

1

2

3,257,361 HAIR STRAIGHTENING COMPOSITIONS AND METHOD OF USE

John J. Miskel, East Orange, Philip J. Breivogel, Glen Ridge, and Teresa M. Etlinger, Bloomfield, N.J., assignors to White Laboratories, Inc., Kenilworth, N.J., a corporation of New Jersey

No Drawing. Filed Aug. 15, 1962, Ser. No. 216,957
2 Claims. (Cl. 260—79.5)

This invention relates to compositions comprising a means for altering the physical form and shape of hair (and other keratin-containing substances such as wool) and to processes for making and using such compositions.

The invention sought to be patented in its composition aspect is described as residing in the concept of a salt of a mercapto lower alkyl amine with a film-forming, linear polymer of a water-soluble ethylenically unsaturated monomeric acid.

The invention sought to be patented in its process aspect is described as residing in the concept of using the tangible embodiment of a composition of matter comprising a salt of a mercapto lower alkyl amine with a film-forming, linear, polymer of a water-soluble, ethylenically unsaturated monomeric acid by applying such a composition to human hair or other keratin-containing substance, for the purpose of altering its physical form and shape.

Formulations containing the tangible embodiments of our invention provide, as will be described later, an improved means for straightening hair without the disadvantages and drawbacks inherent in presently known means.

As used herein the term "film-forming, linear polymer of a water-soluble, ethylenically unsaturated monomeric acid" means and includes those polymeric acids characterized as described and which are capable of forming salts with weak bases such as amines. Such a polymer is specifically and preferably represented by polyacrylic acid but also includes for the purpose of illustration, but without limiting the generality of the descriptive term, other polymeric acids such as polymethacrylic acid, poly- α -ethylacrylic acid, polymaleic acid, sulfonated polystyrene, polyvinylsulfuric acid, polyitaconic acid, and the like. In the term "mercapto lower alkyl amine" of which 2-mercaptoethylamine is a preferred species, the phrase lower alkyl includes carbon chains having up to 3 or 4 atoms separating the mercapto group from the amino group. The amino group may be primary, secondary or tertiary, and when tertiary, the amino group may be part of a cyclic amino structure such as a piperidino, pyrrolidino or morpholino. Among mercapto lower alkyl amines contemplated by the foregoing, in addition to the preferred species are N-methyl-2-mercaptoethylamine, N-ethyl-2-mercaptoethylamine, NN-dimethyl-2-mercaptoethylamine, NN-diethyl-2-mercaptoethylamine, N-lauryl-2-mercaptoethylamine, N-(2-mercaptoethyl)morpholine, N-(2-mercaptoethyl)piperidine, N-(2-mercaptoethyl)-pyrrolidine, the corresponding mercapto propyl analogs represented by 2-mercaptopropylamine, 3-mercaptopropylamine and the like.

It is a particular object of this invention to provide a means and method for straightening tightly curled or kinky hair.

The problems incident to the straightening of kinky hair are special and give rise to greater difficulties than the waving of normally straight hair. The methods in use today, both physical and chemical, are generally inconvenient and result in excessive hair damage and depilation. For example, the use of hot irons often results in embrittlement of the hair and, further, the straightening action is far from permanent. Chemical straighteners such as alkalis and mercaptan reducing agents have been em-

ployed but these require a high pH during application which results in excessive hair damage, scalp irritation and hair loss. Moreover, these aforementioned methods generally require the use of mechanical holders to maintain the hair in a stretched condition during or immediately following the straightening operation. Furthermore, neutralization of alkali and reducing agent is a necessary step.

By virtue of our novel salts and compositions containing such salts, we have provided a means for straightening hair that exhibits many advantages over the art. First, the tangible embodiments of our invention are safer from the aspect of hair and scalp damage since they are effectively applied at a pH of 3.0–6.8. It is indeed surprising that hair straightening is efficiently induced at a pH less than 7 since the art tends to contraindicate such action. Mercaptans such as thioglycolic acid and 2-mercaptoethylamine are known to alter the physical form of hair but require a pH in the range of 9 to 10 wherein excessive damage and depilation occurs. Secondly, the straightening is made to occur at about room temperature. In animal tests, our compositions have been found to be free of irritation and sensitization. Indeed, no hair damage has been observed after application to living human hair over a prolonged period of time. As will be shown, our compositions require no mechanical holding device during the straightening period nor do they require any independent neutralization step since they are automatically oxidized by air. Our compositions are indeed designed for self-application and home use. We do not claim novelty in any particular mercapto lower alkyl amine or the use of such alone as a keratin-reducing agent. This property of mercaptans is known in the art. Novelty resides in the combination of such a substance with one of the aforementioned polymers. It is believed that the mercapto lower alkyl amine component alters the physical shape of the hair by reducing the cystine disulfite linkages which are then regenerated by air oxidation. All of this occurs at a pH of 3.0 to 6.8 while the hair is held in a straightened position by the action of the linear film-forming polymer component. Upon drying from an aqueous solution, the salts, for example, 2-mercaptoethylamine polyacrylate, forms a relatively stiff film which holds the hair being treated in place. Thus, the need for a cap, stocking or other mechanical holding device is eliminated.

The ability of our salts and compositions containing such salts to alter the physical form and shape of hair, and in particular to straighten hair, is in part dependent upon the relative concentration of sulfhydryl groups. As is known, the keratin-reducing property is a function of the sulfhydryl group and if this becomes too diluted by a heavy molecule, its efficacy is markedly reduced. For these reasons, the class of mercapto alkyl amines which suitably may be used in somewhat limited as defined. Indeed, of those represented, 2-mercaptoethylamine is the most active because of the high percentage weight of sulfhydryl moiety in the molecule.

The acid polymer component is not limited by molecular structure but principally by its physical properties. As a linear polymer the carboxyl (or other acid group) is repeated in each monomeric unit. Substantially each of these acid groups is neutralized by the mercapto lower alkyl amine. We prefer to employ polyacrylic acid as the polymeric component for reasons of its easy availability and utilizations; however, other equivalent polymers as described heretofore may be so utilized.

In compounding our hair-treating preparations, the salt itself may be employed as the essential active ingredient. This would entail separate preparation and purification of the salt, for example, 2-mercaptoethyl ammonium polyacrylate. Preferably, the salt is generated in situ in the hair-treating formulation by admixing, for exam-

ple, 2-mercaptoethylamine hydrochloride with polyacrylic acid in the presence of alkali at an appropriate pH. Alternatively, a sodium polyacrylate may be employed as described hereinbelow.

The formulations are aqueous in nature and may contain a perfume or other odorant in order to make a more elegant composition. Although compositions containing only 2-mercaptoethylammonium polyacrylate as the active ingredient are more than satisfactory, we prefer to have an excess of polyacrylic acid present in order to maintain the pH 6.5 and to provide a more efficient film which holds the hair in a straightened position during the softening reaction. Other water-soluble or water-dispersible polymers may similarly be employed such as polyvinyl alcohol, polyvinylalcohol-polyvinyl acetate emulsions, methyl acrylate emulsions, sodium carboxymethyl cellulose, hydroxyethyl cellulose, methyl cellulose and the like. These additives to the salt containing compositions provide an enhanced "varnish effect."

In order to make more elegant formulations, various humectants such as sorbitol, glycerine and polyglycols may be added as plasticizers. Also, there may be employed water-dispersible fats such as glyceryl monostearate, polyethylene glycol monostearate, stearyl dimethylbenzylammonium chloride and the like.

The following examples illustrates methods for the manufacture of the novel salts of this invention and the preparation of formulations containing such salts.

EXAMPLE 1.—2-MERCAPTOETHYLAMINE SALT OF POLYACRYLIC ACID

To 37 g. of a 12.5% aqueous solution of sodium polyacrylate (Acrysol GS, Rohm and Haas, Philadelphia, Pa.) add 5.7 g. of mercaptoethylamine hydrochloride. Stir under a blanket of nitrogen until solution is complete. Add water to a total weight of 57.0 g. The solution thus formed has a composition of 13.3% mercaptoethylammonium polyacrylate and 5.1% sodium chloride (pH=6.5 to 6.6). This solution is used in the following formulations.

In order to obtain a crystalline product, the solution is concentrated in vacuo under nitrogen. When solids appear, concentration is halted and the mixture cooled and filtered to yield the salt of this example.

Other mercaptoalkylamine salts of polyacrylic acid are prepared in situ in the same manner as mercaptoethyl ammonium polyacrylate described above. By substituting an equivalent quantity of the mercaptoalkylamine hydrochloride for mercaptoethylamine hydrochloride and utilizing the aforementioned quantities of sodium polyacrylate, the desired product is formed in situ. For example, by employing 8.7 g. of di-(2-mercaptoethyl)amine hydrochloride in the foregoing procedure, the corresponding di-2-mercaptoethyl ammonium polyacrylate is prepared in situ. Similarly, by employing 6.25 g. of N-methyl-N-2-mercaptoethylamine hydrochloride, the analogous salts of this secondary amine is prepared in situ. These and others are utilized in preparing formulations such as described below in the same manner as that set forth for mercaptoethyl ammonium polyacrylate.

Formulation A

A preparation comprising the following ingredients is prepared as described below:

	Weight percent
2-mercaptoethylamine polyacrylate -----	7.0
Glycerine -----	12.7
Polyethylene sorbitan monooleate -----	2.5
Perfume -----	0.25
Distilled water, q.s. to 100%.	

To 52.6 ml. of the solution prepared in Example 1 (13.3% mercaptoethylamine salt of polyacrylic acid) add 12.7 g. of glycerine and 2.5 g. of the monooleate. Under an atmosphere of nitrogen, stir until the mixture be-

comes clear and homogeneous. Dilute to 100 g. with distilled water.

The following formulation allows for the preparation of 2-mercaptoethylammonium polyacrylate in situ:

Formulation B

	Weight percent
2-mercaptoethylamine hydrochloride -----	10.0
Polyacrylic acid (Acrysol A-3, Rohm and Haas) --	70.0
Sodium hydroxide -----	10.0
Perfume oil -----	0.75
Distilled water -----	9.25

Dissolve the sodium hydroxide in the water, warm and bubble nitrogen through the solution. While stirring, add the polyacrylic acid and then cool the solution to 40° C. while maintaining the nitrogen blanket. Add the 2-mercaptoethylamine hydrochloride and then the perfume oil. Stir until homogeneous, cool and transfer into containers preferably under nitrogen atmosphere. The pH of the preparation is about 6.5.

The following formulation utilizes the salt prepared in situ from sodium polyacrylate and 2-mercaptoethylamine hydrochloride:

Formulation C

	Weight percent
Mercaptoethylamine hydrochloride -----	7.0
Sodium polyacrylate (12.5%)—Acrysol GS—Rohm and Haas -----	45.5
Glycerine -----	25.0
Isopropyl palmitate -----	0.5
Distilled water -----	22.0
pH -----	6.4

To a vessel equipped with a stirrer and adapter for maintaining an atmosphere of nitrogen, transfer a sufficient quantity of sodium polyacrylate aqueous solution (12.5%—Acrysol GS—Rohm and Haas) to furnish 45.5 g. of sodium salt. While bubbling nitrogen through the solution, add 7 g. of 2-mercaptoethylamine hydrochloride in 22 ml. of water and stir. Add 0.5 g. of isopropyl palmitate and stir all the while under nitrogen until the reaction medium becomes homogeneous. Add 25 g. of glycerine and continue stirring until homogeneity is obtained. Transfer the formulation into suitable containers under an atmosphere of nitrogen. Avoid any exposure to light or contact with metals.

Any one of the foregoing formulations or an equivalent thereof may be applied to tightly curled or kinky hair for altering its physical form and shape such as straightening. It is preferable to cleanse the hair by shampooing, followed by a rinse and then blocking the hair in sections. The hair straightener formulation is then applied with a stiff brush to each portion of the hair. After the entire head has been covered with the hair straightening compound, it is desirable to comb the hair with a fine comb so as to stretch the hair fibres. The hair straightening action is allowed to work for two or three hours or until the hair feels dry after which time the hair straightener is removed such as by rinsing. Hair straightened in this manner does not revert to its normal state. New growth hair will be tightly curled at the scalp portion and may be straightened as desired. In this case, however, the entire head need not be treated.

In addition to acting as hair straightening compositions, the foregoing formulations may be applied in a manner to produce a wave. These waves in the hair may be produced by winding the hair around supports such as mandrels prior to the application of the formulation. The chemical linkages in the hair are broken down and reformed while the hair is in the broadly curled position, said position resulting in soft waves.

We claim:

1. The amine salt of a mercapto lower alkylamine with

a water soluble film-forming linear polymer of an ethylenically unsaturated monomeric acid.

2. 2-mercaptoethyl ammonium polyacrylate.

References Cited by the Examiner

UNITED STATES PATENTS

2,925,406	2/1960	McCurdy et al.	260—79.5
2,944,942	7/1960	Charle et al.	167—87.1

2,976,216	3/1961	De Mytt	167—87.1
3,070,581	12/1962	Miller	260—79.5

JAMES A. SEIDLECK, *Primary Examiner.*

⁵ FRANK CACCIAPAGLIA, JOSEPH L. SCHOFER,
Examiners.

V. C. CARKE, E. J. SMITH, *Assistant Examiners.*