minutes provided its length exceeds the length of the first (contacting) step.

Our experience shows that the advantages of our continuous, step-wise process are obtained with the use of the following types of reagents well-known in the art: chlorine, bromine, sodium or calcium hypochlorite, sodium hypobromite, sodium chlorite, hypochlorous or hypobromous acid, potassium dichromate, potassium permanganate, chlorinated hydantoins, sulfuryl chloride and nitrosyl chloride. In fact, the experience we have had based on our discovery indicates that any of the reagents disclosed in the prior art of shrinkage control in woolen goods, except the enzyme type which would be rendered ineffective by high temperature, can be used in conformance with the instant disclosure.

The advantages of our continuous stepwise process are preferably obtained with the use of hypohalites. As will more fully appear from the following examples, available halogen concentrations of the order of from about 1% to about 6¾% in the treating solution are preferred. These hypohalite solutions are effective at the elevated temperatures contemplated by our invention, without injuring the fabric.

Due to the material shortening of the treating time, it is possible to employ treating baths having an available halogen concentration and an acidity or alkalinity of a magnitude that was heretofore considered noxious to the wool.

The available halogen concentration in the following examples has been computed by the method set forth in Rogers' Manual of Industrial Chemistry, 6th edition, 1942, vol. 1, page 451:

"It has become accepted practice to speak of the strength of hypochlorite preparations in terms of 'available chlorine,' by which is meant the $Cl_2$ equivalent of the (OCl) present. Hypochlorite chlorine is electropositive in character, and has a valence of plus one, in distinction to chloride chlorine, which is electronegative and has a valence of minus one, so it is a bivalent oxidizing agent. Hence, pure calcium hypochlorite ($Ca(OCl)_2$) having a molecular weight of 142.99, contains the equivalent of $2Cl_2$ molecules weighing 141.84, and would therefore contain 141.84/142.99=99.19 per cent 'available chlorine.'"

We are aware that wool has been made shrink resistant by coating with an artificial resin at high temperatures; such a process does not fall within the scope and spirit of our invention, because in that process no chemical reaction between the resin and the wool takes place.

The following are detailed examples of the application and results of continuous plural-step processes according to our invention:

*Example 1*

A 10½ ounce flannel shirting was passed through a solution at room temperature of 65 mls. of 15% commercial sodium hypochlorite per liter (available halogen concentration .975%) and 20 gms. alkyl sulphate wetting agent per liter into a padder. The rate of feed of the material was adjusted to provide an elapsed time of five seconds between wetting and entry into the padder whose rolls were adjusted to provide a solution retention of 100 weight percent of the fabric. Thence the material was passed through an oven maintained at 200° F. The course of the material through the oven was arranged to provide complete dryness upon exit at the selected rate of feed. The shrinkage effect of laundering on the thus treated, finished material had been reduced from 25% to 12% area basis.



*Example 2*

An all-wool knit material was passed through a solution at room temperature of 50 gms. sodium hypobromite (mol. wt. 118.92) per liter (available halogen concentration 6.73%) and 20 gms. alkyl sulphate wetting agent per liter to a padder in five seconds. The padder was adjusted to leave 100 weight percent of solution in the fabric. From the padder, the material was passed immediately into an atmosphere of saturated steam with provision of a reaction time of five minutes before exit. A reduction in the shrinkage due to laundering of from 68% to 30%, area basis, resulted.

*Example 3*

Flannel shirting material was passed through a solution of 100 gms. sulphuric acid. 50 gms, sulfamic acid and 40 gms. sodium hypochlorite (mol. wt. 74.45) per liter (available halogen concentration 3.8%) to a vacuum slot adjusted to leave 50 weight percent solution in the fabric, thence through a heater at 220° F. The continuously fed goods were moved at a rate selected to provide five seconds between wetting and slotting, with a course through the heater arranged to allow ten minutes exposure. Shrinkage in area due to laundering of the thus treated material was found to be 9% as against 25% on the untreated fabric.

*Example 4*

A piece of all wool blanketing was padded with a solution containing 2.36% available chlorine and 5.5% sulfamic acid, the solution having been adjusted to pH 0.7 with sulfuric acid. The padder was adjusted to leave 63 weight percent solution in the fabric, consequently 1.49% available chlorine was put on the fabric. The fabric was oven dried at 220° F. for fifteen minutes. A shrinkage in area of 4.6% was observed compared to 49.0% for the corresponding untreated fabric.

The area loss determination in the above examples was made after the samples were subjected to a washing test in a standard commercial laundry wheel. 24" in diameter by 20" long, running at 30 to 32 R. P. M. and inverting every five revolutions; enough water containing 0.2 sodium carbonate was used to keep the level 2" above the bottom of the inside cylinder; enough soap was added to give a running suds (i. e., a suds coming halfway up the cylinder). Four pounds of the material were washed for two hours at 140° F., given two 5 minute rinses at 120° F., centrifuged and dried at room temperature. Such a test is equivalent to ten ordinary washings.

The materials treated in accordance with the given examples exhibited a marked uniformity in response to dyeing. Tests on the fully finished samples disclosed a complete absence of tendering.

The foregoing description and examples set forth typical instances of the application of the principle of our invention, but do not constitute an exhaustive enumeration of the ways in which it can be practiced. Modifications within the scope and spirit of our invention will readily occur to the expert. It is thus our intention to claim our invention broadly and to be limited only by the appended claims.

We claim:

1. A process for imparting shrink resistance to

woolen and wool containing material, comprising: the step of applying, for a few seconds only, at a predetermined temperature not higher than room temperature and under conditions wherein substantially no reaction occurs, to said material a hypohalite solution containing from about 1% to about 6¾% available halogen; and the immediately subsequent step of abruptly increasing the said first named predetermined temperature to a predetermined point not less than 125° F. at which a reaction between the said wool and said substance takes place.

2. The process according to claim 1, wherein said increased temperature is in the range of about 200° F. to about 220° F.

3. A process for imparting shrink resistance to woolen and wool containing materials, comprising the steps of (1) rapidly contacting said material at a temperature not higher than room temperature with a hypohalite solution containing from about 1% to about 6¾% available halogen, and (2) substantially immediately thereafter and before a reaction between said material and said substance has gone to completion, exposing said material abruptly to at least 125° F. for a period greatly exceeding the length of said first step and for not substantially exceeding 15 minutes, whereby a reaction between the said material and said shrink resistance imparting agent is caused to take place.

4. A process according to claim 3, wherein said contacting step is performed in about five seconds.

5. A process according to claim 3, wherein said heat is applied in the form of steam.

6. A process according to claim 3, wherein said heat is applied in the form of neutral steam.

7. A process according to claim 3, wherein said heat is applied in the form of acidified steam.

8. A process for imparting shrink resistance to woolen or wool containing material comprising (1) the step of rapidly impregnating the said material at room temperature with a solution comprising water, a hypohalite in an amount to impart to said solution an available halogen content of from about 1% to about 6¾%, sulfamic acid and sulfuric acid, and (2) the step of substantially immediately thereafter and before a reaction between said material and said solution has gone to completion, exposing the impregnated material abruptly to heat for a period greatly exceeding the length of said first step and for not substantially exceeding 15 minutes substantially above room temperature.



9. A process for imparting shrink resistance to woolen or wool containing material, comprising (1) the step of rapidly impregnating the said material at room temperature with a solution comprising water, available halogen in a concentration from about 1% to about 6¾%, sulfamic acid and sulfuric acid, said solution having a pH below 1, and (2) the step of substantially immediately thereafter and before a reaction between said material and said solution has gone to completion, exposing the impregnated material abruptly to heat for a period greatly exceeding the length of said first step and for not substantially exceeding 15 minutes substantially above room temperature.

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