HAIR TREATING COSMETIC COMPOSITIONS CONTAINING PIPERAZINE BASED CATIONIC POLYMER

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Filed: Nov. 28, 1972

Foreign Application Priority Data
Nov. 29, 1971 Luxembourg...................... 064371

U.S. Cl. .............. 424/70; 8/10.1; 252/DIG. 2; 252/DIG. 3; 252/DIG. 13; 252/DIG. 14; 252/545; 252/546; 252/547; 260/2 BP; 260/2 EP; 260/29.6 HN; 260/268 PL; 424/DIG. 1; 424/DIG. 2; 424/DIG. 4; 424/47; 424/71; 424/78; 424/81; 424/258; 424/311; 424/329; 424/362; 424/365

Int. Cl. .... A61K 7/06

Field of Search ....... 260/2 EP, 2 BP, 268 PL,

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ABSTRACT

Piperazine-based low molecular weight film-forming cationic polymer is employed in a cosmetic hair-conditioner composition comprising a solution of the polymer in amounts of about 0.1-5 weight percent of the composition in a solvent such as water, acidified water, or aqueous alcohol solution.

7 Claims, No Drawings
HAIR TREATING COSMETIC COMPOSITIONS CONTAINING PIPERAZINE BASED CATIONIC POLYMER

The present invention relates to compositions for treating and conditioning the hair. More particularly, the present invention relates to cosmetic compositions for the hair, containing a low molecular weight, film-forming cationic polymer.

Many people's hair, because of its general state or its sensitivity due to relatively frequent periodic treatments such as bleaching, dyeing or permanent waving, often is difficult to comb out and to arrange especially in the case of abundant hair. To varying degrees, such hair often is also dry, dull and rough or lacks vigor and life. Further, such hair is very sensitive to humidity in the air, which explains why hair-sets do not hold for an acceptable length of time. Consequently, the frequency of such treatments, as described above has to be increased, which, in turn increases the above-mentioned disadvantages.

The present invention now makes it possible to substantially limit or avoid these disadvantages by applying to the hair as a conditioner therefor a low molecular weight, film-forming cationic polymer that imparts to the hair an appearance of vigor and radiance.

This polymer can be used alone or as the main component in a hair-dressing lotion, cream, gel, hairsetting lotion hair-setting reinforcer, or alternatively, as an adjuvant in a composition for shampooing, setting, in permanent waving the hair, in a hair dyeing composition, in a cream for treating dry or greasy hair, in an antidandruff lotion or in similar compositions for application to the hair.

The application of the hair conditioner cosmetic composition of this invention results in improving the facility and ease of combing out wet hair and imparts thereto brilliancy, softness and manageability of dry hair. Hair treated with the composition of this invention feels lighter, while at the same time appears thicker and more alive.

The conditioner according to the invention, which acts as a softener and emollient, produces after application of the same to the hair in a shampoo, for example, a more brilliant, more voluminous and more aerated hair without the appearance of static electricity. Further, the cationic polymer of the present invention, provides the additional advantage of avoiding a powdery phenomenon which is frequently observed with numerous conventionally employed polymers. Moreover, the inclusion of the hair conditioner of the present invention in cosmetic compositions for the hair, which compositions also include other components or hair treating agents, does not entail any appreciable reduction of the properties or effectiveness of these other components.

The cationic polymer of the present invention is characterized by the fact that the cationic groups are part of the main polymer chain and that they are essentially derived from bissecondary heterocyclic amines, preferably, piperazine. Structurally, the low molecular weight, filmforming, cationic polymer of the present invention can be represented by the pattern

\[ \text{A} \rightarrow \text{Z} \rightarrow \text{A} \rightarrow \text{Z} \rightarrow \text{A} \rightarrow \text{Z} \]

wherein A represents a radical derived from a heterocycle containing two secondary amine functions and, preferably, the radical

\[ \text{N} \rightarrow \text{N} \rightarrow \text{CON} \rightarrow \text{CON} \]

and Z represents the symbol P or B', each of which independently represents a bivalent radical selected from the group consisting of (i) hydroxypropylene, (ii) alkylene having up to 5 carbon atoms inclusive and interrupted by 1 - 2 members selected from the group consisting of -CONH and

\[ \text{CON} \rightarrow \text{NCO} \rightarrow \text{CON} \rightarrow \text{NCO} \]

and (iii) hydroxy alkylene wherein the alkylene moiety has up to 6 carbon atoms inclusive and is interrupted by a member selected from the group consisting of alkylamine wherein the alkyl moiety has 12 to 16 carbon atoms, benzylamine, oleylamine and oxygen.

Often, polymers of the present invention are those in which A has the meaning given above and B and B' each independently represent a branched or straight chain radical selected from the group consisting of hydroxyalkylene having 1-6 and, preferably, 3 carbon atoms, lower alkylcarbonamide-lower alkylcarbonamide-lower alkyl having 5 to 8 and, preferably 6 to 7 carbon atoms, hydroxypropyl-oxyhydroxypropyl, hydroxypropyl-alkylaminohydroxypropyl wherein the alkyl moiety has 12 to 16 carbon atoms, hydroxypropyl-alkylamino-hydroxypropyl wherein the alkyl moiety has 18 carbon atoms, hydroxypropylpiperazinyl-hydroxypropyl, propionyl-piperazinyl-propionyl and hydroxypropyl-benzylamino-hydroxypropyl.

The polymers of the present invention are more generally strictly alternated, i.e. of the type

\[ \text{A} \rightarrow \text{B} \rightarrow \text{A} \rightarrow \text{B} \rightarrow \text{A} \rightarrow \text{B} \quad \text{(II)} \]

wherein A and B have the meanings given above.

These rigorously alternated polymers, which can be used in the present invention, can be prepared by conventional processes such as by polyaddition or polycondensation of (a) piperazine or a derivative thereof such as, for example, N,N'bis(hydroxyethyl)piperazine, on (b) a bifunctional compound such as

1. alkyl or alkyl-aryl dihalides, such as ethylene chloride, ethylene bromide or bis chloromethyl 1,4-benzene wherein the alkyl moiety has 1-4 carbon atoms, the aryl moiety is phenyl and the halide moiety is chloride or bromide;

2. more complex dihalogen derivatives, such as bis (chloroacetyl) ethylene diamine;

3. bis halohydrins, such as bis 3-chloro 2-hydroxy propyl ether, or other bis chlorohydrins which can be obtained by conventional procedures such as by the condensation of epichlorohydrin on (i) a primary amine, optionally hydroxylated, (ii) on a bissecondary diamine such as piperazine, 4,4'-dipiperidyl, bis 4,4' (N-methylaminophenyl) methane, N,N' dimethylethylene diamine or propylene diamine, (iii) on α,ω-dimercaptoalkane, (iv) on a diol such as ethylene glycol or (v) on a bis phenol such as hydroquinone or "bis phenol A";
3. bis epoxide such as diglycidyl ether or N,N’bis (epoxy-2,3 propyl)piperazine, eventually obtained from the corresponding bis halohydrin;
4. epihalohydrins, such as epichlorohydrin or epibromohydrin;
5. bisepoxide unsaturated derivatives, such as divinyl sulfone, bis maleimide derived from ethylene diamine, or bis acrylamide such as methylene bis acrylamide or piperazine bis acrylamide derived from biprimary or bissecondary diamines;
6. unsaturated acids, such as acrylic or methacrylic acid or their methyl or ethyl esters;
7. diacids, such as succinic, adipic, 2,2,4-trimethyl or 2,4,4-trimethyl adipic or terephthalic acids, acid chlorides or the corresponding methyl or ethyl esters; and
diisocyanates, such as toluene diisocyanate or 2,2,4- or 2,4,4-trimethyl hexamethylene isocyanate.

The polyaddition or polycondensation reaction can be carried out at ambient pressure and at a temperature ranging from about 0°C to 100°C, the molar ratio of (a) to (b), defined above being 0.85 ; 1 to 1.15 ; 1.

Of course, the polymers of the present invention can in certain cases advantageously be prepared in essentially the same way by polycondensation or polyaddition of (a’) N,N’bis (3-chloro 2-hydroxy propyl) piperazine or N,N’bis (2,3-epoxy propyl) piperazine on (b’) a bifunctional compound such as a bissecondary diimide, a diol, a diphenol, a diacid, and a primary amine such as an alkanolamine, alkenylamine, aralkylamine, of which the two hydrogen atoms can be substituted and which behaves as a bifunctional compound, the molar ratio of (a’) to (b’) ranging from 0.85 : 1 to 1.15 : 1.

Further, the cationic polymers of the present invention can also, in certain cases, be of the pattern

\[- \text{A} - \text{Z} - \text{A} - \text{Z} - \text{A} - \text{Z} - \] (I)
in which A represents a radical derived from a heterocycle carrying two secondary amine functions and preferably the radical

\[N - \text{CONH} - \]

and Z represents the symbol B or B’; B and B’ each independently representing a bivalent radical selected from the group consisting of (i) hydroxypropylene, (ii) alkyne having up to 5 carbon atoms inclusive and interrupted by 1-2 members selected from the group consisting of CONH and

\[\text{CO} - \text{CON} - \]

and (iii) hydroxy alkylene wherein the alkylene moiety has up to 6 carbon atoms inclusive and interrupted by a member selected from the group consisting of alkylene wherein the alkyl moiety has 12-16 carbon atoms, benzylamine, oleylamine and oxygen, or (2) quaternary ammonium salt of the cationic polymer in (1) or (3) the oxidation product of the cationic polymer in (1).

Many of the polymers of formula (1) are new compounds.

Among the preferred polymers are those in which A is

\[- \text{N} - \text{CO} - \text{CO} - \text{N} - \]
and B and B', each independently represent a branched or straight chain radical selected from the group consisting of hydroxyalkylene having 1-6 carbon atoms, preferably 3 carbon atoms, lower alkyl-carbonamidomethyl, lower alkyl-carbonamide, lower alkyl having 5-8 and preferably 6-7 carbon atoms, hydroxypropoxyl-oxhydroxypropyl, hydroxypropyl-alkylamino-hydroxypropyl wherein the alkyl moiety has 12-16 carbon atoms, hydroxypropyl-alkenylamino-hydroxypropyl wherein the alkenyl moiety has 18 carbon atoms, hydroxypropyl-piperizylin-hydroxypropyl, propionylpiperizylin-propionyl and hydroxypropyl-benzylamino-hydroxypropyl.

The polymer can be present in amounts of about 0.1 to 5 percent and preferably 0.2 to 3 percent by weight in the various cosmetic compositions of the present invention. These compositions include hair-dressing lotions, creams or gels as the main constituent, shampoo, hair-setting compositions, permanent wave composition or hair dye compositions, etc. The polymers can also be employed as an adjunct in the presence of other components such as anionic, cationic, non-ionic, amphoteric or zwitterionic surfactants, oxidizing agents, foam stabilizers or synergists, sequestrants, superfattating agents, thickeners, softeners, antiseptics, preservatives, dyes, perfumes, germicides or other anionic, cationic, amphoteric or non-ionic polymers.

The polymers can be used in different compositions whose pH varies from 3 to 11, or in the form of inorganic or organic acid salts, or in the form of free base or, alternatively, the quaternary ammonium salt thereof.

The cosmetic compositions for hair according to the present invention can be in the form of an aqueous solution, a dilute alcohol solution, a cream, a paste, a gel, or a powder. They can also contain a conventional aerosol propellant such as dichlorodifluoromethane, monochlorotrifluoromethane, mixtures thereof or other conventional aerosol propellants, and can be packaged in a conventional aerosol bomb under pressure.

The hair shampoo compositions according to the invention are characterized by the fact that they contain, in addition to an anionic, cationic, non-ionic, amphoteric and/or zwitterionic surfactant, one or more compounds of formula I and optionally foam stabilizers or synergists, sequestrants, superfattating agents, thickeners, one or more other cosmetic resins, softeners, dyes, perfumes, antiseptics, preservatives and any other adjuvant usually used in cosmetic compositions.

The polymers of formula I also make it possible to prepare hair-setting lotions, hair-setting reinforcers, treatment creams, hair conditioners, anti-dandruff lotions and other similar compositions characterized by the fact that they contain one or more polymers of formula I having a molecular weight, determined by lowering of the vapor tension, between 1,000 and 15,000, or the quaternary ammonium salt or amine oxide of these polymers, possibly mixed with other cosmetic resins.

EXAMPLES OF PREPARATION OF THE POLYMER

Example 1

Polycondensation of piperazine and epichlorohydrin.

To a solution of 97 g (0.5 mole) of piperazine hexahydrate in 125 g of water, there are added, drop by drop, over a period of 1 hour, 46.3 g (0.5 mole) of epichlorohydrin, with stirring, while maintaining the temperature at 20°C. Stirring is continued for 1 hour at 20°C. The reaction mass is then heated at 90°C-95°C for 2 hours. Over a period of one hour, 0.5 mole of sodium hydroxide is then added in the form of a 40 percent (50 g) NaOH solution. A cloud then appears in the solution. The temperature is maintained at the 90°C-95°C level for 15 minutes more, and the reaction mixture is then cooled with stirring while adding 182 g of water to bring the concentration to 20 percent of dry extract (14.5 percent active material and a 5.5 percent NaCl).

A limp and almost colorless solution is thus obtained, having a viscosity of 2.5 poises, measured at 25°C.

On evaporation of a dilute solution of the polymer thus prepared, there is obtained a wrinkled film, opaque because of the presence of the sodium chloride, but hard and not sticky.

Example 2

Polycondensation of N,N'-bis-(2,3-epoxy propyl) piperazine and piperazine.

A polymer similar to that of Example 1, but free of sodium chloride, is prepared by polycondensation of piperazine and N,N'-bis-(2,3-epoxy propyl) piperazine in an aqueous medium and in stoichiometric proportions. The N,N'-bis-(2,3-epoxy propyl) piperazine can be prepared in the following way:

To 86 g (1 mole) of anhydrous piperazine, dissolved in 540 g of isopropanol, there are added, over a 30 minute period at a temperature of 10°C-15°C, 185 g of epichlorohydrin (2 moles). This temperature is maintained at this level while the reaction mixture is stirred for 7 hours.

The resulting dichlorohydrin, derived from piperazine, is filtered and dried. It is a white crystallized product having a melting point of 108°C-110°C.

50.7 g (0.18 mole) of the dichlorohydrin thus obtained are dispersed in 100 ml of benzene. The suspension is cooled to 10°C. There are then added by fractions, over a 30 minute period, 15.5 g (0.37 mole) of ground sodium hydroxide. This temperature is maintained for 2 ½ hours. The sodium chloride precipitate is filtered and rinsed with three times 100 ml of benzene.

After elimination of the benzene, under partial vacuum, there are collected 26 g of white solid, corresponding, according to functional analyses, to N,N'-bis-(2,3-epoxy propyl) piperazine.

25 g (0.125 mole) of product thus prepared are heated with reflux with 10.8 g (0.125 mole) of anhydrous piperazine in 60 g of isopropanol for 3 ½ hours. The cationic polymer is then partially precipitated. The solvent is eliminated under partial vacuum and an almost white powder is thus obtained which is soluble in water and which exhibits a melting point of 190°C. The molecular weight of this polymer, measured in chloroform by the method of lowering the vapor tension, is 2460.

Example 3

Quaternization of the piperazine and epichlorohydrin polycondensate.

To 200 g of solution obtained according to Example 1 which contains 0.4 equivalent of base nitrogen, there are added 170 g of absolute ethyl alcohol and then 25.3 g (0.2 mole) of benzyl chloride. The reaction mixture is heated at 80°C for 1½ hours. The ethanol is then elimi-
nated under partial vacuum, while re-adding water, to obtain a 10 percent solution of dry extract.

Example 4  
Oxidation of the piperazine and epichlorohydrin polycondensate.

To 100 g of solution obtained according to Example 1 which contains 0.2 equivalent of base nitrogen, there are added, at a temperature of 50°C, 7.2 ml (0.13 mole) of 200 volume hydrogen peroxide. The temperature is maintained at this level for 10 hours.

The polymer solution obtained is perfectly limpid and yields, on evaporation, films comparable to those of Example 1.

Example 5  
Polycondensation of piperazine, benzylamine and epichlorohydrin.

To a solution of 97 g (0.5 mole) of hexahydrated piperazine, in 384 g of isopropyl alcohol, there are added, drop by drop at 15°C, 92.5 g (1 mole) of epichlorohydrin. The resulting solution is maintained, with stirring, at 15°C for 2 hours. Thereafter it is heated to 70°C, and over a 15 minute period, 54 g (0.5 mole) of benzylamine are added. The resulting mixture is heated with reflux for an hour. Then there are added, drop by drop, 160 g of methanol solution of sodium methyleate (0.98 mole). Heating is continued for 1 hour.

After cooling, the formed sodium chloride is filtered and the isopropanol is eliminated under partial vacuum. After drying under vacuum and in the presence of phosphoric anhydride, a hard, brittle, colorless solid is obtained, having a softening point of 65°C and a molecular weight, measured in absolute ethanol of 1600.

The compound thus obtained is soluble in water in an acid medium and in a dilute alcohol medium. It yields beautiful, hard and very brilliant films, after evaporation of its dilute alcohol solvent.

Example 6  
Polycondensation of N,N'-bis(2,3-epoxy propyl) piperazine and cetylamine.

24 g (0.1 mole) of cetylamine and 20 g (0.1 mole) of N,N'-bis(2,3-epoxy propyl) piperazine prepared according to Example 2, are heated with reflux in 45 g of isopropanol for 15 hours. The resin obtained is soluble in water in an acid medium, such as hydrochloric acid.

Example 7  
Quaternization of the product obtained in Example 6.

To 68 g of the above isopropanol solution (0.23 equivalent in base nitrogen) there are added, drop by drop at 30°C, 14.6 (0.11 mole) of dimethyl sulfate. Stirring is continued for 2 hours. The solvent is then eliminated under partial vacuum, with water being added to produce a final aqueous solution that is 10 percent by weight.

The film obtained by evaporation of the dilute solution is rather hard and not sticky.

Example 8  
Polycondensation of N,N'-bis(2,3-epoxy propyl) piperazine and dodecylamine.

18.5 g (0.1 mole) of dodecylamine and 20 g (0.1 mole) of N,N'-bis(2,3-epoxy propyl) piperazine, prepared according to Example 2, are dissolved in 90 g of isopropanol alcohol. After 10 hours of heating at reflux, the solvent is eliminated under partial vacuum. There is thus obtained a soft, colorless, transparent resin, soluble in water in the presence of hydrochloric acid and also in alcohol such as ethanol or isopropanol.

The molecular weight of this resin, measured in absolute ethanol, is 2900.

By evaporation of dilute solutions, soft, slightly sticky films are obtained.

Example 9  
Quaternization of the N,N'-bis(2,3-epoxy propyl) piperazine and dodecylamine polycondensate.

To 30 g of resin obtained according to Example 7 (0.21 equivalent in base nitrogen), dissolved in 40 g of isopropanol, there are added, drop by drop at 30°C, 13.5 g (0.1 mole) of dimethyl sulfate. Stirring is kept up for 2 hours at the same temperature. Then the solvent is eliminated under partial vacuum, with water being added to obtain the resin in the form of a 10 weight percent solution in water.

On evaporation of the dilute aqueous solutions, films are obtained which are always soft but which are no longer sticky.

Example 10  
Polycondensation of piperazine and N,N'-bis(chloroacetyl)ethylene diamine.

To a solution of 10.6 g (0.05 mole) of N,N'-bis chloroacetyl ethylene diamine in 125 g of water, there are added 9.7 g (0.05 mole) of hexahydrated piperazine. The mixture is heated for 3 hours at 100°C. While the heating is continued, the acid formed is neutralized by addition, in several fractions, of 0.1 mole of sodium hydroxide in the form of a 40 percent (10 g) NaOH solution.

A colloidal solution having good film-forming properties is thus obtained.

Example 11  
Polycondensation of N,N'-bis(2,3-epoxy propyl) piperazine, oleyamine and piperazine.

To a solution of 20 g (0.1 mole) of N,N'-bis(2,3-epoxy propyl) piperazine in 47 g of isopropanol, there are added 10.7 g (0.04 mole) of oleyamine and 5.16 g (0.06 mole) of anhydrous piperazine. After 4 hours of heating at reflux, the solvent is eliminated under reduced pressure. There is then obtained a white solid having a softening point of around 100°C, the product being insoluble in neutral water, but soluble in ethanol and in water in an acid medium such as HCl.

Films obtained by evaporation of the dilute solution are transparent, not sticky and slightly hard.

Example 12  
Polycondensation of piperazine and diglycidyl ether.

To 6.63 g (0.077 mole) of anhydrous piperazine in 11 g of isopropanol there are added over a 15 minute period at 30°C, 10 g (0.077 mole) of diglycidyl ether. The mixture is heated at reflux for 4½ hours. Then the solvent is eliminated, at reduced pressure, while water is added to obtain a colloidal solution of 5 weight percent active material, i.e. polymer.

Films obtained by evaporation of the dilute solution are opalescent, hard and not sticky.

The diglycidyl ether is prepared by reacting at 15°–20°C a stoichiometric amount of sodium hydroxide on bis(3-chloro 2-hydroxy propyl) ether. The diglycidyl ether is isolated by distillation under reduced pres-
Polycrystallization of piperazine and methylene bisacrylamide.

Example 13

To 15.4 g (0.1 mole) of methylene bisacrylamide provided in paste form with 18.6 g of water, there are added with agitation at a temperature between 0° and 5°C and under a nitrogen atmosphere 86 g of a 10 percent aqueous solution of piperazine (0.1 mole). The mixture is left standing for 30 hours at 25°C.

The polymer is precipitated by flowing the aqueous solution into a great excess of acetone.

There is thus obtained a white solid having a softening point of about 205°C and a melting point of 260°C.

By evaporating the dilute aqueous solution, there is obtained very hard, transparent and non-sticky films.

Example 14

Polycrystallization of piperazine and piperazine bisacrylamide.

To a solution of 19.4 g (0.1 mole) of piperazine bisacrylamide in 35 g of water, there are added with agitation at a temperature between 0° and 5°C and under a nitrogen atmosphere, 86 g of a 10 percent aqueous solution of piperazine (0.1 mole). The mixture is left standing for 30 hours at 25°C.

The polymer is precipitated by flowing the aqueous solution into a great excess of acetone.

There is thus obtained a white solid having a softening point of about 205°C and a melting point greater than 260°C.

By evaporating the dilute aqueous solution, there is obtained very hard, transparent and non-sticky films.

Example 15

Polycrystallization of 1,3-bis-piperazine-2-propanol and methylene bisacrylamide.

Method 1

To 15.4 g (0.1 mole) of methylene bisacrylamide provided in paste form with 23.1 g of water there are added with agitation at a temperature between 0° and 5°C under a nitrogen atmosphere, 152.5 g of a standardized aqueous solution containing 22.8 g (0.1 mole) of 1,3-bis-piperazine-2-propanol. The mixture is left standing for 30 hours at 25°C.

The polymer is precipitated in accordance with the procedure outlined in Example 14.

There is thus obtained a white solid exhibiting a softening point of 176°C and a melting point of 200°-210°C.

By evaporating the dilute aqueous solution, there is obtained very hard, transparent and non-sticky films.

The 1,3-bis-piperazine-2-propanol, utilized above, can be prepared as follows:

\[
\text{A} - \text{B} - \text{A} - \text{B} - \text{A} - \text{B} - \text{A} - \text{B} - \text{A} - \text{B} - \text{A} - \text{B}\]

Example 16

Polycrystallization of 1,3-bis-piperazine-2-propanol and piperazine bisacrylamide.

To a solution of 19.4 g (0.1 mole) of piperazine bisacrylamide in 39.1 g of water there are added with stirring at a temperature between 0° to 5°C and under a nitrogen atmosphere 152.5 g of a standardized aqueous solution containing 22.8 g (0.1 mole) of 1,3-bis-piperazine-2-propanol. The resulting reaction mixture is left to stand for 30 hours at 25°C. The polymer is precipitated from the reaction mixture in accordance with the procedure outlined in Example 14 thereby yielding a white solid having a melting point of about 205°-210°C. By evaporating the dilute aqueous solution there is obtained a hard, transparent and non-sticky film.

The following Table summarizes the reactions of Examples 1-16.

<table>
<thead>
<tr>
<th>Example No.</th>
<th>Reaction Pattern</th>
<th>Structural Pattern</th>
<th>Formula of Repeating Units</th>
</tr>
</thead>
</table>
| 1           | Condensation of piperazine and epichlorohydrin | - A - B - A - B - (I) | \[
\text{N} - \text{CH}_2 - \text{CH}_2 \text{O} \]
\text{H} \]
|             |                 |                               | \[
\text{OH} \]
|             |                 |                               | \[
\text{A} - \text{B} \]

Table
Condensation of \( \text{N,N}' \) bis-(2,3-epoxy propyl)piperazine and piperazine-MW=2460

Condensation of piperazine, benzylamine and epichlorohydrin-MW=1500

Condensation of \( \text{N,N}' \) bis-(2,3-epoxy propyl) piperazine and cetylamine

wherein \( R = \text{cetyl} \)

Condensation of \( \text{N,N}' \) same as above but \( R = \text{dodecyl} \) bis-(2,3-epoxy propyl) piperazine and dodecylamine

Condensation of piperazine and \( \text{N,N}' \) bis(chloroacetyl) ethylene diamine

Condensation of \( \text{N,N}' \) bis(2,3-epoxy propyl) piperazine, oleylamine and piperazine

wherein \( R = \text{oleyl} \)

Condensation of piperazine and diglycidyl ether

Condensation of piperazine and methylene bis-acrylamide

Condensation of piperazine and piperazine bis-acrylamide
EXAMPLES OF APPLICATION

Example 17
Setting lotion for very dry hair
Compound prepared according to Example 1
1 g
Propyl para-hydroxybenzoate
0.4 g
Neolane pink dye, C.I. 18810
0.005 g
Perfume
0.2 g
Water, q.s.p.
100 g

Example 18
Setting reinforcer for damaged hair
Compound prepared according to Example 1
1 g
Polyvinyl pyrrolidone/vinyl acetate 60/40
copolymer (M.W. 50,000 to 70,000)
0.5 g
Trimethyl cetlyl ammonium bromide
0.2 g
Perfume
0.1 g
Methyl para-hydroxybenzoate
0.1 g
Water, q.s.p.
100 g

Example 19
Setting reinforcer for normal hair
Compound prepared according to Example 1
1 g
Vinyl acetate/eritonic acid copolymer
(M.W. 20,000)
1 g
Trimethyl cetlyl ammonium bromide
0.1 g
Methyl violet dye, C.I. 42535
0.002 g
Perfume
0.1 g
Ethyl alcohol, q.s.p. 50°
Water, q.s.p.
100 g

Example 20
Anionic shampoo
R−(OCH₂−CH₂)₅−OSO₃Na wherein R = alkyl C₁₀−C₁₄
10 g
Diethanolamide of copra fatty acids
3 g
Water, q.s.p.
100 g
pH = 7

Example 21
Anionic shampoo
Compound prepared according to Example 1
0.75 g
Triethanolamine alkyl sulfate wherein alkyl =
C₁₀C₁₄ 70/30
15 g
Monoeithanolamide of copra fatty acid
4 g
Sodium N-lauryl sarcosinate
3 g
Acetylated lanolin
3 g
Water, q.s.p.
100 g
pH = 7.5

Example 22
Non-ionic shampoo
Compound prepared according to Example 1
2.5 g
R−(OCH₂−CH₂)₅−OH wherein R = alkyl C₁₈H₃₈
15 g
Alkoxylated anhydrous lanolin sold under the
trademark “Lantrol AWS” by Malstrom
Chemical Corporation, New Jersey
1.5 g
Hydroxypropyl methyl cellulose
0.3 g
Citric acid, q.s.p. pH 6
Water, q.s.p.
100 g
EXAMPLES OF APPLICATION

Example 17
Setting lotion for very dry hair
Compound prepared according to Example 1

Example 23
Cationic shampoo
Compound prepared according to Example 8
Bromide of dodecyl, tetradecyl and hexadecyl trimethylammonium sold under the tradename "Cetavlon"
Lauryl alcohol oxyethylated with 12 moles of ethylene oxide
Lauryl diethanolamide
Ethyl cellulose
Lactic acid, q.s.p. pH 4
Water, q.s.p.

Example 24
Amphoteric shampoo
Compound prepared according to Example 1

Example 25
Amphoteric shampoo
Compound prepared according to Example 1
Sodium salt of N,N-diethylamino propyl)N-dodecyl asparagin

Example 26
Anionic shampoo
Compound prepared according to Example 9
Sodium alkyl sulfate (alkyl = C₁₂₋₁₄)
Sodium N-lauryl sarcosinate
Lauryl monoethanolamide
Glycol distearate
Water, q.s.p.

Example 27
Anionic shampoo
Compound prepared according to Example 9
Sodium salt of N,N-diethylamino propyl)N-dodecyl asparagin
Diethanolamide of copra fatty acids
Hydroxypropyl cellulose
Water, q.s.p.

Example 28
Cream for treating dry hair
Compound prepared according to Example 1, q.s.p.
Cetyl alcohol
Stearyl alcohol
Cetyl-stearyl alcohol oxyethylated with 15 moles of ethylene oxide
Hydroxyethylecellulose
Perfume
Water, q.s.p.

Example 29
Conditioner for dry hair
Compound prepared according to Example 1, q.s.p.
Polyvinylpyrrolidone/vinyl acetate copolymer 70/30 (M.W. 40,000)
Perfume
Water, q.s.p.

Example 30
Anti-dandruff lotion for daily use
EXAMPLES OF APPLICATION

Example 17

Setting lotion for very dry hair

Compound prepared according to Example 1, q.s.p. 0.5 g
Lauryl isoquoinium bromide 1.3 g
Lactic acid, q.s.p. pH = 3.5-3.5 55 cc
Ethyl alcohol 0.1 g
Menthol panthenolate 0.3 g
Water, q.s.p. 100 g

Anionic shampoo in the form of a clear solution

Ammonium alkylsulfate (alkyl derived from copra) 3 g
Sodium alkyl ether sulfate wherein the alkyl moiety is derived from copra fatty acids and oxyethylated with 2 moles of ethylene oxide 7 g
Compound prepared according to Example 4 1 g
Lauryl diethanolamide 3 g
Mixture of mono- and di-glycerides of fatty acids, sold under the tradename "Aralcel 186" by Atlas 0.5 g
Lactic acid, q.s.p. pH 7.5 100 g
Water, q.s.p. 100 g

Example 32

Anionic shampoo

Sodium alkyl ether sulfate wherein the alkyl moiety is derived from copra fatty acids and oxyethylated with 2 moles of ethylene oxide 10 g
Copra diethanolamide 3 g
Compound prepared according to Example 13 0.8 g
Sodium monolauryl sulfosuccinate 2 g
Lactic acid, q.s.p. pH 7.5 100 g
Water, q.s.p. 100 g

Hair treated with the above composition is easy to comb, exhibits good volume, is shiny and lively.

Example 33

Anionic shampoo

The composition of Example 32 is repeated except that the compound prepared according to Example 13 is replaced with an essentially equivalent amount of the compound prepared according to Example 14. Essentially similar effective and advantageous results are achieved with this composition.

Example 34

Anionic shampoo

Triethanolamine lauryl sulfate 10 g
Lauryl diethanolamide 2 g
Compound prepared according to Example 3 1 g
Hydroxy propyl methyl cellulose 0.1 g
Lactic acid, q.s.p. pH 7.2 100 g
Water, q.s.p. 100 g

This solution imparts to hair a lively and shiny appearance.

Further in Examples 17–22, 24, 25 and 28–30, the compound prepared in accordance with Example 1 is replaced with the compound prepared in accordance with Example 15 and 16 in essentially equivalent amounts to provide equally effective hair treating compositions.

In all of the Examples of Application given above, the weight of the polymer is expressed as the active material therein.

What is claimed is:

1. Cosmetic composition for conditioning the hair comprising a solution in a solvent selected from the group consisting of water and water-lower alkanol, of a member selected from the group consisting of a film-forming cationic polymer having a molecular weight of about 1,000 to 15,000 and having the formula

\[
\]

wherein A represents

\[
\begin{array}{c}
\text{N} \\
\text{H}
\end{array}
\]

and Z represents B and B' wherein B and B' each independently represent a bivalent radical selected from the group consisting of (i) hydroxypropylene, (ii) alkylene having up to 5 carbon atoms inclusive and interrupted by 1–2 members selected from the group consisting of \(-\text{CONH}\) and

\[
\begin{array}{c}
\text{CON} \\
\text{NC}\end{array}
\]

(iii) hydroxy alkylene wherein the alkylene moiety has up to 6 carbon atoms inclusive and interrupted by a member selected from the group consisting of alkylamine wherein the alkyl moiety has 12–16 carbon atoms, benzylamine, oleylamine and oxygen, and (iv) hydroxy propyl-piperazinyl-hydroxypropyl,

2. Quaternary ammonium salt of the cationic polymer in (1) and

3. The oxidation product of the cationic polymer in (1), said member being present in an amount of about 0.1 to 5 percent by weight of said composition.

2. The composition according to claim 1, characterized by the fact that it is in the form of a cream.
3. A method of conditioning the hair comprising applying to said hair an effective amount of the composition of claim 1.

4. A cosmetic composition for conditioning the hair comprising a solution in water-ethanol of a member selected from the group consisting of

1. a film-forming cationic polymer having a molecular weight of about 1,000 to 15,000 and having the formula


wherein A represents

\[-\text{CONH}\]

and Z represents B and B' wherein B and B' each independently represent a bivalent radical selected from the group consisting of (i) hydroxypropylene, (ii) alkylene having up to 5 carbon atoms inclusive and interrupted by 1-2 members selected from the group consisting of \(-\text{CONH}\) and

\[-\text{CONNH}_2\] (iii) hydroxy alkylene wherein the alkylene moiety has up to 6 carbon atoms inclusive and interrupted by a member selected from the group consisting of alkylamine wherein the alkyl moiety has 12-16 carbon atoms, benzylamine, oleylamine and oxygen and (iv) hydroxy propyl-piperazinyl-hydroxypropyl,

2. quaternary ammonium salt of the cationic polymer in (1) and

3. the oxidation product of the cationic polymer in (1), said member being present in an amount of about 0.1 to 5 percent by weight of said composition.

5. The composition of claim 4 which also includes an anionic, cationic, non-ionic or amphoteric surfactant.

6. The composition according to claim 4 having a pH between 3 and 11.

7. The composition according to claim 4 wherein said cationic polymer has a molecular weight less than 15,000.

* * * * *