UNITED STATES PATENT OFFICE

2.201.929

TREATMENT OF FIBERS OR FIBROUS MA-TERIALS CONTAINING KERATIN

John Bamber Speakman, Far Headingley, Leeds. England

Application December 9, 1935, Se-No Drawing. rial No. 53,687. In Great Britain December 10, 1934

> 14 Claims. (Cl. 167—87.1)

This invention relates to improvements in the treatment of substances containing keratin, such as hair and wool, and is particularly concerned with treatments for the application of a permasoment set to such substances or for the removal of stress from materials made from keratins such as wool and with the treatment of keratin materials in or before finishing operations.

Keratin-containing fibers are customarily sublo jected to steaming or hot water treatment in the presence of compounds which may include reducing agents at or only slightly below the boiling point for the release of strain and the application of a permanent set.

It has been proposed to treat wool or hair with a boiling bisulphite solution while tensioning the wool or hair. Such treatment at the boiling point will result in disruption of the disulphide bonds of keratin and the immediate formation of S-NH bonds as indicated by the following equations:

where R-S-S-R and R'-NH2 represent the peptide and associated side chains of keratin. The reactions take place with such readiness that the maximum possible number of linkages result. Bisulphite solutions, however, have the disadvantage that at the boiling point of water they are capable of serious destructive attack on hair and at lower temperatures the rate of formation of linkages as represented by equation 2 above, is negligibly small and therefore proceeds far too slowly to be commercially useful.

The object of the present invention is to pro-40 vide a treatment whereby release of strain and permanent set are obtained over a much larger range of temperatures below the customary temperatures and especially down through temperatures to as low as room temperatures where de-45 sired.

The invention consists in treating fibrous substance containing keratin, such as wool or hair. with a solution of a reducing agent in alkaline solution to effect reduction and disruption of the 50 constituent disulphide or cystine bond of keratin with the formation of sulphydryl groups, and then, while maintaining the substance in a tormation which it is desired to render permanent, causing the formation of further disulphide bonds 55 in or between the fiber molecules by subjecting

the fibrous substance to the action of an oxidising agent.

The invention differs essentially from the prior processes of treating keratin-containing fibers with sulphites or bisulphites at the boiling point 5 in that release of strain in the keratin containing fibers is brought about by disruption of the disulphide bonds of keratin, and in the following scheme:

and permanent set or form is given to the relaxed fibers according to the invention, not by the formation of -SNH- bonds by interaction of sulphonic groups with amino groups as in the 15 prior treatment referred to, but solely by direct union of sulphydryl groups themselves to form cystine bonds, brought about by the action of the oxidising agent.

Suitable reducing agents which may be em- 20 ployed according to the invention are sodium sulphide, sodium hydrosulphide, cysteine hydrochloride, sodium hypophosphite, sodium thiosulphate, sodium hydrosulphite, sodium dithionate. They may be employed in the form of so- 25 lutions whose concentrations may be varied in accordance with the nature of the reducing agent and its mode of use. The reducing agent may be formed in situ, for example if keratin containing fibers are moistened with alkali, e. g., 30 baryta, hydrosulphide is formed by reaction with the fiber molecules, which may be caused to attain the required concentration by maintaining the total volume of solution low.

Reducing agents as a whole, in alkaline solution 35 disrupt the disulphide bond of keratin with the formation of sulphydryl groups, although the quantity of disulphide bonds disrupted in any keratin-containing substance varies not only from one reducing agent to another, but also with 40the conditions of concentration, pH and temperature at which a reducing agent is employed.

The disruption of the disulphide bond by reduction according to the invention may be employed to relieve stress in the fibers, which 45 stress may have been caused, for example, by deforming the fibers in giving them a desired configuration, and leaves them in such a state that formation of further bonds may take place whereby the fibers will retain permanently any 50set or configuration which may have been given them.

Although the solution of reducing agent may be of any desired alkalinity, it is found in certain cases that for the application of permanent sets 55

10

2,201,929 2

or waves to keratin fibers, conditions are most favourable to the permanence and efficiency of the set when the reducing solution actually in contact with the hair is at or above pH 10, pref-

5 erably pH 11.

At or above pH 10 reducing agents are effective, since alkalies are able to attack unstrained as well as strained disulphide bonds, sulphydryl and sulphenic acid groups being produced by hy-10 drolysis, which latter groups are reduced by the reducing agent to sulphydryl groups. Disulphide bonds may be formed from the suphydryl groups by the subsequent action of an oxidising agent.

The following are examples of convenient ways 15 of carrying the invention into effect as applied by way of example to the permanent waving of

35

Example I

The hair after a preliminary shampoo is treated with a 6 per cent, sodium hydrosulphide solution brought to pH 10 by addition of sodium carbonate or sodium metasilicate. The hair may be treated with the solution before or after wind-25 ing on a curler or the solution may be circulated about the hair wound on a curler. The solution is maintained in contact with the hair for about 15 minutes at room temperature or above, after which a solution of an oxidising agent may be 30 applied, for example a 10 volume hydrogen peroxide solution or 10 per cent. ammonium persulphate solution.

The hair may then be subjected to the usual finishing operations of permanent waving.

Example II

In this case, a solution of a reducing agent as specified in Example I, is employed, but instead of winding the hair on curlers it is formed into 40 waves or curls on the head after being moistened with the solution. To facilitate manipulation of the hair, a thickening agent may be added to the solution of reducing agent, for instance agaragar or kieselguhr in sufficient amount to give 45 the consistency of a paste. Suitable wetting agents may also be added. The solution is allowed to remain in contact with the hair for about 15 minutes at about room temperature or above. The formation of further bonds to give 50 permanence to the waves or curls can then be brought about by treatment with an oxidising agent.

An alternative manner of carrying out the treatment of the invention according to this ex-55 ample, is to form the hair into waves or curls on the head and treat it with the reducing agent, the subsequent procedure being as set out above.

The reagents may be applied by spraying or

any other suitable means.

In the application of the invention to fabrics made of keratins such as wool, the usual crabbing and blowing operations for the removal or equalising of stress in the fabrics may be replaced by a reduction treatment in cold or warm alkaline 65 solutions of reducing agents, for example a 0.6 per cent. solution of sodium sulphide. fabrics may then be treated for the removal of the reducing agent, for example by washing, or by treatment in a solution of an oxidising agent 70 such as a 10-volume hydrogen peroxide solution. Removal of reducing agents generally may be effected by treatment with an oxidising agent.

The invention may also be applied to the removal of distortion in knitted fabrics of wool 75 arising from uneven stress in the yarn, by treat-

ment of the fabric with an alkaline solution of a reducing agent and subsequent removal of the reducing agent as by treatment with an oxidising agent.

Yarns for the manufacture of curl fabrics or 5 artificial astrakhan and Persian lamb fabrics may have a permanent set given to them by treatment of the deformed yarn with a reducing agent followed by removal of the reducing agent as by treatment with an oxidising agent.

I claim:

1. A method of giving a desired permanent form to fibrous substances containing keratin, which comprises treating the substance with an alkaline solution of a reducing agent free from 15 sulphite at a pH of at least 10 and a temperature appreciably below the boiling point whereby to effect reduction and disruption of the constitutent disulphide or cystine bond of keratin with the formation of sulphydryl groups, and 20 then, while maintaining the substance in a form which it is desired to render permanent, causing the formation of further cystine bonds through interaction of the sulphydryl groups by subjecting the fibers to the action of an oxidising agent 25 comprising a per-compound.

2. A method as claimed in claim 1, wherein the reducing agent is removed from the substance prior to treatment with the oxidising agent.

- 3. A method of permanently waving hair which 30 comprises treating the hair with an alkaline solution of a reducing agent free from sulphite at apH of at least 10 and a temperature appreciably below the boiling point whereby to effect reduction and disruption of the constituent disulphide or cystine 35 bond of keratin of the hair with the formation of sulphydryl groups and then, while maintaining the hair in a curled or waved condition, causing the formation of further cystine bonds in the hair molecules by interaction of the sulphydryl 40 groups by subjecting the hair to the action of an oxidising agent comprising a per-compound.
- 4. A method as claimed in claim 3 wherein the hair in the form of strands is wound on curlers after treatment with the reducing agent and is 45 treated with the oxidising agent while it is on the curlers.
- 5. A method as claimed in claim 3 wherein the reducing agent employed is an alkali metal sulphide, cysteine hydrochloride, sodium hypophos- 50 phite, sodium thiosulphate, sodium hydo-sulphite and sodium dithionate.
- 6. A method as claimed in claim 3 wherein the reducing agent employed is an alkali metal hydrosulphide.
- 7. A method as claimed in claim 3 wherein the reducing agent employed is cysteine hydrochloride.
- 8. A method as claimed in claim 1 wherein the per-compound is hydrogen peroxide.
- 9. A method as claimed in claim 1 wherein the per-compound is ammonium persulphate.
- 10. A method as claimed in claim 3 wherein the per-compound is hydrogen peroxide.
- 11. A method as claimed in claim 3 wherein the 65 per-compound is ammonium persulphate.
- 12. A method as claimed in claim 3, wherein reducing agent is removed from the hair prior to treatment with the oxidising agent.
- 13. A method of permanently waving hair 70 which comprises treating the hair with a solution of an alkaline reducing agent free from sulphite at a pH of at least 10 and a temperature appreciably below the boiling point whereby to effect reduction and disruption of the constituent 75

2,201,929

3.

disulphide or cystine bond of keratin of the hair with the formation of sulphydryl groups and then, while maintaining the hair in a curled or waved condition, causing the formation of further cystine bonds in the hair molecules by interaction of the sulphydryl groups by subjecting the hair to the action of a solution of a peroxide.

14. A method of permanently waving hair which comprises treating the hair with a solution of an
11) alkaline reducing agent free from sulphite at a pH of at least 10 and a temperature appreciably

below the boiling point whereby to effect reduction and disruption of the constituent disulphide or cystine bond of keratin of the hair with the formation of sulphydryl groups and then, while maintaining the hair in a curled or waved condition, causing the formation of further cystine bonds in the hair molecules by interaction of the sulphydryl groups by subjecting the hair to the action of a solution of a per-salt.

JOHN BAMBER SPEAKMAN.

10