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PROCESS FOR SHEENING PILE FABRICS

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This invention relates to an improved process for sheening pile fabrics, and more particularly to a method for sheening the pile of wool rugs, carpets, and fabrics of a like nature, all of which will be referred to herein as wool rugs.

The process commonly known as "rug sheening" has come to be of increasing importance in the manufacture of pile fabrics. The methods of treatment known to and used by the art, most of which have involved treatments with solutions containing bleaching powder or sodium hypochlorite, have not, in spite of attempts at rigid control, produced completely satisfactory results.

Operators employing the methods of the art or variations of them have encountered numerous difficulties and found that they possess many serious disadvantages. Outstanding among these have been the inability of such methods to give reproducible results. This is a particular disadvantage in the manufacture of carpets where a certain pattern and finish must remain substantially uniform throughout a large quantity of material which is manufactured over a long period of time. For various reasons which are more fully discussed below, it has been very difficult if not impossible to obtain a constant sheen and a uniform shade of color by the use of the methods of sheening known to the art. In many cases it has been found that a treatment appropriate to produce the desired sheen also causes a loss or fading of the color in the pile and thus in the fabric itself, or sometimes introduces an entirely different tint in the starting color. A further outstanding difficulty has been that a serious loss of tensile strength of the pile often accompanies the sheening process. This, of course, greatly reduces the life of the fabric by lowering its resistance to wear. Certain of the known methods have also been found to possess a tendency to remove the twist from the yarn, thus making sheening difficult and causing the finished product to have an undesirable fuzzy appearance. This tends to disturb the original demarkations in the patterns. Further disadvantages which the proposed methods of sheening have failed to overcome are the running together of colors in pattern fabrics, uneven or streaky sheening, and variations in color or degree of sheen from the top of the pile to the bottom.

It is the primary object of the present invention to provide a chemical method of pile fabric sheening which will eliminate or minimize the above recited difficulties and disadvantages which

attend the use of the methods known to the art. The process of the present invention accomplishes this by providing a chemical treatment which produces a minimum loss of fiber strength and at the same time produces a uniform maximum sheen without altering or destroying the color or the texture of the fabric. This invention, furthermore, provides a process which is easily operated and controlled.

I have found that a high degree of sheen may be attained and the disadvantages hitherto involved either eliminated or minimized by subjecting the pile of the rug to a two-stage treatment in successive aqueous solutions, the initial stage comprising treatment in a solution containing hydroxyl ions and peroxide in concentrations within the limits prescribed below, and the second stage comprising treatment in a dilute but well regulated solution of a hypochlorite. The presence, within the prescribed limits of concentrations, of the peroxide and of the hydroxyl ions in the first-stage treating solution and of the hypochlorite in the second-stage treating solution are the prerequisites for the effectiveness of the treating process.

The peroxide may be supplied to the solution by dissolving therein a peroxide forming material which may be organic or inorganic in character. Inorganic peroxide forming materials include the peroxides such as hydrogen peroxide, alkali metal peroxides (sodium peroxide for example), alkaline earth metal peroxide (barium peroxide for example) and per salts which in aqueous solution form hydrogen peroxide, sodium perphosphate, sodium perborate, sodium percarbonate, and the like, being examples of such per salts. Organic peracids and their salts also serve as sources of hydrogen peroxide, peracetic acid, perbenzoic acid, sodium peracetate and sodium perbenzoate being examples of such organic materials. Organic and organo-metallic salts of hydrogen peroxide, such as tetramethyl ammonium peroxide and triethyltin peroxide may also be used for this purpose.

The hydroxyl ions essential for the effectiveness of the peroxide solution may be supplied by any suitable hydroxyl forming compound capable of producing hydroxyl ions in such concentrations as to provide a pH within the range prescribed hereinafter. Thus, inorganic bases such as the alkali and alkaline earth hydroxides (sodium hydroxide, calcium hydroxide, etc., for example) may be used as the source of hydroxyl ions. Organic bases and organo-metallic bases which are

sufficiently soluble in water also may be used as a source of the hydroxyl ions. Tetramethyl ammonium hydroxide and triethyltin hydroxide are illustrative examples of such compounds. In general, salts of strong bases and weak acids, whether organic or inorganic, hydrolyze in water to form free hydroxyl ions and these salts may also be used as a source of hydroxyl ions. Trisodium phosphate, sodium carbonate, sodium tetraborate sodium acetate, and the like, are appropriate examples of such salts. Hydroxyl ions may also be supplied by hydrolysis of the peroxide forming material where such material hydrolyzes in water to form hydroxyl ions. In an operation employing hydrogen peroxide, H_2O_2 , as the source of peroxide, the hydroxyl ion concentration may be supplied for example by an alkali metal hydroxide, such as sodium hydroxide. In operations in which alkaline peroxides or per salts are employed, sodium peroxide, Na_2O_2 , for example, a sufficient hydroxyl ion concentration may result from the hydrolysis of the peroxide or per salt in aqueous solution. In some cases, an additional quantity of hydroxyl ion may be added with advantage.

In the second stage of the process the treatment is effected with hypochlorite chlorine such as is provided by alkali metal and alkaline earth metal hypochlorites. High test hypochlorite, bleaching powder, sodium hypochlorite and lithium hypochlorite may be used advantageously for this purpose. Where a considerable portion of sheening is to be done by hypochlorite in this second stage, necessitating a relatively high concentration of available chlorine, I prefer to use, for example, a calcium hypochlorite substantially free of calcium chloride and containing a minimum of free lime.

A rinsing step preferably follows both the initial treatment by the alkaline peroxide solution and also the second stage hypochlorite treatment. The process may be completed by a souring treatment in accordance with known methods. The present process may be carried out in any apparatus suitable for subjecting fabrics to successive treatments in different solutions with intermediate rinses.

It has been found that in most cases best results are obtained when the solution during the first stage is maintained at a temperature between about 50° and 120° F., and at a somewhat higher temperature in the second stage, and when the period of treatment is from about 15 minutes to about one hour. The treatment of my present invention may be followed by rinsing and "souring" steps in accordance with known methods.

Wool fiber is composed of a cortex covered with epithelial scales. While I am unable to state definitely the course of the chemical reaction occurring between a substance such as bleaching powder and a wool fiber in a single stage sheening process, it appears that the imbricated or serrated surface of the fiber is first attacked and the protecting scales removed. This exposes the fibrous material itself to the destructive action of the chemicals in the treating solution and the resulting damaged fiber is unsatisfactory for many purposes, including use in pile fabrics. Treatment of a pile rug with an aqueous solution containing hydroxyl ions and peroxide as herein described on the other hand apparently does not destroy the epithelial scales or attack the fiber directly, thus eliminating the disadvantages attending the destructive action on the

pile itself. I am unable to state definitely the reasons for the improved results obtained by the use of the herein described process, but it would appear that the reaction taking place is a very limited one which tends to change the imbricated surface of the fiber to a relatively smooth surface which has an increased tendency to reflect directly rather than scatter light rays. In this way the desired sheen is imparted to the fabric. It is to be understood, however, that my invention is not limited to any particular hypothesis as to the mechanism of the process.

As indicated above the difficulties encountered in producing or enhancing the sheen on wool rugs are more pronounced with certain weights of rug and colors than with others. An improved process for sheening the pile of wool rugs which involves a two-stage treatment comprising treating the fabric for a limited period of time with a dilute caustic solution and thereafter with an aqueous solution of calcium hypochlorite is described and claimed in Patent No. 2,185,210, granted to myself, Lorenzo D. Taylor and Varton M. Kalusdian. The present process may be used alternatively with the said two-stage process with equal advantage on many types of rug and in certain instances with superior results. This is particularly true on types of rug which, due to an easily altered color, or to particular pile characteristics, are difficult to sheen without adversely affecting either the color or the pile or both. For example, on certain rugs the first stage alkali treatment of the referred to process has a tendency to "muss" the pile which may, to a certain extent, be corrected by the subsequent hypochlorite treatment. When an alkaline peroxide solution is used in the first step, however, the twist of the yarn is not materially disturbed, even in those cases in which alkali alone has a detrimental effect. Furthermore, it is possible, other conditions being the same, to obtain a desired sheen with a lower concentration of available chlorine in the second step when peroxide is present in the first step. Inasmuch as hypochlorite has greater "tendering" action than peroxide this has the advantage of minimizing tensile strength losses in the pile of the fabric. I have also observed that in certain instances, notably with some shades of green, the presence of peroxide in the caustic step aids in color retention by preventing the inception of a slight yellow tint in the green color which sometimes accompanies the use of alkali and hypochlorite without peroxide.

In many instances a satisfactory sheen may be attained with no loss of color or fiber degradation by the use of a single-stage treatment involving a solution containing hydroxyl ions and peroxide. Such a process is described and claimed in a copending application, Serial No. 462,838 filed by myself and Lorenzo D. Taylor on Oct. 21, 1942. On rugs which are amenable to a hypochlorite treatment, the two-stage process of the present invention has the following advantages over the use of a single-stage process employing peroxide and alkali alone. The cost of materials is less because of the fact that less peroxide is necessary in the two-stage process, the less expensive hypochlorite serving to accomplish some of the results caused by the peroxide. The reaction of hypochlorite is more rapid and hence the total treating time for a rug is decreased. A higher sheen may be obtained in rugs whose color is not adversely affected by the hypochlorite. Certain shades of blue have been found to be unduly

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brightened by the use of sufficient peroxide to produce the desired sheen; whereas in the present process the subsequent action of hypochlorite tends to subdue the brightness thus produced and the net result is a color substantially the same as that of the original rug.

In the improved sheening process of the present invention optimum results with respect to the attainment of a maximum sheen concurrently with a minimum effect on the color and pile are attained by the proper proportioning in the first stage of hydroxyl ion and peroxide concentrations within the limits given below and by the choice of the most advantageous time and temperature for carrying out the reaction in each individual instance, and in the second stage by the proper choice and proportioning, with respect to the conditions of the first stage, of the hypochlorite concentration as well as the time and temperature of this step. The exact concentrations used must be determined by experiment on the particular rug to be sheened. Variations in color and weight of the fabric result in variation of the hydroxyl ion and peroxide concentrations which produce best results. The aqueous solution employed in the first step of the process of the present invention should have as its lower limit a pH of about 7.5 and as its upper limit a pH of about 13. While the foregoing represents the lower and upper limits of hydroxyl ion concentrations to be employed, we have found that in the practice of the invention there appears to be an optimum inner range, having as its lower limit a pH of about 10 and as its upper limit a pH of about 12.5.

In the two-stage treatment of the present invention substantially no sheening occurs at pH values below about 7.5 in the treating solution used in the first step of the process even with large concentrations of peroxide. Considerable quantities of peroxide, near the upper limit of peroxide concentration as defined hereinbelow, are required when the pH of the treating solution is at least 7.5 but below about 10. It is more economical to use a treating solution having a pH of at least 10 or higher because more reasonable concentrations of peroxide are effective at such pH values. The alkalinity of the treating solution used in the first stage of the process produces deleterious effects on the pile when pH values closely approaching the upper limit of 13 are used. Thus, at such high pH values the pile tends to become untwisted and the rug is rendered fuzzy and difficult to shear. The color of a rug is also adversely affected by too high an alkalinity, green rugs for example becoming increasingly yellow as the upper pH limit is approached. It is advantageous, however, to use as high a pH as is consistent with the texture and color of the rug being treated because the required effective concentration of peroxide, which is relatively expensive with respect to the hydroxyl ion forming material, is lower at relatively high pH values. With a given quantity of peroxide, sheening is more pronounced as the pH increases to about 12.5. I prefer, therefore, to operate in a pH range between about 10 and about 12.5 in order to attain the most effective and economical results.

The alkalinity of the treating solution used in the first stage of the process of the invention generally decreases during the sheening operation so that an initial pH of about 7.5 to 10 may decrease to a value below 7.5 and even decrease to the acid side during the first treat-

ing operation, under which latter conditions little or no sheening takes place. I have found it to be advantageous to buffer the treating solution where its initial pH is between about 7.5 and 10 in order to obtain maximum sheening effectiveness. I have found in actual practice that a buffered treating solution having an initial pH of 8.0 drifted only to a pH of 7.55 during the treatment and that wholly effective sheening was thus obtained. Any compound capable of acting as a buffer in an alkaline solution and inert with respect to peroxide may be used. Acetic acid and a $\text{Na}_2\text{HPO}_4\text{--KH}_2\text{PO}_4$ mixture are examples of buffers which may be used with advantage in accordance with my invention.

We shall herein and in the appended claims define the concentration or proportions of the peroxide and other peroxygen compounds used in the preparation of the treating solution of the invention in terms of the equivalent weight of the peroxygen group, $-\text{O}_2-$, contained in the amount of the peroxygen compound used. By reference herein and in the appended claims to the concentration of the peroxygen compound in terms of the equivalent of the peroxygen group, or to the peroxygen group concentration, we do not intend to indicate that either the particular peroxygen compound or the peroxygen group is present, as such, in the solution in the specified concentration. It will be understood that in thus defining the concentration of the peroxygen compound in the solution we include all of the peroxygen group present and capable of reaction whether at any given instance it be in the form of the peroxygen compound, peroxide ion, hydrogen peroxide, or in any other form. By thus defining the concentration of the peroxygen compound in terms of the equivalent of the peroxygen group we wish to avoid any academic question such as whether the peroxygen compound is present, as such, or reacts in the form of an ion or, in the case of a salt, the extent of hydrolysis at any given time or the degree of ionization of hydrogen peroxide which may be produced by such hydrolysis.

As above defined, the concentration of the peroxygen compound in the treating solution of the invention should be equivalent to a concentration of the peroxygen group, $-\text{O}_2-$, falling within an outer range of about 0.14 gram per liter to about 2, and advantageously not more than about 1.5 grams per liter. No appreciable sheening is effected by peroxide solutions containing less than about 0.14 gram per liter of the peroxygen group and amounts of the peroxygen group in excess of about 2 grams per liter are unnecessary and frequently undesirable in my two-stage process. I have found that there is a preferred inner range adapted, in the two-stage process of the invention, to give a maximum sheen with minimum color change and pile degradation in the case of a number of types of rugs when treated in solutions in which the peroxygen group concentration range is from about 0.36 gram per liter to about 0.8 gram per liter. The use of peroxide solutions containing the peroxygen group within this inner range has been found to give particularly satisfactory sheening in conjunction with the subsequent hypochlorite treatment. The outer range corresponds to about 0.5 to 6-8 grams per liter of "100 volume hydrogen peroxide" solution, while the inner range corresponds to about 1.4 to 3.1

grams per liter of "100 volume hydrogen peroxide" solution. The designation "100 volume peroxide" is used in the trade to indicate that one volume of the aqueous solution employed is capable of liberating 100 volumes of oxygen. Such a solution is approximately 27-28% hydrogen peroxide, H_2O_2 , by weight. In cases where per salts or other sources of peroxide are used directly, the limits of hydroxyl and peroxygen group concentrations should also be within the limits just stated. In certain instances, it is desirable to maintain a hydroxyl ion concentration in excess of that provided by hydrolysis of an alkaline peroxide or per salt where the peroxide or per salt is used as the source of the peroxygen group. This may be accomplished by adding the necessary quantity of an alkali metal hydroxide, but the total hydroxyl ion concentration, including that produced by hydrolysis, (i. e. the pH) should nevertheless be within the above stated limits in order to produce best results.

The temperature of the solution during treatment during both stages is advantageously maintained between about 50° and about 120° F., but as in the case of the concentrations of the reactants the temperature value giving the maximum sheen with the minimum of harmful effects must be determined for each particular type of rug. In general it may be said that best results are obtained by operating during the first stage at approximately room temperatures, i. e. within the range of about 60° to 95° F. The temperature used in this step should also be chosen with regard to that used in the hypochlorite stage which is advantageously higher than that employed in the first stage. The most desirable time of treatment in the first step must be determined with respect to the rug being treated, the solutions used, and the conditions to be used in the second step. In general a treating time within the range 15 minutes to 60 minutes will be satisfactory, a treatment approximating 30 minutes being particularly desirable.

Following the treatment with the alkaline peroxide solution the rug is advantageously given a water rinse of from 2 to 5 minutes at about 90° F. It is then subjected to treatment in an aqueous solution of a hypochlorite, in which the concentration of available chlorine is insufficient to cause a substantial loss of tensile strength of the fiber and insufficient to alter the color of the pile. The time of treatment and temperature will vary depending on the type, weight and color of the rug being sheened, the strength of the hypochlorite solution and the conditions and results obtained in the first stage.

To obtain the full benefits of the process of the present invention when using calcium hypochlorite in the second stage of the process, the calcium hypochlorite should be substantially free of calcium chloride and containing a minimum of free lime. The presence of free lime in the second or hypochlorite stage of the process sometimes tends to increase the sheen obtained, but if present to excess frequently causes undesirable changes in the color of the fabric. Calcium hypochlorite compositions prepared as described in United States Letters Patent No. 1,787,048, granted to Robert B. MacMullin and Maurice C. Taylor on an application filed August 16, 1928, are particularly useful in the process of the present invention. Such calcium hypochlorites are not to be confused with conventional bleaching powder or chlorinated lime. They differ ma-

terially from bleaching powder or chlorinated lime both in chemical constitution and in content of available chlorine. Instead of being a complex compound including chloride and hypochlorite, they are true calcium hypochlorites. The difference in available chlorine content, however, is more readily determined than is the more fundamental difference in chemical constitution. Characteristically such calcium hypochlorites contain upwards of 50% available chlorine and, with advantage with respect to this invention, upwards of 60% available chlorine.

The concentration of the hypochlorite in the solution of the second stage should be such as to produce an available chlorine concentration within the limits 0.5 to 3.0 grams per liter, the exact amount depending largely on the fabric undergoing treatment, and being insufficient to cause a substantial loss of tensile strength of the fiber and insufficient to alter the color of the pile. In certain instances I have found that a concentration within these limits which is suitable for one rug may be too great for another rug similar to the first in all respects except color. The concentrations in such cases must be kept within an even narrower range.

The effectiveness of the alkaline peroxide solution used in the first stage of my process may be enhanced by the additional presence of a detergent or wetting agent such as those referred to in the above-identified copending application of myself and Lorenzo D. Taylor, and with the same beneficial results enumerated therein. Such detergents or wetting agents are preferably synthetic water-soluble detergents or wetting agents such as the higher organic sulfates, sulfonates or quaternary ammonium compounds (sodium lauryl sulfate, sodium oleyl sulfate, sodium dodecyl benzene sulfonate, and trimethyl octyl ammonium chloride, for example). The detergent, or a mixture of such detergents, is preferably used in concentrations ranging from about 0.1 to 1.0 gram per liter and, with particular advantage, within the range of about 0.1 to 0.5 gram per liter.

Following the hypochlorite treatment the rug is with advantage subjected to one or preferably two 5-minute water rinses at a moderate temperature, 90° F. for example. This may be followed by a souring step comprising treatment in any appropriate dilute acid solution such as one containing 2 cc. per liter formic acid.

The following specific examples will serve to illustrate the process of the invention as well as to indicate, by comparison of Example II with Example I the improved results obtained in one specific instance when compared to an operation in which peroxide is not present in the first step.

Example I

A sample of a Wilton rug of green color and medium weight was treated in a washwheel containing 3 liters of an aqueous solution made up from 5 g. p. l. NaOH having a pH of about 13, and the treatment continued for 30 minutes at a temperature of 65° F. The rug was rinsed in water at a temperature of 90° F., following which 3 liters of a calcium hypochlorite solution of the above recited properties was introduced. This solution had an available chlorine concentration of 0.75 g. p. l. and was maintained at a temperature of approximately 90° F. during a 15 minute treatment. The rug was then given two 5 minute water washes at 90° F. and soured in a dilute

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formic acid solution for 10 minutes. As a result of this treatment the rug had acquired a good sheen and the pile was in good condition, but the color could only be graded as fair due to the inception of a yellow tint in the original green.

Example II

A similar sample from the same green rug used in Example I was subjected to a similar treatment, the exception being that the solution in the first step of this example was made up from 5 g. p. l. NaOH and also 1.45 g. p. l. "100 volume H₂O₂," this being equivalent to approximately 0.37 g. p. l. of the peroxygen group (and having a pH of about 12.5), and in the second stage the concentration of available chlorine from the hypochlorite approximated 1.5 g. p. l. After drying, the rug was found to possess the desired high degree of sheen, the pile to be in better condition with no noticeable fiber degradation, and in addition the original green color remained unchanged showing no trace of the yellow tint obtained when peroxide was not present in the first step.

Example III

A similar sample of the same green rug was treated in a solution made up from 5 g. p. l. of sodium hydroxide, 4 g. p. l. of 100-volume hydrogen peroxide for a period of 30 minutes at a temperature of 70° F. The rug was rinsed in water at a temperature of 90° F. and then treated with a sodium hypochlorite solution containing 0.75 g. p. l. of available chlorine. This treatment was of 15 minutes duration at approximately 90° F. The rug was given two 5-minute water washes at 90° F. and soured in a dilute formic acid solution for ten minutes. The color, sheen and pile of the rug were substantially the same as obtained by the process of Example II.

Example IV

A similar sample from the same green rug used in Example I was treated in a wash wheel containing 3 liters of aqueous solution made up from 5 g. p. l. of sodium hydroxide, 3 g. p. l. of 100-volume hydrogen peroxide, during the period of 30 minutes at a temperature of about 70° F. After this first treatment, the rug was rinsed in water at about 90° F. and treated with 3 liters of a hypochlorite solution containing 1 g. p. l. of available chlorine as lithium hypochlorite. This treatment lasted for 15 minutes at 90° F. after which the rug was rinsed twice and soured with dilute formic acid solution as before. The color, sheen and pile of the rug after this treatment were substantially the same as found in Example II.

This application is a continuation-in-part of my application Serial No. 310,575, filed December 22, 1939.

I claim:

1. The method of sheening the pile of wool rugs which comprises subjecting the pile to treatment in an aqueous solution containing hydroxyl ions and a peroxygen compound, the concentration of the said compound in the solution being equivalent to not less than about 0.14 nor more than about 2 grams of the peroxygen group, —O₂—, per liter and the pH of the solution being between about 7.5 and 13, and thereafter subjecting the pile to treatment in an aqueous solution of a hypochlorite in which the concentration of available chlorine is within the range of about 0.5 to 3 grams per liter and insufficient to cause a substantial loss of tensile strength of the fiber of the pile and insufficient to alter the color of the pile.

2. The method of sheening the pile of wool rugs which comprises subjecting the pile to treatment in an aqueous solution containing hydroxyl ions and a peroxygen compound, the concentration of the said compound in the solution being equivalent to not less than about 0.14 nor more than about 1.5 grams of the peroxygen group, —O₂—, per liter and the pH of the solution being between about 7.5 and 13, and thereafter subjecting the pile to treatment in an aqueous solution of a hypochlorite in which the concentration of available chlorine is within the range of about 0.5 to 3 grams per liter and insufficient to cause a substantial loss of tensile strength of the fiber of the pile and insufficient to alter the color of the pile.

3. The method of sheening the pile of wool rugs which comprises subjecting the pile to treatment in an aqueous solution containing hydroxyl ions and a peroxygen compound, the concentration of said compound in the solution being equivalent to not less than about 0.36 nor more than about 0.8 grams of the peroxygen group, —O₂—, per liter and the pH of the solution being between about 10 and 12.5, and thereafter subjecting the pile to treatment in an aqueous solution of a hypochlorite in which the concentration of available chlorine is within the range of about 0.5 to 3 grams per liter and insufficient to cause a substantial loss of tensile strength of the fiber of the pile and insufficient to alter the color of the pile.

4. The method of sheening the pile of wool rugs which comprises subjecting the pile to treatment in an aqueous solution containing hydroxyl ions and a peroxygen compound, the concentration of the said compound in the solution being equivalent to not less than about 0.14 nor more than about 2 grams of the peroxygen group, —O₂—, per liter and the pH of the solution being between about 7.5 and 13, and thereafter subjecting the pile to treatment in an aqueous solution of calcium hypochlorite in which the concentration of available chlorine is within the range of about 0.5 to 3 grams per liter and insufficient to cause a substantial loss of tensile strength of the fiber of the pile and insufficient to alter the color of the pile.

5. The method of sheening the pile of wool rugs which comprises subjecting the pile to treatment in an aqueous solution containing hydroxyl ions and a peroxygen compound, the concentration of the said compound in the solution being equivalent to not less than about 0.14 nor more than about 2 grams of the peroxygen group, —O₂—, per liter and the pH of the solution being between about 7.5 and 13, and thereafter subjecting the pile to treatment in an aqueous solution of sodium hypochlorite in which the concentration of available chlorine is within the range of about 0.5 to 3 grams per liter and insufficient to cause a substantial loss of tensile strength of the fiber of the pile and insufficient to alter the color of the pile.

6. The method of sheening the pile of wool rugs which comprises subjecting the pile to treatment in an aqueous solution containing hydroxyl ions and a peroxygen compound, the concentration of said compound in the solution being equivalent to not less than about 0.14 nor more than about 2 grams of the peroxygen group, —O₂—, per liter and the pH of the solution being between about 7.5 and 13, and thereafter subjecting the pile to treatment in an aqueous solution of lithium hypochlorite in which the concentration of available chlorine is within the range of about 0.5 to 3 grams per liter and insufficient to cause a sub-

stantial loss of tensile strength of the fiber of the pile and insufficient to alter the color of the pile.

7. The method of sheening the pile of wool rugs which comprises subjecting the pile to treatment in an aqueous solution of an alkali metal hydroxide and hydrogen peroxide, the concentration of the hydrogen peroxide in the solution being equivalent to not less than about 0.14 nor more than about 2 grams of the peroxygen group, $-O_2-$, per liter and the pH of the solution being between about 7.5 and 13, and thereafter subjecting the pile to treatment in an aqueous solution of a hypochlorite in which the concentration of available chlorine is within the range of about 0.5 to 3 grams per liter and insufficient to cause a substantial loss of tensile strength of the fiber of the pile and insufficient to alter the color of the pile.

8. The method of sheening the pile of wool rugs which comprises subjecting the pile to treatment in an aqueous solution of sodium hydroxide and hydrogen peroxide, the concentration of the hydrogen peroxide in the solution being equivalent to not less than about 0.14 nor more than about 2 grams of the peroxygen group, $-O_2-$, per liter and the pH of the solution being between about 7.5 and 13, and thereafter subjecting the pile to treatment in an aqueous solution of a hypochlorite in which the concentration of available chlorine is within the range of about 0.5 to 3 grams per liter and insufficient to cause a substantial loss of tensile strength of the fiber of the pile and insufficient to alter the color of the pile.

9. The method of sheening the pile of wool rugs which comprises subjecting the pile to treatment in an aqueous solution of sodium hydroxide and hydrogen peroxide, the concentration of the hydrogen peroxide in the solution being equivalent to not less than about 0.14 nor more than about 2 grams of the peroxygen group, $-O_2-$, per liter and the pH of the solution being between about 7.5 and 13, and thereafter subjecting the pile to treatment in an aqueous solution having dissolved therein a hypochlorite containing upwards of 50% available chlorine, the concentration of available chlorine in said solution being within the range of about 0.5 to 3 grams per liter but less than an amount sufficient to introduce an appreciable loss of tensile strength in the fiber of the pile and less than an amount sufficient to alter the color of the pile.

10. The method of sheening the pile of wool rugs which comprises subjecting the pile to treat-

ment in an aqueous solution of sodium peroxide, the concentration of the peroxide in the solution being equivalent to not less than about 0.14 nor more than about 2 grams of the peroxygen group, $-O_2-$, per liter and the pH of the solution being between about 7.5 and 13, and thereafter subjecting the pile to treatment in an aqueous solution having dissolved therein a hypochlorite containing upwards of 50% available chlorine, the concentration of available chlorine in said solution being within the range of about 0.5 to 3 grams per liter but less than an amount sufficient to introduce an appreciable loss of tensile strength in the fiber of the pile and less than an amount sufficient to alter the color of the pile.

11. The method of sheening the pile of wool rugs which comprises subjecting the pile to treatment in an aqueous solution of sodium perborate, the concentration of the perborate in the solution being equivalent to not less than about 0.14 nor more than about 2 grams of the peroxygen group, $-O_2-$, per liter and the pH of the solution being between about 7.5 and 13, and thereafter subjecting the pile to treatment in an aqueous solution having dissolved therein a hypochlorite containing upwards of 50% available chlorine, the concentration of available chlorine in said solution being within the range of about 0.5 to 3 grams per liter but less than an amount sufficient to introduce an appreciable loss of tensile strength in the fiber of the pile and less than an amount sufficient to alter the color of the pile.

12. The method of sheening the pile of wool rugs which comprises subjecting the pile to treatment in an aqueous solution containing hydroxyl ions, a peroxygen compound and a synthetic organic water-soluble detergent selected from the class consisting of higher organic sulfates, sulfonates and quaternary ammonium compounds free of fatty acid ester substituents, the concentration of the said peroxygen compound in the solution being equivalent to not less than about 0.14 nor more than about 2 grams of the peroxygen group, $-O_2-$, per liter, the pH of the solution being between about 7.5 and 13, and the detergent concentration being between about 0.1 and 1.0 gram per liter, and thereafter subjecting the pile to treatment in an aqueous solution of a hypochlorite in which the concentration of available chlorine is within the range of about 0.5 to 3 grams per liter and insufficient to cause a substantial loss of tensile strength of the fiber of the pile and insufficient to alter the color of the pile.

JAMES DOUGLAS MacMAHON.