

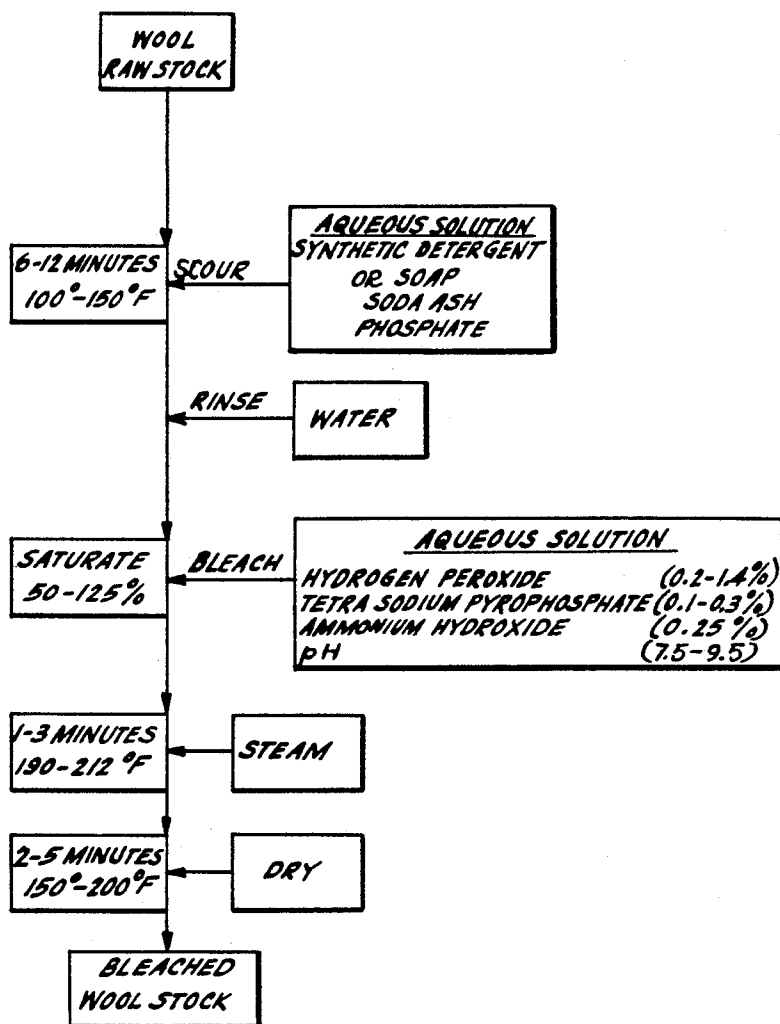
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BLEACHING OF WOOL

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BLEACHING OF WOOL

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This invention relates to the bleaching of wool fibers, and particularly to the bleaching of wool fibers by a rapid and continuous process.

It is known that wool fibers are sensitive to the concomitant action of alkalis and oxidizing chemicals. The effect of these chemicals on such fibers is also known to be magnified with an increasing temperature. For these reasons, it has been the practice heretofore to bleach wool and other protein fibers at comparatively low temperatures, for example at room temperatures to about 135° F., with solutions containing peroxygen chemicals and an alkali such as tetrasodium pyrophosphate and ammonia.

In order to obtain a high degree of bleach on wool under these conditions, it has been necessary to conduct the bleach for several hours, or in some cases overnight. Even under these conditions, and with the exercise of care to avoid deleterious conditions, the alkali solubility of the bleached wool indicates that degradation has started.

The damage suffered by wool even when it is bleached under conditions carefully selected to avoid the concomitant action of heat, alkali and hydrogen peroxide, has led prior workers away from attempting to bleach wool rapidly, by the more severe means commonly employed with other less sensitive fibers. These more severe methods, which have been found useful for example with cotton, involves operation at elevated temperatures, of the order of 200° F. or higher, and the use of aqueous alkaline hydrogen peroxide baths. While some workers have gone so far as to dry the wool after it has been scoured and bleached, by heating it to about 150° to 180° F., they have been careful to avoid applying this heat to wool which is in an alkaline condition for fear of the severe damage to the fibers which would be expected to result.

It is a feature of the present invention to provide a process of bleaching wool in a short time.

It is a further feature of this invention to provide such a process which operates under conditions heretofore considered extreme, yet which frequently results in less damage to the goods than is encountered with previously employed mild bleaching methods.

It has now been found, quite surprisingly, that wool fibers can be bleached and dried in a matter of only a few minutes, by a process which achieves whites as high as are obtained with prior long-time non-continuous bleaching methods, and which at the same time degrades or damages the wool even less than these prior methods, by saturating the goods with about 50% to 120% on the weight of the goods of an aqueous alkaline hydrogen peroxide solution having a pH of about 7.5 to 9.5, and containing about 2 to 14 grams, and preferably about 3 to 9 grams, of hydrogen peroxide on a 100% basis, per liter of solution, and containing about 0.1 to 0.3% of an alkali metal salt of a molecularly dehydrated phosphoric acid, steaming the goods saturated with this aqueous alkaline hydrogen peroxide solution at a temperature of about 190° to 212° F. for a controlled time of about 1 to 3 minutes, and thereafter drying the goods by application of drying heat at a temperature of 150° to 200° F. until the goods contain no more than about 10% of free water.

Preferably, a scouring or cleaning operation will be

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incorporated in the process, preceding the bleach. Goods in the form of raw wool in its natural state may be scoured in an aqueous solution containing about 0.25 to 0.5% of a nonionic or anionic syndet, or a soap, for about 10 minutes at a temperature of about 100° to 150° F., and, prior to saturation with the bleach, washed with water to remove the bulk of the scouring chemicals. Where the scouring solution is alkaline, for example at a pH of about 9 to 10, it is washed until the goods have a pH of not greater than about 8.5. Goods which have previously been scoured in the raw stock, and are in the state of sliver top yarn, or fabric, may be lightly scoured to remove any dirt, oil, or other extraneous material picked up during processing. The scouring operation at most adds only about 6 to 12 minutes to the over-all process, which in the absence of the scour requires as little as 4–8 minutes, and makes possible complete scouring, bleaching and drying of the wool by a continuous, rapid and highly efficient process.

It has been found that this particular series of steps, which even including the scour requires only on the order of about 10 to 20 minutes over-all, provides a highly efficient bleaching action on the wool fibers, yet frequently does not degrade them even as much as prior, supposedly mild, non-continuous bleaching methods. This is particularly surprising when one considers that it is not necessary herein to wash the alkaline bleaching solution from the goods after they have been steamed, prior to drying them at an elevated temperature. One would suspect that the presence of the alkaline materials in the goods would damage them, particularly at the elevated temperatures employed in the steaming and subsequent drying operations.

The single attached drawing represents a flow diagram of applicants' process, showing the progress of the wool from raw stock through a bleached material.

The scouring operation which will be conducted on the fiber prior to bleaching, in cases where raw wool is to be processed or where lubricating oil or other processing impurities are present, involves washing them in an aqueous washing solution containing about 0.25 to 0.5% of an anionic or nonionic detergent such as the nonionic alkyl phenol-ethylene oxide condensates, or the anionic alkyl aryl sulfonates, fatty alkanolamides and the like, or a soap such as a derivative of oleic acid or other low titer soap. If it is desired to create an alkaline condition in the scouring solution, an alkali such as soda ash, or a molecularly dehydrated salt of phosphoric acid may be added to the scouring bath to provide a pH of on the order of 9 to 10.

The scour may be conducted in one or more vessels, and is carried out to free the wool of grease, perspiration, and other natural impurities. The scouring operation is conducted at a temperature of about 100° to 150° F., and for about 6 to 12 minutes. When the scour is employed in the present process, it includes a following wash to remove the bulk of the scouring agents from the wool. Following this step, the goods are saturated with an aqueous alkaline bleaching solution. Saturation herein means that the goods contain from about 50% to 120% of bleach solution on the weight of the goods. Preferably, the goods will be squeezed to remove excess water prior to introduction of the bleach solution, and then run through a vessel containing an aqueous solution having a pH of about 7.5 to 9.5 and containing about 2 to 14, and preferably about 3 to 9, grams of hydrogen peroxide on a 100% basis per liter of solution, and about 0.1 to 0.3% of a salt of a molecularly dehydrated phosphoric acid such as tetrasodium pyrophosphate, sodium tripolyphosphate, or sodium hexametaphosphate. To raise the pH of the bleach solution, it may also advantageously,

but not necessarily, contain up to about 0.5% of ammonium hydroxide, provided that no more of this ingredient is used than will provide a maximum pH of about 9.5.

Following introduction of the bleaching solution, the goods are subjected to the action of steam at a temperature of about 190° to 212° F., for about 1 to 3 minutes. It has been found that steaming of the goods for either less than about 1 minute or more than about 3 minutes reduces the bleaching effect of the peroxide solution.

Following the steam treatment, the goods are dried in about 2 to 5 minutes by the application of dry heat at a temperature of about 150° to 200° F. The drying heat suitably may be applied by contact of the goods with a heated body, for example a body of dry air or other gas, or a moderately heated solid body such as a screen or drum. In this step, the goods desirably are dried until they contain no more than about 10% of free water, and are ready for subsequent processing.

The goods need not be washed before they are dried. It has been found, quite surprisingly, that neither the steaming procedure nor the subsequent dry-heating step, even in the presence of the alkaline system which is impregnated in the wool goods, causes substantial degradation of the fibers. In fact, the goods actually frequently undergo even less degradation than like goods bleached by mild non-continuous bleaching methods, considered heretofore to be necessary to avoid damage.

The process is readily conducted in well known wool-treating equipment, known to the trade as a "scouring train." This equipment comprises a series of 4 or 5 or more vats or bowls laid end-to-end, through which the wool passes. The first two or three bowls may contain a scouring solution consisting of synthetic detergents or soaps. If the scour is employed, the next one or two bowls will contain water for rinsing these scouring materials from the goods. The final bowl in the series contains the hydrogen peroxide bleaching solution. The time the goods spend in each bowl is about 2 to 3 minutes.

As the wool stock leaves the scouring train saturated with aqueous, alkaline hydrogen peroxide solution, it passes onto a conveyor within a housing, or is dropped into a chute, or passes through any other equipment which will provide a holding time of about 2 minutes and at the same time permit the wool to be steamed. It is then passed through a hot air dryer.

The present process, advantageously carried out in the solubility value or degradation of the continuously scouring, bleaching and drying wool stock in a matter of only about 15 minutes. This time consists of about 6 to 12 minutes of scouring, about 1 to 3 minutes of steaming, and about 2 to 5 minutes of drying. The wool produced by this process is as white as that obtained by conventional means in 2 to 3 hours or longer, and the alkali solubility value or degradation of the continuously bleached wool of the present process is normally lower than that obtained by such conventional methods.

The following examples are presented by way of illustration of the process of the present invention only, and are not to be deemed limitative of the breadth of this invention in any way. The wool stock bleached in these examples was scoured in a standard scouring train, by being treated for 2½ minutes in each of three vats containing: in the first vat, 1,500 gallons of water at 90° F.; in the second vat 18 pounds of an alkyl phenol type nonionic detergent in 2,200 gallons of water at 140° F., and in the third vat 8 pounds of the same detergent in 2,000 gallons of water at 140° F., and then water rinsed until substantially all of the detergent was removed.

The wool employed was a wool stock of the type to be used in carpeting, and had a percent reflectance after scouring and rinsing of 40.5%, and an alkali solubility after such treatment of 12.4%.

EXAMPLE 1

Non-Continuous Bleaching Method

One hundred grams of the wool scoured by the above method was bleached in 1 liter of the following solution.

Ingredients:	Amount, gms.
Hydrogen peroxide, 35% -----	26.1
Tetrasodium pyrophosphate (anhydrous) ----	2.4
28% NH ₄ OH -----	1.6
Water to make 1 liter.	

The wool was held in the solution for 2½ hours at a temperature of 135° F., following which it was rinsed with water until it exhibited a neutral pH, and dried at about 150° F. for 15 minutes until it contained 8% of free moisture. The bleached wool had a reflectance of 65%, and had an alkali solubility of 24.9.

EXAMPLE 2

Present Method

One hundred grams of the wool scoured by the above method was soaked for 2 minutes in 1 liter of the following solution at room temperature.

Ingredients:	Amount, gms.
Hydrogen peroxide, 35% -----	40
Tetrasodium pyrophosphate (anhydrous) ----	2
28% NH ₄ OH -----	4.5
Water to make 1 liter.	

The wool was squeezed until it contained its own weight of the above solution, and was steamed for 2 minutes at 210° F. It was then dried at 185° F. in an oven for 15 minutes until it contained about 8% of free moisture.

The wool had a reflectance of 68%, and its alkali solubility value was only about 15.3.

EXAMPLE 3

Present Method

One hundred grams of the wool scoured by the above means was treated as in Example 2, except that the following solution was used.

Ingredients:	Amount, gms.
Hydrogen peroxide, 35% -----	19.2
Tetrasodium pyrophosphate (anhydrous) ----	2.0
28% NH ₄ OH -----	4.5
Water to make 1 liter.	

The whiteness of this wool was 62%, or slightly less than that obtained with the conventional immersion bleach of Example 1; its alkali solubility, however, was only 14.7.

EXAMPLE 4

Present Method

The procedures of Examples 2 and 3 were followed with the exception that the ammonium hydroxide was omitted from the respective bleach solutions of these examples. The wool samples were bleached to reflectances of 68% and 61%, respectively, and their alkali solubilities were 19.6 and 12.1, respectively.

The alkali solubility test employed above was conducted according to ASTM-D1283-53T. Present reflectances were determined with a Gardner Multipurpose Reflectometer using a blue filter.

The above examples demonstrate the present process applied to wool carpet yarn. However, it is to be understood that the process is suitable for the bleaching of wool in any form, including blends of wool with synthetic or other fibers such as nylon, rayon, Dacron, Orlon, Acrilan, cotton, and the like. The wool may be in the form of raw stock, tow, sliver, yarn, or fabric.

Pursuant to the requirements of the patent statutes, the principle of this invention has been explained and

exemplified in a manner so that it can be readily practiced by those skilled in the art, such exemplification including what is considered to represent the best embodiment of the invention. However, it should be clearly understood that, within the scope of the appended claims, the invention may be practiced by those skilled in the art, and having the benefit of this disclosure, otherwise than is specifically described and exemplified herein.

What is claimed is:

1. Process for bleaching and drying wool rapidly and without serious degradation of the wool fibers, comprising saturating said wool with an aqueous bleaching solution having a pH of 7.5 to 9.5 and containing 2 to 14 grams of hydrogen peroxide per liter of solution, and 0.1 to 0.3% by weight of an alkali metal salt of a molecularly dehydrated phosphoric acid, steaming the saturated wool at a temperature of 190° to 212° F. for 1 to 3 minutes, and thereafter drying said steamed wool by dry heating for about 2 to 5 minutes at a temperature of 150° to 200° F. until the wool contains not more than 10% of free water.

2. Process for bleaching and drying wool rapidly and without serious degradation of the wool fibers, comprising saturating said wool with an aqueous bleaching solution having a pH of 7.5 to 9.5 and containing 3 to 9 grams of hydrogen peroxide per liter of solution, and 0.1 to 0.3% by weight of an alkali metal salt of a molecularly dehydrated phosphoric acid selected from the group consisting of tetrasodium pyrophosphate, sodium tripolyphosphate and sodium hexametaphosphate, steaming the saturated wool at a temperature of 190° to 212° F. for 1 to 3 minutes, and thereafter drying said steamed wool by dry heating for about 2 to 5 minutes at a temperature of 150° to 200° F. until the wool contains not more than 10% of free water.

3. Process for bleaching and drying wool rapidly and without serious degradation of the wool fibers, comprising saturating said wool with an aqueous bleaching solution having a pH of 7.5 to 9.5 and containing 2 to 14 grams of hydrogen peroxide per liter of solution, up to 0.5% ammonium hydroxide, and 0.1 to 0.3% by weight of an alkali metal salt of a molecularly dehydrated phosphoric acid, steaming the saturated wool at a temperature of 190° to 212° F. for 1 to 3 minutes, and thereafter drying said steamed wool by dry heating for about 2 to 5 minutes at a temperature of 150° to 200° F. until the wool contains not more than 10% of free water.

4. Process of scouring, bleaching and drying wool rapidly and without serious degradation of the wool fibers, comprising scouring said wool by washing it in an aqueous alkaline solution of an agent from the group consisting of nonionic detergents, anionic non-soap detergents and soaps, washing the scouring agent from the wool with water, saturating said wool with an aqueous bleaching solution having a pH of 7.5 to 9.5 and containing 2 to 14 grams of hydrogen peroxide per liter of solution,

and 0.1 to 0.3% by weight of an alkali metal salt of a molecularly dehydrated phosphoric acid, steaming the saturated wool at a temperature of 190° to 212° F. for 1 to 3 minutes, and thereafter drying said steamed wool by dry heating for about 2 to 5 minutes at a temperature of 150° to 200° F. until the wool contains not more than 10% of free water.

5. Process of scouring, bleaching and drying wool rapidly and without serious degradation of the wool fibers, comprising scouring said wool by washing it for about 6 to 12 minutes in an aqueous alkaline solution of an agent from the group consisting of nonionic detergents, anionic non-soap detergents and soaps, washing the scouring agents from the wool with water, saturating said wool with an aqueous bleaching solution having a pH of 7.5 to 9.5 and containing 3 to 9 grams of hydrogen peroxide per liter of solution, and 0.1 to 0.3% by weight of an alkali metal salt of a molecularly dehydrated phosphoric acid selected from the group consisting of tetrasodium pyrophosphate, sodium tripolyphosphate and sodium hexametaphosphate, steaming the saturated wool at a temperature of 190° to 212° F. for 1 to 3 minutes, and thereafter drying said steamed wool by dry heating for about 2 to 5 minutes at a temperature of 150° to 200° F. until the wool contains not more than 10% of free water.

6. Process of scouring, bleaching and drying wool rapidly and without serious degradation of the wool fibers, comprising scouring said wool by washing it for about 6 to 12 minutes at a temperature of 150° to 200° F. in an aqueous alkaline solution of an agent from the group consisting of nonionic detergents, anionic non-soap detergents and soaps, washing the scouring agent from the wool with water, saturating said wool with an aqueous bleaching solution having a pH of 7.5 to 9.5 and containing 2 to 14 grams of hydrogen peroxide per liter of solution, up to 0.5% ammonium hydroxide, and 0.1 to 0.3% by weight of an alkali metal salt of a molecularly dehydrated phosphoric acid, steaming the saturated wool at a temperature of 190° to 212° F. for 1 to 3 minutes, and thereafter drying said steamed wool by dry heating for about 2 to 5 minutes until the wool contains not more than 10% of free water.

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UNITED STATES PATENT OFFICE
CERTIFICATE OF CORRECTION

252-99
150

Patent No. 3,148,018

September 8, 1964

Bernard Kestner Easton et al.

It is hereby certified that error appears in the above numbered patent requiring correction and that the said Letters Patent should read as corrected below.

Column 3, line 48, strike out "solubility value or degradation of the" and insert instead -- above scouring train, provides a means for --; column 5, line 8, for "is" read -- as --; column 6, line 14, for "agents" read -- agent --; line 29, strike out "at a temperature of 150° to 200° F." and insert the same after "minutes" in line 41, same column 6.

Signed and sealed this 5th day of January 1965.

(SEAL)

Attest:

ERNEST W. SWIDER
Attesting Officer

EDWARD J. BRENNER
Commissioner of Patents

UNITED STATES PATENT OFFICE
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