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PATENT SPECIFICATION

1173.004



NO DRAWINGS

1173.004

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COMPLETE SPECIFICATION

Process for Treating Hair

I, FUMIO UMEZAWA, of No. 12, 1-chome, Shinbashi, Manato-ku, Tokyo-to, Japan, of Japanese Nationality, do hereby declare the invention, for which I pray that a patent may be granted to me, and the method by which it is to be performed, to be particularly described in and by the following statement.

This invention relates to a process for use in the treatment of hair to effect at least one of the operations of waving, straightening or dyeing. More particularly it provides a process which may be carried out at room temperature and, for example, waving (or straightening) and colouring may be simultaneously accomplished.

It is generally known to treat hair with mildly alkaline reacting reagents, such as dilute alkali metal hydroxide solutions, amine solutions or more especially alcoholamine solutions such as triethanolamine. The present invention is a further development of this basic technique and originates from the Applicants observation that hair so treated is readily amenable to treatment with dissolved metal

compounds which appear to form a chelate with the keratin of the hair.

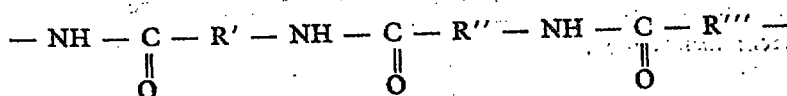
The invention consists in a process for effecting at least one of the operations of hair waving, hair straightening and hair colouring, in which the hair is contacted with a composition made by mixing:—

(I) a source of metal ions produced from (a) (i) a watersoluble salt of Mg, Ca, Zn, Cd, Ba, Sr, Al, Co, Sn, Mn, Fe, Cr, Ag, Au, Pt, Ni or Cu with (ii) an alkaline solution of an enzyme, a protein, a peptide or an amino acid or (b) a suspension of calcium oxide, together with

(II) 0.1—2% by weight of non-ionic or anionic surface active agent;

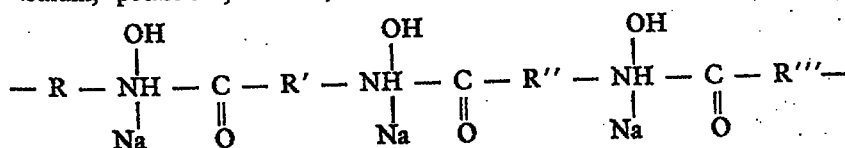
said composition being held at pH 7 to 12 by (III) an organic amine or a source of alkali metal ion or ammonium ion also yielding hydroxyl ions in aqueous solution.

The hair protein keratin is constituted of various kinds of amino acids, having the following structural formula in which one amino acid after another is combined in succession and containing occasional sulphidic linkages.



When hair is swollen after being soaked in alkaline solution, it is combined with ions of sodium, potassium, lithium, ammonium and

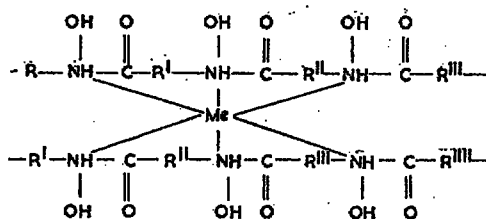
the like. In the case of sodium, it is presumed that the substance having the following structural formula is formed:



[Price 4s. 6d.]

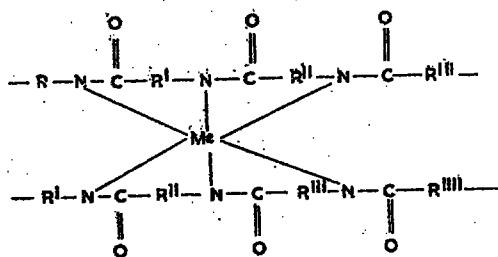
After the swollen hair is wound up on a rod and soaked in alkaline solution the alkali metal ions combined with the hair protein are replaced by the metal atoms of the metal compound forming hair protein chelate compounds of various metals with the resultant creation of waving or straightening of the hair and/or colouring.

The substance resulting from this procedure can be expressed by the following structural formula:



where Me = metal

By dehydrating this, the following is obtained:



Of course, the predicted reaction above is rather hypothetical and should not be taken as limiting on the broad scope of the invention. However, as far as can be ascertained, it does appear that this form of chelating with N atoms in keratin does take place leading to waving or straightening, and/or colouring of the hair. The hair presents different colours in accordance with the different metals used.

The extent to which hair is artificially curled may be classified into the following five categories:

Straight hair	1	
Mild hair-waves	2	30
Medium hair-waves	3	
Strong hair-waves	4	
Very strong hair-waves	5	

When artificially curled hair is immersed in warm water, the hair thus treated tends to revert to its original straight condition. The degree of this reversion is influenced by various factors, such as time of immersion and water temperature. In order to express the relation of the degree of reversion to time (minutes) and water temperature ($^{\circ}\text{C}$), the following method has been adopted:

For instance, if the hair waved to the degree of 5 is soaked in warm water at 40°C for one minute, and is restored to the original straight condition, then we express this fact by the numerals 40151, where 40 indicates 40°C , 1 indicates 1 minute, and 51 indicates that the degree of hair-waving has been changed from 5 to 1.

If the hair waved to degree 5, after being kept in warm water at 40°C for 30 minutes, still retains the same degree of waving, then the process is expressed as 403055.

If artificially straightened natural curl is soaked in warm water at 40°C for 30 minutes and still retains its straightness the condition is expressed as 403011.

The following experimental figures, given for completeness show results obtained in using components individually rather than as one composition.

Hair is wound up on a rod and soaked for 10 minutes at 50°C in a liquid containing 0.25% sodium hydroxide, rinsed in water, soaked again in solutions of various metal compounds (as ionic solutions) at 50°C for 10 minutes, washed in water, air dried, taken off the rod, dipped in water at 40°C for 1 minute and laid in order on the filter paper. The following results are obtained.

Alkali treatment	Metallic ions used for substitution	Degree of reversion
—	—	40151
+	—	40151
—	+	40151
+	aluminum	40153
+	magnesium	40153
+	cadmium	40153
+	zinc	40154
+	astrontium	40154
+	cobalt	40155
+	tin	40155
+	manganese	40155
+	iron	40155
+	chromium	40155
+	silver	40155
+	gold	40155
+	platinum	40155

The sources of the metal ions used in the substitution were metallic chlorides, sulfates, nitrates and acetates. These results have also revealed that the hair can be waved or straightened and/or coloured, if it is treated in metal salt solutions containing various metal ions after being soaked in or coated with alkaline

solution. If the hair to be treated is white or light-coloured, it is tinted with a different colour according to the kind of metal ion used while it is being waved or straightened, and the stronger the hair wave, the darker the hair colour, and vice versa, as is evident from the following table:

5

10

15

metallic ion	hair colour	degree of hair-waving
—	original colour	—
tin	white	strong
zinc	white	strong
cadmium	light yellow	strong
strontium	yellow	strong
manganese	yellowish brown	strong
iron	yellowish brown	strong
cobalt	blond	strong
nickel	grey	strong
chromium	blue	strong
silver	dark purple	strong
gold	purple	strong
platinum	golden	strong
chromium and iron mixed	light blond	strong
chromium and cobalt mixed	dark brown	strong
chromium, iron and cobalt mixed	brownish blond	strong
copper	green	strong

- The following table has been obtained as a result of soaking in warm water (40°C) for one minute, severed hair that has been curled by winding on a rod and soaking in or coating with the solution of sodium hydroxide and leaving for 10 minutes in the solution of 3% ferric chloride at different temperatures:
- 5

Concentration of sodium hydroxide (%)	Temperature for treatment (°C)		Degree of reversion
	Solution of sodium hydroxide	Solution of iron chloride	
0.1	50	50	40151
0.15	50	50	40152
0.2	50	50	40152
0.25	50	50	40555
"	50	40	40554
"	40	40	40152
"	30	40	40151
"	20	40	40151
0.3	50	50	40555
"	50	40	40554
"	40	50	40553
"	40	40	40552
0.4	20—30	20—30	40151
0.5	20—30	20—30	40151
1.0	20—30	20—30	40151

In consequence, it was clarified that in order to complete this chemical reaction, a sufficient quantity of metal ions to replace alkaline metallic ions is necessary, and that the temperature for treatment be raised to 50°C.

If severed hair is curled by winding up on the rod and soaking in alkaline solution and

then further soaking in 3% ferric chloride solution for five minutes, after which it is soaked again in warm water (40°C) for 5 minutes for reversion, the following relation between the pH of the solution and the degree of reversion may be obtained.

5

and
3%

type of alkaline solution	pH	Degree of reversion	
sodium hydroxide (0.2 — 1%)	12.6 — 13.0	40555	5
sodium silicate (1/4 mol)	12.0	40555	
sodium tertiary phosphate (or potassium) — (1/4 mol)	12.0	40555	10
sodium carbonate (1/4 mol)	11.5	40555	
sodium borate (1/4 mol)	9.0	40553	15
ammonium carbonate (1/4 mol)	8.0	40552	
sodium acetate (1/4 mol)	8.5	40552	
sodium lactate (1/4 mol)	8.5	40552	20
sodium citrate (1/4 mol)	8.0	40552	
disodium hydrogen phosphate (or dipotassium) (1/4 mol)	8.9	40551	25
sodium bicarbonate (1/4 mol)	8.1	40551	
mixture of sodium succinate and and sodium silicate	10.2	40554	30
mixture of sodium succinate and sodium carbonate	10.5	40554	
sodium hydroxide again after being treated by sodium carbonate	11.5 — 10.8	40555	35

It has become clear that if the hair is coated with a solution of sodium hydroxide and kept at a temperature of 50°C for 10 minutes, the quantity of alkali in the liquid remaining on the hair is reduced to an extremely small level due to the fact that alkaline ions are consumed by combining with the hair protein. In the case of the alkaline solution of 0.3—0.7% by weight concentration, the hair is waved to a low degree, but in the case of the solution of 2—4% by weight concentration it is waved to a high degree. If the hair is coated with 0.7% solution, and the excess liquid is removed by means of paper, gauze or the like, and coated again with the 0.7% solution, the hair-waving becomes twice as strong. Therefore, the repeated coating with relatively dilute alkaline solution makes it unnecessary to use a concentrated solution.

It is a feature of this invention that if the hair is treated by means of non-ionic (0.1—2% by weight polyoxyethylene alkylphenol ether solution) or anionic (0.1—2% by weight sodium sulfuric acid esters of high alcohol)

surface agent added to the alkaline solution instead of the latter alone, we can produce hair-waving and hair-colouring without applying heat. In experimentation to establish a suitable room temperature process, a hair-lotion I consists of an alkaline solution made of a mixture of 2—4% by weight solution of an alkali metal hydroxide or 8% by weight solution of sodium carbonate (getting hydroxy ions in solution) and an aqueous solution containing 0.2—1.0% by weight non-ionic or anionic surface active agent. As a supplement to the above solution a mixture of 5% by weight calcium oxide and 0.1—2% by weight solution of a surface active agent (polyoxyethylenealkyl ether) was used as required. A hair-lotion II consisted of a solution of 0.2—1% by weight non-ionic or anionic surface active agent in which were dissolved 1—5% by weight of various kinds of soluble metallic salts. Thus, hair-waving or hair-straightening and at the same time, hair-colouring were effected, apparently by substituting other metallic ions for alkaline ions to form chelate

compounds of hair protein, keratin, by means of hair-lotions I and II. In this process, heat was not applied.

- 5 The metal compound may be a magnesium, calcium, zinc, cadmium, barium, strontium, aluminium, cobalt, tin, manganese, iron, chromium, silver, gold, platinum, nickel or copper compound.

- 10 Depending on the circumstances, a suitable quantity of perfumes may be added. It is also possible to make hair cream by adding methyl cellulose, fluid paraffin, lanolin or a wax.

- 15 While the process above has largely described the use of water soluble metallic salts there may also be used according to the invention calcium oxide which produces calcium hydroxide through its reaction with water, even though the solubility of calcium hydroxide is as low as some 0.12% by weight. During this process, alkali metal ions of chelate compounds of alkali metals of hair protein are replaced to produce chelate compounds between calcium and the hair protein.

- 20 Calcium ions in the hair-lotion are consumed, and calcium hydroxide is gradually dissolved into the liquid to supplement calcium by substituting ions of alkali metals in the chelate compounds of hair protein. The alkali metal ions thus released appear to react with hair protein to form new chelate compounds. The hair is thus waved or straightened through the repetition of this reaction. In this sense, the presence of calcium hydroxide or other soluble calcium compound is required. Therefore, the hair lotion of this invention is to be shaken before being used.

- 25 Instead of preparing the first aqueous solution by incorporating ammonium or an alkali metal hydroxide directly in the solution, the solution may be prepared by the incorporation of an alkali metal or ammonium salt, such as a carbonate, bicarbonate, oxalate, tartate, phosphate, carboxylate or citrate and calcium oxide (which reacts to give calcium hydroxide) in the solution. Chemical reaction causes the formation of insoluble calcium carbonate, calcium bicarbonate, calcium oxalate, calcium tartrate, calcium citrate, calcium phosphate or a calcium carboxylate and the formation of ammonium or alkali metal hydroxide.

- 30 Furthermore, by producing a metallic chelate compound which is soluble in alkaline solution e.g. by mixing a solution of a non-ionic or anionic surface active agent and enzymes, proteins, peptides, or amino acids and a solution containing water soluble metallic salts, and adjusting the hydrogen ion concentration of the resulting mixture to pH 7—12, the simple process, using only one kind of mixture in either lotion form or in cream, for hair waving or hair straightening, and colouring may be obtained. Useful proteins and enzymes are casein, peptone, amylase, protease or lipase.

- 65 In order readily to produce chelate com-

pounds of iron, nickel, cobalt or chromium in hair protein keratin, first of all, a chelate compound of an enzyme or protein with iron, nickel, cobalt or chromium from the solution of soluble metallic salt such as iron chloride, nickel sulphate, cobalt chloride and chromium sulphate is produced, and thereafter the said chelate compounds are used with alkaline solution, instead of the metallic ion solution of iron, nickel, cobalt or chromium.

The following are some practical examples of this invention and these examples, as in the remainder of the specification, all proportions are by weight unless otherwise stated.

EXAMPLE I

Three grams of potassium hydroxide and three grams of calcium oxide are added to 100 ml of water, which is shaken in order to dissolve the calcium hydroxide which is formed and to keep the rest of the calcium oxide in suspension so that the whole liquid presents a milky appearance. Into this liquid is dissolved 0.2%—1% non-ionic surface active agent to produce hair-lotion, to which a suitable quantity of perfume is added if need be. The process of hair-waving with this lotion consists of coating the hair with a small quantity of water, winding it on a rod, then coating it with the hair-lotion by means of a brush, and leaving it for fifteen minutes, after which the rod is removed, and the hair is washed in warm water. Then it is wiped with a dry towel and air dried, after which hair-oil is applied and styled by combing. Crinkly hair can easily be straightened by treating with this hair-lotion.

EXAMPLE 2

2% casein is dissolved in 100 ml of the alkaline solution containing 2% non-ionic surface active agent and to the resultant solution is added 1% cobaltous chloride, and sodium hydroxide is further added to adjust the pH of the solution to over 7. Thus a hair-lotion, which, when mixed with 7.5% polyoxyethylene cetyl alcohol ether and a suitable quantity of perfume and pigment, will turn into hair-cream to be used for waving, straightening and colouring the hair is obtained.

WHAT I CLAIM IS:—

1. A process for effecting at least one of the operations of hair waving, hair straightening and hair colouring, in which the hair is contacted with a composition made by mixing:—

(I) a source of metal ions produced from (a) (i) a watersoluble salt of Mg, Ca, Zn, Cd, Ba, Sr, Al, Co, Sn, Mn, Fe, Cr, Ag, Au, Pt, Ni or Cu with (ii) an alkaline solution of an enzyme, a protein, a peptide or an amino acid or (b) a suspension of calcium oxide, together with

(II) 0.1—2% by weight of non-ionic or anionic active agent;

compounds of hair protein, keratin, by means of hair-lotions I and II. In this process, heat was not applied.

5 The metal compound may be a magnesium, calcium, zinc, cadmium, barium, strontium, aluminium, cobalt, tin, manganese, iron, chromium, silver, gold, platinum, nickel or copper compound.

10 Depending on the circumstances, a suitable quantity of perfumes may be added. It is also possible to make hair cream by adding methyl cellulose, fluid paraffin, lanolin or a wax.

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45

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70 The following are some practical examples of this invention and these examples, as in the remainder of the specification, all proportions are by weight unless otherwise stated.

EXAMPLE I

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85 90 95 100

EXAMPLE 2

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105 110

WHAT I CLAIM IS:—

1. A process for effecting at least one of the operations of hair waving, hair straightening and hair colouring, in which the hair is contacted with a composition made by mixing:—

115

(I) a source of metal ions produced from (a) (i) a watersoluble salt of Mg, Ca, Zn, Cd, Ba, Sr, Al, Co, Sn, Mn, Fe, Cr, Ag, Au, Pt, Ni or Cu with (ii) an alkaline solution of an enzyme, a protein, a peptide or an amino acid or (b) a suspension of calcium oxide, together with

120 125

(II) 0.1—2% by weight of non-ionic or anionic active agent;



said composition being held at pH 7 to 12
by

(III) an organic amine or a source of alkali
metal ion or ammonium ion also yielding
5 hydroxyl ions in aqueous solution.

2. A process as claimed in claim 1 in which
the composition is constituted as a hair lotion.

3. A process as claimed in claim 1 in which
the composition is constituted as a hair cream.

4. A process as claimed in claim 1 and 10
substantially as hereinbefore described with
reference to the Examples 1 to 3.

MARKS & CLERK.

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