

- [54] **HAIR SETTING PROCESS**
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2,991,790	7/1961	Bonilla.....	132/7
3,086,534	4/1965	Gorter et al.	132/7

FOREIGN PATENTS OR APPLICATIONS

859,276	1/1961	Great Britain	424/62
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[57] **ABSTRACT**

A process for curling or straightening hair which has been previously oxidized comprising applying a solution of a polyvalent metal ion chelator thereto and subjecting the hair to a longitudinal stress.

17 Claims, No Drawings

- [56] **References Cited**
- UNITED STATES PATENTS**
- 2,990,336 6/1961 Martin 132/7

HAIR SETTING PROCESS

BACKGROUND OF THE INVENTION

This invention relates to a process for deforming hair. More particularly, the invention discloses processes for curling or straightening human hair which has been oxidized by chemical oxidizing agents or by sunlight comprising contacting said hair with a solution of a chelating agent such as the ethylenediaminetetraacetates, nitrilotriacetates and the like, and subjecting the hair to a longitudinal stress.

It is recognized in the art that hair which has been oxidized with hydrogen peroxide, e.g., in a bleaching operation, is relatively easy to set in a more-or-less permanent configuration by simply moistening the hair and subjecting it to a longitudinal stress. Likewise, hair which has been subjected to prolonged exposure to the ultraviolet rays of the sun is also particularly susceptible to a water set. Still a third oxidative process for setting hair which employs certain monopersulfate oxidants is described in the concurrently filed application of Zeffren and Turner, entitled "Hair Setting Process," Ser. No. 294,053, filed Oct. 2, 1972. Presumably, oxidizing agents such as the common peroxide hair bleaches, sunlight and the monopersulfate salts, disrupt chemical bonds in keratin fibers with a net effect similar to that when hair is contacted with the well-known thioglycolate/hydrogen peroxide cold waving solutions. Hair in which the keratin molecules are oxidatively disrupted is thereby rendered deformable and can then be either curled or straightened, as desired, by simply moistening the hair, subjecting it to a longitudinal stress and allowing it to dry in a stressed configuration.

While hair which has been oxidized by any of the foregoing means readily accepts a water set, this effect is lost relatively quickly. It has now been found that polyvalent metal ions, especially calcium and magnesium ions present in ordinary tap water used to set the oxidized hair, have a cumulative adverse effect on its setting properties. That is to say, oxidized hair rinsed with tap water absorbs calcium and/or magnesium ions which combine with the anionic functional groups formed in the oxidized keratin such that the hair can no longer be set by simple treatment with water. After sufficient metal ions are absorbed, the hair must again be treated with the oxidizing agent to restore its deformable properties.

By the present invention it has been found that oxidized hair which has absorbed sufficient polyvalent metal ions that it no longer retains an acceptable water set can be contacted with a solution of a metal ion chelating agent to render it substantially free of polyvalent metal ions. Hair treated with the chelator is once again readily deformed by applying a longitudinal stress to the wet hair and allowing it to dry in the stressed configuration.

U.S. Pat. No. 2,990,832 teaches the use of chelating agents in thioglycolate hair waving processes but does not teach that oxidized hair can be treated with a solution of a chelator and subjected to a longitudinal stress to achieve waving in the manner of the present invention.

SUMMARY OF THE INVENTION

The instant invention encompasses a process for deforming hair which has been subjected to oxidizing conditions comprising: (1) contacting said hair with an

aqueous solution of a metal ion chelator at a pH in the range from about 5 to about 10; (2) subjecting the moist hair to a longitudinal stress; and (3) drying the hair in the stressed configuration. By this means the hair can either be straightened, for example, by fashioning it into a substantially straight configuration, or curled by winding the hair on a mandrel. The present treatment finds particular utility in maintaining or reestablishing the deformable properties of hair which has been oxidized using a monopersulfate oxidant in the manner of Zeffren, et al., above. Thus the invention encompasses an improvement in the process for setting hair comprising the steps of: (1) contacting said hair with an aqueous solution of an oxidant of the formula $MHSO_5$, wherein M is an alkali metal cation, at a pH of from about 5 to about 11 for a period of time of from about 1 to about 30 minutes; (2) removing the monopersulfate oxidant solution from contact with the hair; (3) setting the hair by moistening it with water and subjecting it to a longitudinal stress; (4) thereafter drying the hair in the stressed configuration, the improvement which comprises, in setting step (3), moistening the hair with an aqueous solution of a chelating agent of the type hereinafter detailed.

DETAILED DESCRIPTION OF THE INVENTION

The chelating agents employed in the present process include those compounds having polydentate ligands capable of chelating polyvalent metal ions, especially bivalent ions such as calcium and magnesium ions, as well as trivalent ions such as ferric ion. Of course, to be effective in the present process such chelating agents must be capable of complexing with the metal ions more strongly than do the ionic groups present in the oxidized hair being treated. That is to say, the stability constant of the metal ion-chelator couple must be greater than the stability constant of the metal ion-oxidized keratin stability constant for the metal ion to be effectively removed from the hair by the chelator. It is well recognized that the stability constants of metal ion-chelator couples increase with the increasing number of ligands present in the chelator molecule. That is to say, a bidentate chelator will have a lower stability constant with a given metal ion than will a tridentate chelator. A tridentate chelator will, in turn, have a lower stability constant than a tetradentate chelator, and so forth.

While polyvalent metal cations appear to complex with oxidized hair keratin sufficiently strongly to preclude removal by rinsing or shampooing, it has been found that such cations can be satisfactorily removed from the hair by the application of an aqueous solution of chelating agents containing bidentate ligands, tridentate ligands, tetradentate ligands, pentadentate ligands, and hexadentate ligands. The oxidized hair which has had the polyvalent metal ions removed is again susceptible to being deformed by simply moistening the hair, subjecting it to a longitudinal stress and allowing the hair to dry in the stressed configuration.

Suitable chelating agents for removing the polyvalent metal ions from oxidized hair include a variety of water-soluble chelators, including bidentate chelating agents such as acetylacetonate, salicylaldehyde, and dimethylglyoxime. Tridentate chelating agents useful herein include iminodiacetic acid and its water-soluble salts, and the o-azeophenoxide anion. Tetradentate chelating agents useful herein include triethylene-

tetramine, β',β'' -triaminotriethylamine and bisacetylacetone-ethylenediamine. Pentadentate chelating agents useful herein include ethylenediaminetriacetic acid. Hexadentate chelating agents useful herein include ethylenediaminetetraacetic acid, 1,8-bis(salicylideneamino)-3,6-dithiaoctane and mellitic acid. Any of the water-soluble salts, especially the alkali metal salts, i.e., Li^+ , Na^+ , K^+ , Ca^+ and Rb^+ salts, of the foregoing anionic chelating agents can be used herein. While any of the foregoing chelating agents can be employed in the present process, the water-soluble alkali metal salts of nitrilotriacetic acid, ethylenediaminetetraacetic acid and mellitic acid are preferred for this use. The sodium salts, including the monosodium, disodium, trisodium, and tetrasodium salts of ethylenediaminetetraacetic acid are especially preferred in instant process from the standpoint of economy and commercial availability.

The foregoing chelating agents are employed in the present process as aqueous solutions containing at least 1 percent, preferably about 2 percent to about 20 percent, by weight of said chelating agents. The pH of the solutions can be in the range from about 5 to about 10 and are preferably about 7. Solution pH's can be adjusted by adding acid or base and maintained with standard buffers, as desired.

The aqueous solutions of chelating agent used herein can contain additional components which serve to aid in their application. For example, various gelling agents such as silica gel, high molecular weight polyoxyethylenes, carboxymethylcellulose, magnesium aluminum silicate (Veegum), and the like can be used in such solutions as thickeners for the convenience of the user. Various anionic, non-ionic and ampholytic surfactants, such as the alkylbenzene sulfates and sulfonates, the polyoxyethylene condensates of alkyl phenols, as well as sodium and potassium soaps, can be present in the compositions to improve the rinseability thereof. Perfumes, emollients, conditioning aids such as various silicone and protein derivatives and the like can be copresent in the compositions to provide a pleasing cosmetic and aesthetic aspect thereto. While it is to be understood that all manner of such anionic, nonionic and ampholytic auxiliary materials commonly used to enhance the cosmetic aspect of preparations used on hair can be employed the present compositions since all such materials are stable and compatible with the chelating agents at the intended pH use range herein, hydrocarbyl cationics and zwitterionics are preferably avoided. That is to say, while cationics and zwitterionics are compatible with the chelating agent herein, they apparently interact with the anionic groups in the oxidized hair and interfere with the establishment and maintenance of an optimal set. Accordingly, positively charged materials such as the alkyl quaternary ammonium hair softeners are to be avoided in the processes and compositions herein.

In the process of curling hair by applying a chelator solution to oxidized hair, a longitudinal stress is applied to the wet hair to achieve deformation and the desired hair set. The hair to be curled is wound on a mandrel and held in place during the curling operation. The types of mandrels used for this purpose are those well known in the art, including the spiral rod and the Croquignole curler. When the spiral rod mandrel is employed, the hair on the head is divided into multiple sections, usually four to about eight. The sections are

then subdivided into small squares, ca. 1 in. \times 1 in. Starting at the proximal end, the hair in each block is then wound on the mandrel in the shape of a helix. The distal ends are held by a string, or clip, or other holding device.

When the Croquignole curler is employed to curl hair, the process is somewhat different. The hair on the head is sectioned into blocks ranging in size from about $\frac{1}{2}$ to 1 inch \times 2 to 3 inches, depending on the final hair style envisioned. The hair in each block is then combed and the distal ends are placed centrally on the mandrel, which is then wound and secured in place by means of a rubber band or other clipping device. In both the spiral rod and Croquignole techniques, end papers can be used to facilitate collecting the distal ends.

In still a third technique, which results in the formation of relatively large, loose curls, the hair is wound into a generally circular configuration without special winding devices and set into place by means of hair pins or clips. These so-called pin curls have a very large diameter and result in a very loose curl. By proper selection of the means whereby the longitudinal stress is applied to the hair, curls of varying degrees of tightness can be achieved. Spiral winding results in the formation of relatively tight curls; Croquignole winding yields somewhat looser curls; while pin curls are usually quite loose.

In one method of curling hair by means of a solution of a chelating agent, the oxidized hair is first wound as described above, depending on the type of curl desired. The winding can be done with dry hair, moistened hair or hair moistened with the chelator solution. Preferably, the hair is shampooed and rinsed prior to winding. After winding, the hair is thoroughly saturated with the solution of the chelating agent. Additional chelator solution can be applied from time-to-time during the curling to keep the hair thoroughly saturated. The solution is allowed to remain in place for from about 1 minute to about 30 minutes, depending on the strength of the chelator solution, after which time the hair is dried in the stressed configuration. The treatment time will depend to a certain extent on the degree of oxidation and the metal ion content of the hair. The time necessary to achieve a curl can be determined by the user prior to application to the head by means of a test curl. Generally, hair which has been mildly oxidized will require a curling time of about 10 minutes when a 2 percent solution of the chelator salt is employed. Damaged hair having a high metal ion content will require from about 10 to about 30 minutes to achieve a curl when a 2 percent chelator solution is employed. For most purposes, it is preferable that the hair be in contact with the solution of chelator for a period of time from about 5 minutes to about 15 minutes to drying.

In an alternate procedure, the oxidized hair in its natural configuration is contacted with an aqueous solution of the chelating agent, and is kept thoroughly saturated for from 1 to about 30 minutes. During this time the hair is set as desired by the user. This procedure is preferred over moistening the pre-set hair with the chelator solution inasmuch as the hair can be more thoroughly saturated and the treatment is therefore more effective. Following treatment with the chelator and setting, the hair is dried in the new configuration.

The foregoing procedures can be used with hair which has been oxidized by any oxidant, e.g., sunlight, hydrogen peroxide, monopersulfate salts, etc., to re-

store its ability to retain a water set, since all such oxidized hair eventually absorbs sufficient polyvalent metal ions to prevent maintenance of an acceptable set.

In a preferred process herein the hair being treated with the chelator solution has been oxidized with a monopersulfate oxidant under the conditions of pH, time and temperature as recited in the concurrently filed application of Zeffren and Turner, above. In one method for deforming hair using monopersulfate salts of the formula $MHSO_5$, wherein M is an alkali metal cation, the hair is contacted with a 1 to 20 percent by weight aqueous solution of the $MHSO_5$ salt at a pH of from about 5 to 11 for a period of time from about 1 to 30 minutes. The hair is rinsed with water and then subjected to a longitudinal stress to set the hair in any desired configuration. It will be recognized that, in the setting step, the hair will absorb metal cations from the rinse water and these cations interfere with the ability of the hair to achieve an optimal set. Furthermore, polyvalent metal ions can be inadvertently introduced into the hair from impurities in the monopersulfate solution or from shampoo compositions containing the monopersulfates. Whatever the source of the undesirable polyvalent metal ions, they can be readily removed by rinsing the hair with the chelators herein. Of course, it is desirable that the chelator rinse be used in a latter step of the process to remove metal ions, whatever their source. It has now been found that metal ion uptake by the hair can be substantially avoided when, in the setting step of the monopersulfate setting process, hair is contacted with a chelator solution under the conditions of time, pH, etc., noted hereinabove. This chelator treatment provides a substantial improvement in the setting properties of hair oxidized by the monopersulfate.

The instant process of deforming, i.e., curling or straightening hair, can be carried out over a wide temperature range, i.e., from about 0°F. to about 150°F. Of course, use of the higher temperatures within the range, for example, by means of a hair dryer or electrically heated hair curler, will increase the rate of curling. Use of colder temperatures within the range will reduce the rate. Hair setting processes are generally carried out at room temperatures of from about 65°F. to about 90°F. and this temperature range is preferred in the instant process.

The following examples illustrate the hair curling processes of this invention but are not intended to be limiting thereof.

EXAMPLE I

Human hair switches (2.2 g. per switch) were oxidized by treatment with an aqueous formulation consisting of 10 percent by weight OXONE (DuPont's commercial material comprising 41.5% $KHSO_5$, the balance being equal proportions of K_2SO_4 and $KHSO_4$), 5.5 percent by weight sodium carbonate, 1.5 percent by weight sodium alkyl glycerol ether sulfate, and 2.3 percent by weight magnesium aluminum silicate, the balance being water. The pH of the formulation was ca. 8.8. The formulation was worked evenly through the hair. The hair was exposed to the treatment for a 10 minute period at room temperature (72°F).

Following treatment with the foregoing buffered OXONE solution, the hair was shampooed with a commercial shampoo composition and thoroughly rinsed

with distilled water. The treated hair had a silky-slick feel. Hair treated in this fashion was wound in Croquignole fashion on a mandrel (commercial hair curler) and allowed to dry at room temperature over a period of about 3 hours. Following this, the hair was removed from the mandrel and was found to be in a substantially curled configuration. Curl stability was assessed as percent curl loss by suspending the curled hair in a closed room at 80°F, 80 percent relative humidity for 2 hours. After this time, the curl had unfolded about 74 percent of the total length (i.e., 74 percent curl loss) as compared with a curl from unoxidized hair which unfolded about 85 percent in the same time.

Curled hair oxidized in the foregoing manner, shampooed, and rinsed with tap water had a silky-slick feel at the beginning of the rinse, but this feel disappeared with continued rinsing. Hair treated in this fashion was wound in Croquignole fashion on a mandrel and allowed to dry at room temperature over a period of 3 hours. Curl stability was then assessed in the foregoing manner at 80°F, 89% RH, for 2 hours, after which curl had unfolded to about 82 percent of the total hair length.

Curled hair oxidized with the OXONE, shampooed, and rinsed with tap water was soaked in ca. 100 mls. of a solution comprising 5 g. of disodium ethylenediaminetetraacetate dissolved in 95 g. of water at pH 8.0 (adjusted with NaOH). The soaking took place at room temperature for a period of about 10 minutes. Following this treatment, the hair was removed from the ethylenediaminetetraacetate solution and was found to have regained the silky-slick feel. The moistened hair was wound in Croquignole fashion on a mandrel and allowed to dry at room temperature over a period of about 3 hours. Following this, the hair was removed from the mandrel and tested for curl stability at 80% R.H. The hair was again found to have a curl stability substantially that of hair which had been freshly oxidized by the OXONE solution.

In the foregoing procedure, the disodium ethylenediaminetetraacetate is replaced by an equivalent amount of hexasodium mellitate, disodium nitrilotriacetate, trisodium nitrilotriacetate, and tetrasodium ethylenediaminetetraacetate, respectively, and equivalent results are secured in that polyvalent metal ions in the oxidized hair are removed and the hair can be reset to provide a stable, curled configuration.

EXAMPLE II

Brown human hair switches are oxidatively bleached by immersion in a solution comprising 5 percent hydrogen peroxide, 2 percent ammonium persulfate, and 5 percent of a 28 percent by weight aqueous ammonia solution, the balance of the bleaching solution comprising water. After immersion in the solution for about 20 minutes, the brown hair is substantially bleached to a yellow color. The hair is removed and rinsed in tap water and has a silky-slick feel. The moistened hair is wound in Croquignole fashion on a mandrel and allowed to dry at room temperature over a period of about three hours. Following this, the hair is removed from the mandrel and is in a curled configuration. The hair is placed in a chamber at 80 percent relative humidity. After about two hours in the chamber, the hair has a percent curl loss of only about 60 percent indicating good curl stability.

The bleached, curled hair is subjected to a succession of shampooings and rinses employing a commercial shampoo containing anionic surfactants and using tap water (hardness ca. 9 gr/gal) for the rinse following each shampooing. After a series of ten such treatments, the hair loses substantially all the silky-slick feel. At this point, the hair is again moistened, wound in Croquignole fashion on a mandrel, and allowed to dry at room temperature. After removal from the mandrel, the percent curl loss, as measured in the 80% R.H. chamber, is about 85 percent i.e., essentially that of unoxidized hair.

Hair treated in the foregoing manner, and which no longer has substantial curl stability, is soaked in 25 ml. of a 10 percent by weight aqueous solution of disodium ethylenediaminetetraacetate (solution pH 8) at 70°F. for 10 minutes. Following this treatment, the hair is removed from the ethylenediaminetetraacetate solution; at this point, the hair again has the silky-slick feel of wet, freshly oxidized hair. The moistened hair is wound in Croquignole fashion on a mandrel and allowed to dry at room temperature over a period of about two hours. Following this, the hair is removed from the mandrel and placed in the 80% R.H. chamber. Curl stability of the hair treated with the chelator solution is found to be restored to that of freshly bleached hair.

In the foregoing procedure, the disodium ethylenediaminetetraacetate is replaced by an equivalent amount of potassium acetylacetonate, ammonium dimethylglyoxime, triethanolammonium iminodiacetic acid, sodium o-azophenoxide, triethylenetetraamine, lithium nitrilotriacetate, monosodium ethylenediaminetriacetate, disodium ethylenediaminetriacetate, and dicesium mellitate, respectively, and equivalent results are secured.

EXAMPLE III

Switches of dark brown human hair are exposed to natural sunlight for 96 hours, after which time the dark brown coloration is substantially lightened to a medium brown-blonde stage. The hair is then rinsed in tap water and the moistened hair wound in Croquignole fashion on a mandrel and allowed to dry at room temperature over a period of 3 hours. Following this, the hair is removed from the mandrel and is found to be in a curled configuration. The curl stability of the hair is assessed by placing the curled hair in a chamber at 80 percent relative humidity. After about 2 hours in the chamber, the hair loses only about 60 percent of its curl, indicating good curl stability.

The sun bleached, curled hair is subjected to a succession of shampooings and rinsings employing a commercial shampoo containing anionic surfactants using tap water (hardness ca. 9 gr./gal.) for the rinse following each shampooing. After a series of 10 such treatments, the hair is again moistened, wound in Croquignole fashion on a mandrel and allowed to dry at room temperature. After removal from the mandrel, the curl stability, as measured in the 80 percent relative humidity chamber, is substantially less than that of the freshly sun bleached hair.

Hair treated in the foregoing manner, and which has lost substantially all of its deformable characteristics, is wound on a ¼ inch mandrel and soaked with a 5 percent by weight aqueous solution of disodium ethylenediaminetetraacetate for 10 minutes. Following this treatment, the hair is allowed to dry at room tempera-

ture over a period of two hours. Following this, the hair is removed from the mandrel and placed in the relative humidity chamber. The curl stability of the hair is found to be substantially restored to that of freshly sun bleached hair.

The foregoing illustrates that the use of all manner of metal ion chelators capable of chelating polyvalent metal ions can be used to reestablish the deformable properties of hair which has been oxidized by peroxide, sunlight or monopersulfate salts. As noted hereinabove, the metal ion chelators find particular use with the monopersulfate oxidizing agents as a means for setting hair inasmuch as the monopersulfates have been found to oxidize the keratin of the hair sufficiently to allow the hair to be readily deformed without alteration of the natural melanin coloration.

While the foregoing procedures illustrate the use of metal ion chelators for reestablishing the deformable properties of oxidized hair once these properties have been substantially lost due to complexation of metal ions by the hair, the following example illustrates the use of the present process for maintaining the deformable properties of oxidized hair.

EXAMPLE IV

Hair switches are oxidized by soaking in a solution comprising 10 percent by weight commercial OXONE, 2 percent by weight sodium phosphate, 5 percent by weight sodium hydrogen phosphate (pH buffer), the balance of the composition comprising water. The pH of the solution is about 8.5. Soaking is carried out over a period of 30 minutes at room temperature. At this point, the hair has a silky-slick feel.

Following treatment with the foregoing OXONE solution, the hair is thoroughly rinsed with a 5 percent by weight aqueous solution of disodium ethylenediaminetetraacetate. The hair is then wound in Croquignole fashion on a mandrel and allowed to dry at room temperature over a period of about 2 hours. Following this, the hair is removed from the mandrel and is found to be in a highly curled configuration. The curled hair is then subjected to a succession of shampooings, employing a commercial shampoo containing anionic surfactants. Following each shampooing, the hair is rinsed with a 4 percent by weight aqueous solution of disodium ethylenediaminetetraacetate. This shampooing-chelator rinsing treatment is repeated 10 times, in succession. Following the final shampooing and ethylenediaminetetraacetate rinse, the hair is found to still have the silky-slick feel. At this point, the hair, which is still moist from the ethylenediaminetetraacetate rinse, is wound in Croquignole fashion on a mandrel and allowed to dry at room temperature. After removal from the mandrel, the curl stability of the hair, as measured in the 80% R.H. chamber, is substantially that of hair freshly oxidized with the OXONE solution, thereby demonstrating the ability of the chelator solution to maintain the deformable properties of the oxidized hair.

In the foregoing procedure, the disodium ethylenediaminetetraacetate is replaced by an equivalent amount of dipotassium ethylenediaminetetraacetate, dilithium ethylenediaminetetraacetate, diammonium ethylenediaminetetraacetate, bis-(triethanolammonium) ethylenediaminetetraacetate, ethanolammonium ethylenediaminetetraacetate, and tetrakis(diethanolam-

monium) ethylenediaminetetraacetate, respectively, and equivalent results are secured.

The chelators herein are preferably used to curl or straighten hair which has been oxidized by means of monopersulfate oxidants in the manner of Zeffren and Turner, above. The monopersulfate salts, in turn, are preferably applied to clean hair. That is to say, hair which has had its coating of dirt and excess lipids removed by shampooing is more susceptible to oxidation by the monopersulfate solution and improved treatments are thereby secured. In a preferred method of oxidizing hair, the shampoo and monopersulfate solution are applied to hair concurrently. Accordingly, shampoo compositions containing the monopersulfate oxidizing agent and having a pH from about 5 to about 11 can be prepared and applied to the hair to cleanse and oxidize said hair simultaneously. It has now been found that following treatment with the monopersulfate-containing shampoo, the hair can be subjected to a final rinse with water containing a chelator of the type disclosed herein and the wet hair subjected to a longitudinal stress in the manner hereinabove described and allowed to dry. Hair treated in this manner is found to have improved setting properties inasmuch as polyvalent metal ions are removed from the oxidized hair by the chelator.

It is known that compounds of the formula $MHSO_5$ decompose relatively quickly on contact with water. In contrast, the dry monopersulfates are stable indefinitely. In order to provide the user of shampoo compositions containing a monopersulfate oxidant with fresh oxidant, it is necessary to package the shampoo and dry monopersulfate separately. In this way, the user can mix the ingredients just prior to application to the hair. A separately packaged portion of chelator is provided which the shampoo user can mix with ordinary tap water and use as a final rinse prior to subjecting the hair to a longitudinal stress and drying (i.e., in the setting step). Accordingly, the present invention encompasses hair deforming compositions in kit form, the kit comprising a separately packaged, aqueous shampoo composition, a separately packaged portion of a monopersulfate salt of the formula $MHSO_5$, wherein M is an alkali metal cation and a separately packaged portion of a chelator of the type disclosed herein. The kit can contain sufficient shampoo, monopersulfate salt and chelator to provide multiple treatments, or can provide sufficient material for a single treatment. A kit suitable for single treatment use comprises from about 1.0 oz. to about 7.0 oz. of a separately packaged, water-based shampoo of the type hereinafter disclosed, from about 0.01 oz. to about 1.5 oz., preferably about 0.02 oz. to about 0.2 oz., of a separately packaged monopersulfate salt of the type disclosed herein and from about 0.1 oz. to about 20oz., preferably 0.5 oz. to about 10 oz. of a chelator salt. Kits of the foregoing size provide a sufficient volume of material for thorough saturation of the hair with the monopersulfate shampoo, and sufficient chelator for ca. 12 oz. of the final rinse solution. Larger kits which are integral multiples of the single use kits can be provided. With such multiple-use kits, it is convenient to provide a single, large volume of shampoo and incremental packets of monopersulfate salt and chelator. For use, an aliquot of shampoo is admixed with a single, pre-measured package of the monopersulfate.

As noted hereinabove, the chelators herein can be used to reestablish the deformable properties of oxidized hair which has absorbed metal ions without the need for reoxidizing the hair. Accordingly, a kit which comprises a single-use quantity of separately packaged monopersulfate salt and shampoo, and multiple packets of chelator (i.e., about 15 packets) can be provided. Once the hair is oxidized, the user of such a kit can shampoo the hair as needed and reestablish its deformable characteristics by means of a final rinse employing a packet of chelator dissolved in ca. 6 to 20 oz. of water.

The kits herein are used by simply admixing the dry monopersulfate and the aqueous shampoo and agitating the mixture until the monopersulfate dissolved. The freshly prepared solution is then applied to the hair, preferably hair which has been pre-moistened with water, in the manner disclosed above. The shampoo is rinsed from the hair; the chelator is dissolved in ca. 6-20 oz. of water and employed as a final rinse; the hair is then set and dried.

The shampoo compositions employed in the kits herein comprise water and a surfactant. The surfactant is, of course, an important ingredient in any shampoo composition, and the choice of surfactant for use in conjunction with the monopersulfate salts in the hair deforming kits herein is critical inasmuch as the cationic alkyl ammonium surfactants commonly employed in shampoo compositions known in the art are not suitable for use in the shampoo compositions of this invention. Such positively charged materials apparently interact with the oxidized hair and interfere with its deformation; accordingly, cationic surfactants are preferably avoided in the shampoos herein. Other than the foregoing limitation, all manner of the well-known anionic, nonionic and amphoteric surfactants can be used in the shampoo-in hair setting compositions herein.

Anionic surfactants such as the well-known, water-soluble salts of alkylbenzene sulfates and sulfonates wherein the alkyl group contains from about 10 to about 18 carbon atoms are useful in the shampoo compositions herein. Likewise, the water-soluble salts of fatty acids containing from about 10 to about 20 carbon atoms, i.e., soap, are suitable for use in monopersulfate shampoo compositions. Nonionic surfactants, such as the alkylated polyoxyethylenes, are useful in the shampoo compositions herein. Alkyl glycerol ether sulfates and sulfonates wherein the alkyl group contains from about 9 to about 21 carbon atoms are an especially preferred class of surfactants for use in the monopersulfate shampoo compositions. Amphoteric surfactants, such as the N-alkylsarcosinates having an alkyl group containing 9 to 20 carbon atoms are also useful herein. It is to be understood that the water-soluble salts of the foregoing anionic detergents, especially the alkali metal salts, are all useful as the surfactant component of the shampoo compositions herein.

Any of the foregoing types of surfactants are well known in the art and are commercially available. Especially preferred as the surfactant component of the monopersulfate shampoo compositions herein are the sodium alkyl glycerol ether sulfates, wherein the alkyl group contains from about 10 to about 18 carbon atoms; the sodium alkyl sulfates, wherein the alkyl group contains from about 10 to about 18 carbon

atoms; sodium N-laurylsarcosinate, and mixtures thereof.

Shampoo compositions employed in the kits herein comprise from about 1 percent to about 20 percent by weight of a member selected from the group consisting of anionic, nonionic, and amphoteric surfactants, the balance of said compositions comprising water and minor portions of non-cationic cosmetic ingredients including anionic thickeners, perfumes, and the like, said shampoos having a pH within the range from about 5 to about 11, preferably from about 7 to about 9.5. As in the case of the monopersulfate curling and straightening compositions, the shampoo compositions preferably contain from about 1 percent to about 20 percent by weight of buffer capable of maintaining the pH within the selected basic range. Any of the pH buffer systems disclosed for use in the waving compositions herein are also suitable in the shampoos. The carbonate-bicarbonate buffer system is especially preferred in the shampoos herein. Accordingly, preferred shampoo compositions herein contain, as an additional component, from about 0.5 percent to about 10 percent weight of sodium carbonate and from about 0.5 percent to about 10 percent by weight of sodium bicarbonate, the total mixed carbonate-bicarbonate present in the composition being within the range of from about 1% to about 20 percent by weight of the total composition. The shampoo base is admixed with the monopersulfate salt to give a shampoo-in composition for deforming hair containing at least about 1 percent, preferably 2 to 10 percent, by weight of the monopersulfate salt.

The separately packaged chelating agent used to rinse the hair in the setting step can be any of the chelators disclosed above. The sodium salts of ethylenediaminetetraacetic acid are preferred for use in the kits herein. The chelator can be separately packaged as an aqueous solution, but is preferably packaged in dry form in a foil or plastic packet to save space. The pre-measured packet is simply dissolved in tap water prior to use.

The following examples are intended to illustrate hair styling kits comprising a shampoo and a monopersulfate salt and their use in deforming hair but are not intended to be limiting thereof.

EXAMPLE V

Component 1	Amount
OXONE	0.08 oz.
Component 2	
Shampoo*	4.0 oz.
Component 3	
Disodium ethylenediaminetetraacetate (EDTA)	1.0 oz.

* Shampoo formulation comprising Sodium Alkyl Glycerol Ether Sulfate (Alkyl = mixed C₁₂-C₁₈) 1.8% by weight; Sodium Carbonate, 1.1% by weight; Sodium Bicarbonate, 5.3% by weight; Veegum, 2.8% by weight; Perfume, 0.65% by weight; Water, balance.

The full packet of OXONE is admixed with the full bottle of shampoo and agitated until all the OXONE dissolves. The pH of the composition is ca. 9. The full packet of EDTA is mixed with 15 oz. tap water and agitated until it dissolves. The pH is ca. 7.

The freshly prepared OXONE-shampoo composition of Example V is used to curl hair as follows. The hair to be curled is first moistened with water and then all of the shampoo composition is applied to the hair and worked in until a lather forms. The composition is then allowed to remain in contact with the hair for 10 min-

utes, after which the hair is thoroughly rinsed with water. The hair is then thoroughly rinsed with the EDTA solution, and lightly towel dried. The moist hair is then wound in Croquignole manner using ¼ in. mandrels and affixed in place at the scalp line. The hair is then allowed to dry at room temperature. After drying, the rollers are removed and the resulting hair is found to have a curl which exhibits enhanced stability (at 80% R.H., 80°F) over oxidized hair rinsed in tap water having a hardness of ca. 8 grains/gal.

In the above composition, the packet of OXONE is replaced by 0.10 oz. packets of LiHSO₅, NaHSO₅, RbHSO₅, and CsHSO₅, respectively, and equivalent hair waving results are secured.

In the above kit, the carbonate-bicarbonate buffer system in the shampoo component is replaced by an equivalent amount of a sodium phosphate-sodium hydrogen phosphate buffer. The resulting shampoo composition has superior lipid removal properties. Hair treated with this shampoo in the foregoing manner maintains a wave even under conditions of high relative humidity.

In the above kit, the EDTA is replaced by an equivalent amount of sodium nitrilotriacetate, hexasodium mellitate, and sodium oxydisuccinate, respectively, and equivalent results are secured.

EXAMPLE VI

Component 1	Amount
OXONE	0.1 oz.
Component 2	
Shampoo*	3.0 oz.
Component 3	
Disodium ethylenediaminetetraacetate (EDTA)	0.8 oz.

* Shampoo formulation comprising Sodium Alkyl Sulfate (alkyl = mixed C₁₂ - C₁₈), 1.0% by weight; Potassium Alkyl Glycerol Ether Sulfate (alkyl = mixed C₁₂ - C₁₈), 1% by weight; Sodium Carbonate, 1.1% by weight; Sodium Bicarbonate, 8% by weight; Sodium Carboxymethylcellulose, 3% by weight; Glycerol, 3% by weight; Perfume, 0.5% by weight; Water, balance.

The full packet of OXONE is admixed with the full bottle of shampoo and agitated until all the OXONE dissolves. The pH of the composition is ca. 8.5. The full packet of EDTA is mixed with 10 oz. tap water and agitated until it dissolves. The pH is ca. 7.

The freshly prepared OXONE-shampoo composition of Example VI is used to straighten hair as follows. The hair to be straightened is first moistened with tap water and all of the shampoo composition is applied to the hair and worked in until a lather forms. The composition is then allowed to remain in contact with the hair for approximately 15 minutes, after which the hair is thoroughly rinsed with the EDTA solution and lightly towel dried. The moist hair is then combed into a substantially straight configuration and affixed in place with clips. The hair is then dried using a standard forced air dryer at a temperature of about 120°F. After drying, the clips are removed and the hair is in the substantially straight configuration. The straightened hair does not curl or kink under conditions of high relative humidity. The natural color of the hair is not substantially affected by this treatment.

In the above composition, the shampoo formulation is modified by replacing the sodium alkyl sulfate by an equivalent amount of sodium alkyl benzene sulfate (alkyl = 13.8 carbon atoms, avg.), sodium N-lauryl sarcosinate, sodium alkyl-glycerol sulfate (alkyl = C₁₀ - C₁₈) and sodium coconut soap, respectively, and equivalent results are secured.

The foregoing examples illustrate the hair deforming kits of the instant invention comprising a separately packaged, water-based, non-cationic shampoo, a separately packaged portion of a water-soluble monopersulfate oxidant, said portion being sufficient to provide a concentration of oxidant in the shampoo of at least about 1 percent by weight and a separately packaged portion of a polyvalent metal ion chelator, said portion being sufficient to provide a sufficient amount (i.e. at least about 6 oz.) of a 1 percent aqueous chelator solution to remove the shampoo from the hair. It is to be recognized that such compositions can contain the ingredients such as tints, emollients, hair control agents, and the like, so long as such additional ingredients are not cationic in nature.

What is claimed is:

1. A process for deforming hair which has been oxidized comprising contacting said hair with an aqueous solution containing at least about 1% by weight of a metal ion chelator, said solution having a pH in the range from about 5 to about 10, and subjecting the moist hair to a longitudinal stress, thereafter drying the hair in the stressed configuration.

2. A process according to claim 1 wherein the chelator is a member selected from the group consisting of the water-soluble alkali metal salts of nitrilotriacetic acid, ethylenediaminetetraacetic acid and mellitic acid.

3. A process according to claim 1 wherein the chelator is a sodium salt of ethylenediaminetetraacetic acid.

4. A process according to claim 1 wherein the chelator solution has a pH of about 7.

5. A process according to claim 1 wherein the chelator solution remains in contact with the hair for a period of from about 1 minute to about 30 minutes.

6. A process according to claim 1 wherein the moist hair treated with the chelator is wound on a mandrel and dried, whereby the hair is curled.

7. A process according to claim 1 wherein the moist hair treated with the chelator is fashioned into a substantially straight configuration and dried, whereby the hair is straightened.

8. In a process for waving hair comprising the steps of: (1) contacting said hair with an aqueous solution of an oxidant of the formula $MHSO_5$ wherein M is an alkali metal cation, at a pH of from about 5 to about 11

for a period of time of from about 1 to about 30 minutes; (2) removing the monopersulfate oxidant solution from contact with the hair; (3) setting the hair by moistening it with water and subjecting it to a longitudinal stress; and (4) thereafter drying the hair in the stressed configuration, the improvement which comprises, in the setting step (3), moistening the hair with an aqueous solution of a polyvalent metal ion chelator at a pH of from about 5 to about 10.

9. A process according to claim 8 wherein the oxidant is OXONE.

10. A process according to claim 8 wherein the chelator is a member selected from the group consisting of the water-soluble alkali metal salts of nitrilotriacetic acid, mellitic acid and ethylenediaminetetraacetic acid.

11. A process according to claim 8 wherein the moist hair treated with the chelator is wound on a mandrel and dried, whereby the hair is curled.

12. A process according to claim 8 wherein the moist hair treated with the chelator is fashioned into a substantially straight configuration and dried, whereby the hair is straightened.

13. A hair deforming composition in kit form comprising: (1) a separately packaged portion of a water-based shampoo, said portion being in sufficient amount to provide at least one shampooing of the hair; (2) a separately packaged portion of a water-soluble monopersulfate oxidant, said portion being sufficient to provide a concentration of oxidant in the shampoo of at least about 1 percent by weight; and (3) a separately packaged portion of a polyvalent metal ion chelator, said portion being sufficient to provide an amount of a 1% aqueous chelator solution sufficient to remove the shampoo from the hair.

14. A kit according to claim 13 wherein the oxidant is OXONE.

15. A kit according to claim 13 wherein the shampoo contains a carbonate-bicarbonate buffer at a pH of from about 7 to about 9.5.

16. A kit according to claim 13 wherein the chelator is a member selected from the group consisting of the water-soluble alkali metal salts of nitrilotriacetic acid, ethylenediaminetetraacetic acid, and mellitic acid.

17. A kit according to claim 13 wherein the chelator is a sodium salt of ethylenediaminetetraacetic acid.

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UNITED STATES PATENT OFFICE
CERTIFICATE OF CORRECTION

Patent No. 3,809,098 Dated May 7, 1974

Inventor(s) John Alfred Anderson

It is certified that error appears in the above-identified patent and that said Letters Patent are hereby corrected as shown below:

Col. 2, line 45, "that" should read --- than ---.

Col. 3, line 17, after "in" and before "instant" should read --- the ---.

Col. 6, line 21, "89% RH" should read --- 80% RH ---.

Col. 12, line 17, "soidum" should read --- sodium ---.

Signed and sealed this 17th day of September 1974.

(SEAL)
Attest:

McCOY M. GIBSON JR.
Attesting Officer

C. MARSHALL DANN
Commissioner of Patents