Jan. 13, 1925. 1,522,555

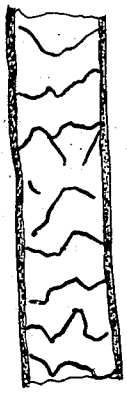
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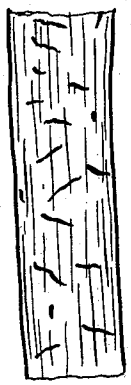
UNSHRINKABLE WOOL AND WOOLEN MATERIAL

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Fig. 1. Fig. 2. Fig. 3. Fig. 4.









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UNSHRINKABLE WOOL AND WOOLEN MATERIAL.

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To all whom it may concern:

Be it known that I, SAMUEL RUSSELL TROTMAN, a subject of the King of England, residing at Nottingham, England, have invented certain new and useful Improvements in Unshrinkable Wool and Woolen Materials; and I do hereby declare the following to be a full, clear, and exact description of the invention, such as will enable others skilled in the art to which it appertains to make and use the same.

This invention relates to the preparation of socalled unshrinkable wool and woolen materials.

Hitherto, chlorination of wool, for the purpose of making it unshrinkable, has been unsatisfactory. Insufficiently treated material is unsatisfactory because it is not sufficiently unshrinkable, and when present methods are carried far enough to render the material unshrinkable for ordinary purposes, the wool loses much weight, loses tensile strength, wears badly, becomes more soluble and contains a high percentage of damaged fibres.

It is the object of this invention to provide an improved process of rendering wool and woolen material substantially unshrinkable with a minimum loss of weight and strength and with substantial retention of the original properties of the wool. It is the further object of this invention to provide such a process that can easily be controlled and that can be effectively operated on a large scale.

Wool fibre is composed of a cortex covered with epithelial scales. In ordinary wool, the free edges of the epithelial scales are always distinctly visible. In chlorinated wool, these edges are no longer visible but appear to adhere to the cortex. In properly chlorinated wool, the epithelial scales present a smooth, opaque surface with perfectly plain outlines. With progressive chlorination, the outlines of these scales become less distinct and gradually they disappear. As the scales become thinner, the cortex becomes visible. Damage is denoted by the disappearance of the epithelial scales exposing the cortex of the fibre. This gives the fibre a rough surface marked with longitudinal streaks. Damage of this character may be either local or general.

The accompanying drawings show diagrammatic and enlarged portions of wool fibre in progressive stages of chlorination. Figure 1 illustrates the untreated wool fibre clearly showing the free edges of the epithelial scales. Figure 2 illustrates a well chlorinated sample still showing the outlines of the epithelial scales. Figure 3 illustrates a further stage of chlorination showing incipient damage. Figure 4 illustrates excessive chlorination showing complete destruction of the epithelial scales.

The tensile strength of the wool is principally a function of the cortex and, unless large numbers of the epithelial scales are damaged or unless damage proceeds to the cortical cells, the tensile strength is not materially affected. Increased affinity for dyes is approximately proportional to the percentage of damaged fibre and properly chlorinated wool does not show any marked increase. If the fibre is badly damaged, it wets immediately and becomes soft and almost slimy. Such damage causes a break-down rapidly in wearing and in washing. Undamaged material does not show any marked increase in affinity for moisture over the untreated material. The epithelial scales are more resistant than the cortical cells and if any damage is done to the scales, the solubility of the wool is increased. Such damage decreases the wear ability and washability of the wool and also causes undue loss in weight.

When wool is treated with solutions containing free chlorine, such as chlorine water, or bleaching powder solutions containing excess acid, the initial absorption is so rapid that the reaction becomes uncontrollable and the wool fibre is immediately damaged. Likewise when the wool is soured or subjected to a preliminary acid drench, the wool fibre is damaged upon immersion in a solution containing a hypochlorite, first because the acid upon the fibre liberates chlorine when the wool first enters the bath and second because the amount of acid carried over is, practically without exception, in excess of that required to combine with the base.

Moreover, when wool is treated with such solutions containing free chlorine, sensible damage is produced with very low absorption of chlorine. With such solutions, damage results even with an absorption of 1% or less of chlorine on the weight of the wool.

I have found that by subjecting the wool or woolen material to treatment with hypo-

chlorous acid substantially free from chlorine, the wool or woolen material is rendered unshrinkable for all practical purposes without undue loss of weight or strength and with substantial retention of the original properties of the wool.

I have found that with hypochlorous acid, the initial rate of absorption is comparatively slow, and that this reaction can be easily controlled. For instance, using solutions containing 0.5 grams available chlorine per litre with a total chlorine content equal to 7.5% of the weight of the wool I found the following chlorine absorptions in percent of the weight of wool treated in the indicated time:

Time in minutes.	Percentage of chlorine absorbed.	
	Hypochlorous acid.	Bleaching powder and hydrochloric acid.
5	1.4	3.0
10	2.9	4.8
15	3.7	5.0
20	4.3	5.1
30	4.9	5.2
40	5.2	5.6

Using solutions containing 0.8 grams available chlorine per litre with a total chlorine content equal to 7.0% of the weight of the wool, I found the following:

Time in minutes.	Percentage of chlorine absorbed.	
	Hypochlorous acid.	Bleaching powder and hydrochloric acid.
5	1.8	4.0
10	3.6	5.6
15	4.2	6.0
20	4.7	6.1
30	5.1	6.3
40	5.3	6.5

The rate of chlorine absorption from acidified bleaching powder solutions was approximately three times as great as that from a solution of hypochlorous acid substantially chlorine free. In each of these comparisons the concentration of available chlorine and the total available chlorine in the solutions compared was identical. Chlorine water acts in the same manner as the acidified solution of bleaching powder.

I have further found, that with hypochlorous acid, sensible damage is not produced in the wool until over 4% of chlorine on the weight of the wool has been absorbed. With an absorption between 4% and 5%, traces of damage are sometimes visible, but this damage is not serious when it does occur and is of a nature not unusual in the best class of goods which have been made unshrinkable. When free chlorine is present, however, absorption in amounts as low as 1% or less cause damage, as has been pointed out.

I have found that the slow reaction rate of hypochlorous acid combined with the comparatively high chlorine absorption possible without injury with this material makes hypochlorous acid peculiarly suitable for the treatment of wool and woolen materials to render them unshrinkable. The slow reaction rate renders it easy to control the reaction and the high chlorine absorption possible without injury, diminishes the effect of any variation in the time factor and materially cuts down spoilage. In the first of the examples just given, for instance, a dangerous amount of chlorine, over 1%, would have been absorbed in less than two minutes using the acidified bleaching powder solution, whereas, using a solution of hypochlorous acid, over thirty minutes would have to elapse before a dangerous amount of chlorine was absorbed. In the second example, using the acidified bleaching powder solution a dangerous amount of chlorine would have been absorbed in less than two minutes, whereas, with a solution of hypochlorous acid over twenty minutes would have to elapse before a dangerous amount of chlorine was absorbed. Chlorine water acts in the same manner as acidified bleaching powder solutions.

I have further found, however, that it is not necessary to use as large a quantity of chlorine to produce unshrinkability as can safely be used as hypochlorous acid. A chlorine absorption of between 4 and 5% is possible without damage from a solution of hypochlorous acid, but I have found that unshrinkability, for practical purposes, accompanied by absence of damage, is produced by chlorine absorptions as low as 0.25% to 0.5% from hypochlorous acid solutions. With such small quantities of chlorine as hypochlorous acid, say less than 1%, there is the further important advantage that time is not an important factor. Wool and woolen materials may be left in such solutions for a long time with impunity, in fact until all the hypochlorous acid is used up. Using hypochlorous acid in small percentages, there is practically no loss of weight during the process beyond that due to water alone.

Low concentrations of hypochlorous acid do not give absolute unshrinkability but will produce a fabric unshrinkable for all ordinary purposes. Concentrations of 0.25 to 1.00% of chlorine on the weight of the fabric will produce unshrinkability to what is commonly called "normal" washing but prolonged boiling with soap and water will produce slight shrinkage. Test as to shrinkability may be conveniently made as

follows: Boil a piece of fabric of measured area, say six inches square, for one hour in strong soap and water with occasional rubbing during this period, then wash the fabric several times in clear hot water, and remeasure after drying it. Fabrics treated with such low percentages will show a slight shrinkage by this test. On the other hand, if the washing is carried out with soap and water at a temperature of about 40° or 50° C. no shrinking or felting will occur. Where more complete unshrinkability is required, it is better to use a higher concentration of available chlorine, say from 1.5 to 3.0% on the weight of the fabric or even higher percentages up to the point where loss in weight or damage to the fibres becomes appreciable. It will be apparent that the quantity used depends upon the result desired.

Using low chlorine absorptions, dilute solutions can be used without unnecessary increase in bulk, however, control of the solution concentration is a readily used means to control the process. Concentration control may be operated separately from or in conjunction with the control of total available chlorine. The permissible time of immersion without a dangerous absorption of chlorine is shorter, the more concentrated the solution. I have found that sensible damage becomes apparent only when the concentration of chlorine as hypochlorous acid exceeds 3 grams per litre. Chlorine concentrations, as hypochlorous acid, of 2 grams per litre or less will cause neither damage nor undue loss in weight.

Using even concentrated solutions and a high total chlorine content, the time required to cause commercial damage to the wool or woolen material treated with solutions of hypochlorous acid substantially chlorine free is so long that the process is practically fool-proof on a large scale. In the ordinary commercial processes, the loss in weight and amount of damage is considerable and variable. The results of the present process, on the other hand are readily predicable.

The following specific example will serve to more fully illustrate the practice of my invention, but it is not intended as any limitation of the scope of the invention.

To treat a woolen fabric, I find it preferable to first wet it with about twenty times its own weight of water. I then lift the fabric out of the bath and stir in a sufficient amount of a solution of hypochlorous acid, substantially chlorine free, of known concentration, to obtain the necessary percentage of chlorine in the bath, say 0.5% of the weight of the fabric. I find it preferable for ease of working to then dilute the bath to about thirty times the weight of the fabric although this is not essential. I then return the fabric to the bath and work occasionally for a period between 30 and 45 minutes. I then rinse the fabric and treat with a suitable dechlorinating agent such as sulfurous acid or bisulphite. Following this, the fabric may be washed in the usual way and then subjected to any ordinary treatment. The fabric so treated is unshrinkable and suffers no damage or undue loss of weight.

In the preferred practice of this invention, the solution of hypochlorous acid should be substantially chlorine free. Such solutions can be readily prepared by passing chlorine gas into water containing chalk in suspension or a carbonate in solution. Such a solution should be allowed to stand for some time and the avaliable chlorine then determined by titration or other suitable means. If solution containing calcium carbonate in suspension is not to be used forthwith, it is preferable to keep it in contact with the chalk. Or a suitable solution can be prepared by treating bleaching powder solutions with boric acid. With low concentrations of either bleaching powder or boric acid such solutions substantially consist of hypochlorous acid. With any concentrations the ratio of hypochlorous acid to free chlorine is very high. If strong bleach solutions and an excess of boric acid are used, free chlorine is also produced. For concentrations up to 3 grams of available chlorine per litre a mixture of bleaching powder and boric acid is hypochlorous acid substantially chlorine free.

Solutions of bleaching powder, up to concentrations of about 4% of chlorine on the weight of the fabric, can be used without causing more than slight damage, such as is not infrequent in the best class of unshrinkable goods as previously prepared, if the proper precautions are taken to avoid free chlorine. Solutions of this character can be prepared in several ways. For example, a clear solution of bleaching powder can be made up, the available chlorine determined as by titration, and then properly diluted to the requisite concentration of available chlorine. This solution can then be treated with just sufficient hydrochloric or sulphuric acid to neutralize the alkalinity and liberate the combined hypochlorous acid. This quantity can be calculated or experimentally determined. In practice it is usually sufficient to carefully stir in dilute acid until the mixture is no longer alkaline to litmus. Weaker acids can be used with corresponding advantage for slight excesses are not as critical, and in the concentrations used for the chlorination of wool, an excess will produce hypochlorous acid substantially free from free chlorine. Weak acids such as acetic acid or phosphoric acid are suitable and boric acid is particularly advantageous as has been pointed out. Or this

solution of bleaching powder may be treated with a slight excess of sodium bicarbonate, sodium acid phosphate or the like, or solutions of the same.

The present invention can be practiced in conjunction with any method for preparing solutions of hypochlorous acid which are substantially free from chlorine. Solutions of hypochlorous acid which contain quantities of cholorine that are without effect on the fabric are substantially free from chlorine for the purpose of the present invention. Due to the safety and simplicity of operation, I prefer to use solutions of hypochlorous acid substantially free from chlorine, but it will be apparent that, by restricting the relative amount of free chlorine present, as compared to the amount of hypochlorous acid, below the point at which material loss of weight and fibre damage begins, the advantages of this invention can be realized with a corresponding sacrifice in ease of operation. The advantages of the present invention will be partially realized by the use of solutions containing chlorine of which the preponderating amount is present as hypochlorous acid and to an extent roughly approximated by the ratio of hypochlorous acid to free chlorine. I prefer, however, to use solutions substantially free from chlorine as such.

It will thus be seen that the present invention provides an improved process of rendering wool and woolen material substantially unshrinkable without undue loss of weight or strength or damage to the material by treatment with solutions of hypochlorous acid which can be easily controlled and effectively practiced on a commercial scale.

Having described my invention, I claim:

1. The process of rendering wool or woolen material unshrinkable, comprising subjecting said wool or woolen material to treatment with a solution of hypochlorous acid substantially chlorine free.

2. The process of rendering wool or woolen material unshrinkable, comprising subjecting said wool or woolen material to treatment with a solution of hypochlorous acid substantially free from chlorine and hydrochloric acid.

3. The process of rendering wool or woolen material unshrinkable, comprising subjecting said wool or woolen material to treatment with a solution of hypochlorous acid substantially free from chlorine, said solution containing not more than 3 grams of available chlorine per litre.

4. The process of rendering wool or woolen material unshrinkable, comprising subjecting said wool or woolen material to treatment with a solution of hypochlorous acid substantially chlorine free, said solution containing not more than 1% of available chlorine on the weight of the wool or woolen material.

5. The process of rendering wool or woolen material unshrinkable, comprising subjecting said wool or woolen material to treatment with a solution of hypochlorous acid substantially chlorine free, said solution containing not more than 5% of available chlorine on the weight of the wool or woolen material.

6. The process of rendering wool or woolen material unshrinkable, comprising subjecting said wool or woolen material to treatment with a solution of hypochlorous acid substantially chlorine free, said solution containing not more than 1% of available chlorine on the weight of the wool or woolen material and not more than 2 grams of available chlorine per litre.

7. The process of rendering wool or woolen material unshrinkable, comprising subjecting said wool or woolen material to treatment with a solution of hypochlorous acid substantially free from chlorine and containing about 0.5% of available chlorine on the weight of the wool or woolen material for a period of between about 30 and 45 minutes.

8. The process of rendering wool or woolen material unshrinkable, comprising subjecting said wool or woolen material to treatment with a solution containing hypochlorous acid and maintaining the concentration of free chlorine below the point at which loss of weight and fibre damage becomes appreciable.

9. The process of rendering wool or woolen material unshrinkable, comprising subjecting said wool or woolen material to treatment with a solution of hypochlorous acid substantially free from chlorine and then subjecting the wool or woolen material to treatment with a dechlorinating agent.

In testimony whereof I affix my signature.

SAMUEL RUSSELL TROTMAN.