

[54] **METHOD OF TREATING HUMAN HAIR TO ENHANCE SOFTNESS AND IMPROVE THE GENERAL APPEARANCE THEREOF**

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Related U.S. Application Data

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[51] Int. Cl. **A61k 7/06**

[58] Field of Search 424/70; 252/109, 528, 532, 252/531, 535, 536, 538, 539, DIG. 13, 135

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[57] **ABSTRACT**

Human hair is intimately contacted with an aqueous treating composition containing water and a catalytically effective amount of a novel catalyst to enhance softness and improve the general appearance thereof. In one variant of the method of the invention, the hair is shampooed with an aqueous shampooing composition containing water, a surface active agent, and a catalytically effective amount of the novel catalyst. A water softening agent may also be present when desired. The novel catalyst is prepared by steps including admixing a water soluble alkali metal silicate with an aqueous medium containing carefully controlled amounts of dissolved water soluble substances which are sources of calcium ion and magnesium ion, reacting the same to produce an aqueous colloidal suspension of the reaction product, admixing a micelle-forming surfactant with the aqueous medium, and agitating the aqueous medium containing the colloidal particles and the surfactant to form catalyst-containing micelles.

21 Claims, No Drawings

METHOD OF TREATING HUMAN HAIR TO ENHANCE SOFTNESS AND IMPROVE THE GENERAL APPEARANCE THEREOF

RELATED APPLICATIONS

This application is a continuation-in-part of copending application Ser. No. 317,097, filed Dec. 20, 1972 on behalf of John W. Willard, Sr. for NOVEL CATALYST AND PROCESS FOR PREPARING THE SAME. Application Ser. No. 317,097, in turn, is a continuation of application Ser. No. 108,198 filed Jan. 20, 1971, now abandoned.

BACKGROUND OF THE INVENTION

This invention broadly relates to a method of treating human hair. In one of its more specific variants, the invention further relates to a method of treating human hair with an aqueous composition containing a novel catalyst to impart certain desirable properties thereto.

As is well known, shampooing removes natural oils from the hair and there is often a tendency for the hair to be difficult to control immediately following shampooing. There is also a tendency for the hair to lose its softness and sheen, and to take on a relatively dull appearance which is less pleasing in appearance and feel.

A wide variety of substances have been used heretofore for the purpose of improving the general appearance of the hair and especially following shampooing. For instance, preparations have been applied to the hair heretofore for the purpose of providing a film or coating thereon, such as lanolin containing compositions and hair sprays containing tacky substances. Many of these compositions leave a visible film or coating on the hair and the hair loses its natural appearance and feel.

As a general rule, the above mentioned compositions are applied to the hair immediately following shampooing or thereafter at intervals as required to control the hair. Heretofore, an entirely satisfactory preparation was not available which could be combined with a prior art shampoo and used during the shampooing step.

The present invention provides for the first time an entirely satisfactory method of treating hair which is effective to improve the general appearance thereof and which may be practiced while shampooing or following shampooing. The active ingredients used in the method of the invention do not leave a noticeable film or coating on the hair, and the hair remains natural in appearance and in feel. The hair is soft and may be easily controlled as treated and has a high sheen immediately following the treatment, and it is not necessary to perform a further rinse with other chemicals. The treated hair also retains its acquired desirable properties for a much longer period of time. The method of the present invention does not adversely affect the growth of the hair, and uses ingredients which are non-poisonous and nonallergic.

It is an object of the present invention to provide a method of treating human hair to enhance softness and improve the general appearance thereof.

It is a further object to provide a novel method of treating human hair with an aqueous treating composition containing water and a catalytically effective amount of a catalyst to achieve the above mentioned desirable results.

It is still a further object to provide a novel method of shampooing hair which employs an aqueous sham-

pooning composition of the prior art having surface active properties and a novel catalyst whereby the general appearance of the hair following shampooing is greatly enhanced.

5 Still other objects and advantages of the invention will be apparent to those skilled in the art upon reference to the following detailed description and the specific examples.

DETAILED DESCRIPTION OF THE INVENTION INCLUDING PREFERRED VARIANTS THEREOF

In accordance with the present invention, human hair is treated by intimately contacting it with an aqueous treating composition containing water and a catalytically effective amount of a novel catalyst until, upon drying, the softness of the hair is enhanced and the general appearance thereof is improved. The novel catalyst will be described more fully hereinafter.

In practicing one variant of the invention, the hair may be wetted with the aqueous composition containing the catalytically effective amount of the catalyst in instances where it is not desired to shampoo the hair. The aqueous treating composition is allowed to remain in contact with the hair until the desired improvement is obtained and then it is dried.

In practicing another variant of the invention, hair having at least one undesirable substance thereon is shampooed under conditions to remove at least a portion thereof with an aqueous composition containing water and a catalytically effective amount of the catalyst following the usual techniques and procedures for shampooing with a conventional shampoo. The aqueous medium may or may not contain a surface active agent and/or a water softening agent. In many instances the presence of a surface active agent and/or a water softening agent gives improved results and is preferred. If desired, the hair may be shampooed with a prior art shampoo which contains a catalytically effective amount of the catalyst.

Regardless of the specific variant that is practiced, the liquid aqueous treating composition is allowed to remain in intimate contact with the hair until, upon drying, the softness of the hair is enhanced and it may be easily controlled, and the desired general improvement is obtained. The period of contact is at least one minute as a general rule, and usually from one minute to about fifteen minutes, and preferably about 2-5 minutes. The amount of time normally required for shampooing the hair following conventional prior art practices is effective, but the period of contact may be longer or shorter as necessary to achieve the desired results.

The treated hair may be allowed to dry naturally or it may be dried faster by using a commercially available hair dryer. The residue from the aqueous treating composition may be allowed to remain in contact with the hair after it is dried when this is desirable. It is also possible to rinse the hair in water following the treatment with the aqueous treating composition and this is preferred before drying in instances where a substantial amount of a surface active agent is present. The rinsing step may be in accordance with the usual practice followed when shampooing with a conventional detergent.

Any suitable prior art surface active agent which is commonly used or recommended for shampooing the hair is effective when used in practicing the invention

in the presence of the catalytically effective amount of the catalyst. Also, the generally recommended prior art procedures therefor may be used when practicing the present invention.

Suitable surface active agents of the prior art may be used to enhance the cleansing action. Examples of surface active agents include the alkali metal soaps of long chain fatty acids and especially the sodium and potassium soaps of fatty acids containing about 14-25 carbon atoms and preferably about 16-18 carbon atoms. Other surface active agents include detergents which are not derived directly from fatty acids such as synthetic anionic, cationic and nonionic detergents. Specific examples of synthetic anionic detergents include the alkali metal salts of organic sulfonates or organic sulfates, and especially the alkali metal salts of high molecular weight alkyl or alkylaryl sulfonates such as sodium or potassium dodecyl benzene sulfonate, and the sodium and potassium sulfates of straight chain primary alcohols such as sodium lauryl sulfate and other sodium and potassium sulfates of fatty alcohols or products of the "Oxo" process. Specific examples of cationic detergents include the quaternary ammonium halides such as benzethonium chloride, and often members of this group have outstanding germicidal activity as well as surface active properties. Specific examples of nonionic detergents include compounds having a polyoxyethylene or other oxygenated side chain and the remainder of the molecule may be derived from fatty acids, alcohols, phenols, amides or amines.

Further examples of surface active agents are disclosed in the section on detergency appearing in the Kirk-Othmer Encyclopedia of Chemical Technology (2nd Edition), Volume 6, pages 853-895, the disclosure of which is incorporated herein by reference. Still additional specific examples of detergents are found in numerous United States patents, including the following which are incorporated herein by reference:

3,031,510	3,119,848	3,222,287	3,382,177
3,043,780	3,140,261	3,223,647	3,382,285
3,048,548	3,144,412	3,282,852	3,422,021
3,053,771	3,156,655	3,314,891	3,424,689
3,061,551	3,173,877	3,320,172	3,429,822
3,067,143	3,203,955	3,326,807	3,437,697
3,082,172	3,208,949	3,337,463	3,444,242
3,095,381	3,101,374	3,349,038	3,499,841
3,101,297	3,213,028	3,359,205	3,507,798
3,118,000	3,215,633	3,382,176	

It is understood that the above surface active agents are given by way of example only, and that other suitable surface active agents may be used. A mixture of one or more of the above surface active agents may be used when desired. It is also common practice to admix therewith inert diluents such as sodium sulfate, or builders such as polyphosphates, polysilicates and sodium carboxymethyl cellulose.

Water softening agents may be employed in the aqueous treating medium in the presence or absence of a surface active agent. Examples of water softening agents include washing soda, trisodium phosphate, sodium metaphosphate, sodium tetraphosphate and other substances effective to remove calcium and/or magnesium ions from the water used in preparing the aqueous treating medium. The water softening agent may be employed in the usual amount necessary to soften the water to the desired degree.

The water used in preparing the aqueous treating composition may be untreated or softened tap water derived from municipal water sources, wells or springs. A soft water often gives better results and is preferred as with a conventional shampoo. The surface active agent, when present, may be added in an amount much less than is normally required due to the pronounced catalytic effect of the catalyst. The catalyst seems to enhance the effectiveness of a given quantity of surface active agent as well as achieve the other desirable benefits in improving the appearance and feel of the hair.

The catalyst is present in the aqueous treating medium in a catalytic quantity which results in the desired improvement. Usually the catalyst is present in an amount of about 0.00001-0.1 weight percent, and preferably about 0.0004-0.0001 weight percent of the aqueous treating medium, but larger or smaller amounts may be present as it is only necessary to provide the catalyst in catalytic amount. The aqueous treating medium may be prepared by diluting the aqueous suspension of the catalyst as produced. In such instances, the aqueous suspension of catalyst as produced may be diluted with about 100-10,000 volumes of water and then used in treating the hair. For better results, the aqueous suspension of catalyst as produced may be diluted with about 250-2000 parts of water before use, and for still better results it may be diluted with about 500-1000 parts by weight of water before use. A surface active agent suitable for use in shampooing the hair may be added thereto either before or during the shampooing step. Alternatively, the catalyst may be added to prior art shampoo formulations.

The catalyst that is employed in practicing the present invention is unique and many unusual and unexpected properties. It is presently thought that these unusual and unexpected properties result from the way the catalyst is prepared, and thus it should be prepared following the process described hereinafter.

PREPARATION OF THE CATALYST

The catalyst used in practicing the present invention may be prepared as described below. In the presently preferred process for preparing an aqueous suspension of the catalyst, a water soluble alkali metal silicate is admixed and reacted with an aqueous solution of a water soluble dissolved substance which is a source of calcium ion and a water soluble dissolved substance which is a source of magnesium ion to produce a finely divided or colloidal suspension of the reaction product. The aqueous solution contains the dissolved substances initially in amounts to provide between about 1×10^{-4} and 1×10^{-1} mole per liter each of calcium ion and magnesium ion, preferably between about 1×10^{-3} and 1×10^{-2} mole per liter, and for still better results between 1×10^{-3} and 6×10^{-3} mole per liter. The dissolved substances should also be present in amounts to provide a molar ratio of calcium ion to magnesium ion between about 2.0:1.0 and 1.0:2.0, and preferably about 1.5:1.0 and 1.0:1.5. For best results, the aqueous medium should contain the dissolved substances in amounts to provide between about 2.5×10^{-3} and 3.0×10^{-3} mole per liter each of calcium ion and magnesium ion, and the molar ratio of calcium ion to magnesium ion should be about 1.0:1.0, e.g., 2.9×10^{-3} mole per liter of calcium ion and 2.7×10^{-3} mole per liter of magnesium ion. The alkali metal silicate should have an alkali metal oxide to silicon dioxide ratio between

about 0.9:1.0 and less than 2.0:1.0, and preferably between about 0.9:1.0 and 1.2:1.0. The alkali metal silicate should be admixed with the aqueous medium in an amount of about 0.05–2 moles per liter, preferably about 0.1–1 mole per liter, and for still better results about 0.2–0.5 mole per liter. For best results, the alkali silicate should be an alkali metal meta-silicate having an alkali metal oxide to silicon dioxide ratio of about 1:1, and it should be admixed with the aqueous medium in an amount to provide about 0.2–0.3 mole per liter, e.g., about 0.25 mole per liter.

Examples of sources of calcium ion and magnesium ion for use in preparing the aqueous solution include mineral acid salts such as the halides, sulfates, bisulfates, nitrites, and nitrates of calcium and magnesium. The chlorides are usually the preferred halides, and both calcium and magnesium chloride are soluble and may be used. Magnesium sulfate and bisulfate are soluble and often are the preferred sources of magnesium ion. Calcium sulfate is only slightly soluble in water and usually is not a preferred source of calcium ion, but calcium bisulfate is somewhat more soluble. While calcium and magnesium nitrite or nitrate are soluble in water and may be used, these substances are not preferred in most instances. The sources of calcium ion and magnesium ion are dissolved in the aqueous medium in amounts to provide calcium ion and magnesium ion within the above ranges. Complete ionization is assumed when calculating the quantities to be dissolved and any desired order of addition is satisfactory. For example, the source of calcium ion may be added to the aqueous medium before, during or after the source of magnesium ion.

The alkali metal silicate to be admixed with the aqueous medium is preferably a water soluble sodium or potassium silicate having an alkali metal oxide (M_2O) to silicon dioxide (SiO_2) mole ratio between about 0.9:1.0 and less than 2.0:1.0, and preferably between about 0.9:1.0 and 1.2:1.0. The best results are usually obtained with an alkali metal metasilicate having an alkali metal oxide to silicon dioxide ratio of about 1:1. Hydrated alkali metal silicates dissolve faster and should be used for best results when the alkali metal silicate is added in solid form. In instances where an anhydrous alkali metal silicate is used, it may be desirable to dissolve it in water and then add the solution to the aqueous medium. Sodium metasilicate is preferred and usually a hydrated sodium metasilicate such as the pentahydrate gives the best results.

Carbonate ion and/or bicarbonate ion should not be present in the aqueous medium in substantial concentrations as the calcium ion and magnesium ion are precipitated in the form of their respective carbonates. The free carbonate ion and/or bicarbonate ion concentrations in the aqueous medium should not exceed about 10 parts per million by weight based upon the combined weight of the water and the ingredients added thereto and for this reason, the alkali metal silicates should be substantially free of carbonate ion and bicarbonate ion. A small amount of precipitated calcium carbonate and/or magnesium carbonate may be present in the aqueous medium provided additional calcium ion and magnesium ion are available to meet the above defined concentrations.

Distilled water and/or deionized water are usually preferred over a natural or untreated water when preparing the aqueous medium. In instances where water

is used which contains substantial initial concentrations or alkaline earth metal ions, then this should be taken into consideration in calculating the amounts of the sources of calcium ion and magnesium ion which are necessary to arrive at the final concentrations previously discussed.

An electrolyte which aids in the preparation of colloidal suspensions may be present in the aqueous medium at the time of admixing the alkali metal silicate therewith. Examples of electrolytes include those used in preparing prior art colloidal suspensions such as the alkali metal halides, sulfates and bisulfates. Sodium chloride, sodium sulfate and sodium bisulfate are usually preferred. The electrolyte should be added in small amounts such as, for example, about 0.00001–0.1 mole per liter, but often larger or smaller amounts may be present.

The conditions under which the alkali metal silicate is admixed with the aqueous medium and reacted with the sources of calcium ion and magnesium ion are not critical provided the reaction mixture is maintained in the liquid phase. The reaction temperature may be for example, between the freezing point and boiling point of water under the existing pressure conditions. At atmospheric pressure, the temperature is usually about 10°–90°C and often a more convenient temperature is about 20°–50°C. In many instances, ambient or normal room temperature is satisfactory.

The degree of agitation is not critical, and mild to vigorous agitation may be employed during addition of the alkali metal silicate. For the best results, the aqueous medium should be agitated sufficiently to assure rapid and uniform admixing of the alkali metal silicate. After completing the addition of the alkali metal silicate, when desired the agitation may be continued for a sufficient period of time to assure complete reaction and aging of the resulting colloidal suspension, such as for approximately 1–5 minutes to one hour or longer.

Upon admixing the alkali metal silicate with the aqueous medium, it takes on a turbid appearance but in most instances no significant amount of visible precipitate is formed. The colloidal suspension of the reaction product thus produced should be strongly basic and may have a pH value of, for example, approximately 10–14 and preferably about 11–13, and for best results about 12. In view of this, the initial pH value of the aqueous medium containing the dissolved sources of calcium ion and magnesium ion is of importance and should be about 6–9 and preferably about 7–8. When necessary, it is possible to adjust the pH value of the aqueous medium to the foregoing levels either before, during or after addition of the alkali metal silicate by adding bases such as sodium or potassium hydroxide, or mineral acids such as sulfuric or hydrochloric acid.

The colloidal suspension may be stored for several weeks or longer while awaiting the further treatment described hereinafter. In instances where the colloidal suspension is to be stored over a substantial period of time, the pH value should be maintained at the above described level and the storage vessel is preferably a tightly capped polyethylene bottle or other inert plastic container which prevents the contents from absorbing carbon dioxide from the atmosphere.

The colloidal suspension of the reaction product is not suitable for use as a catalyst as prepared and it should be agitated sufficiently in the presence of a micelle-forming surfactant to form catalyst-containing

micellees. The degree of agitation, the length of the agitation period, and the amount of the micelle forming surfactant that is present in the colloidal suspension are controlled at levels favorable to the formation of micelles. For example, the surfactant may be present in an amount of about 0.001–0.1 mole per liter and preferably about 0.03–0.07 mole per liter for most surfactants. Smaller or larger amounts may be effective with some surfactants such as 0.0001 mole per liter or less, or 0.2 mole per liter or more. About 0.05 mole per liter often gives the best results with many surfactants.

The minimum period of agitation and the minimum degree of agitation that are required for micelle formation varies somewhat with temperature and the type and amount of surfactant. As is well understood in this art, gradually increasing these variants in the presence of an effective amount of the micelle-forming surfactant will result in micelle formation when the proper levels are reached. As a general rule, longer periods of agitation and/or more vigorous agitation are required to form micelles at lower temperatures approaching the freezing point of the colloidal suspension than at higher temperatures approaching the boiling point. In instances where the aqueous suspension has a temperature of approximately 50°–90°C., then mild agitation over a period of about 10–60 minutes is satisfactory. Often longer or shorter periods of mild to vigorous agitation may be employed such as from about 1–5 minutes to several hours at temperatures varying, respectively, between the boiling point and the freezing point. When desired, the agitation may be continued long after the catalyst-containing micelles are formed as continued agitation does not seem to have an adverse affect.

As a general rule, the micelle-forming surfactants know in the prior art may be used in practicing the present invention. Micelle-forming surfactants used in the emulsion polymerization of monomeric organic compounds are disclosed in the text *Synthetic Rubber*, by G. S. Whitby, et al, John Wiley & Sons Incorporated, New York (1954), and surface active agents in general are disclosed on pages 418–424 of the text *Organic Chemistry*, Fieser and Fieser, 2nd Edition, Reinhold Publishing Corporation, New York, New York (1950), the disclosures of which are incorporated herein by reference. Examples of surfactants disclosed in the above texts include the alkali metal soaps of long chain fatty acids, and especially the sodium and potassium soaps of fatty acids containing about 14–25 carbon atoms and preferably about 16–18 carbon atoms, and the sodium and potassium soaps of the rosin acids, abietic acid and the derivatives thereof. Other micelle-forming surfactants include fats and oils such as corn oil, cotton seed oil, castor oil, soy bean oil and safflower oil which have been fully or partially saponified with alkali metal bases to produce mixtures including saponified long chain fatty acids, the mono- or diglycerides thereof, and glycerin.

Examples of synthetic micelle-forming surfactants include the sulfonates of long chain alcohols prepared by hydrogenation of naturally occurring fats and oils of the above types and especially sulfonated long chain alcohols containing about 10–20 and preferably about 12–14 carbon atoms, the alkali metal salts of the mono-sulfonates of monoglycerides such as sodium glyceryl monolaurate sulfonate, the sulfonates of succinic acid esters such as dioctyl sodium sulfosuccinate and the al-

kylyl alkali metal sulfonates. Specific examples of presently preferred micelle-forming surfactants include sodium and potassium sulforicinoleate, tetrahydronaphthalene sulfonate, octahydroanthracene sulfonic acid, butyl naphthalene sulfonic acid, sodium xylene sulfonate, alkyl benzene sulfonic acid and potassium benzene sulfonate.

Sulfated long chain hydroxycarboxylic acids containing about 14–25 carbon atoms and preferably about 16–18 carbon atoms, and sulfated fats and oils containing hydroxycarboxylic acids of this type produce exceptionally good micelle-forming surfactants. At least 25 percent of the hydroxyl groups and preferably at least 50 percent should be sulfated, and up to 95–100 percent may be sulfated. It is usually preferred that the sulfated oils and/or long chain hydroxycarboxylic acids be neutralized with an alkali metal base, and that the corresponding alkali metal salts be added to the colloidal suspension in the form of an aqueous solution. The aqueous solution may contain at least 25 percent of water and preferably at least 35–40 percent by weight. Much larger percentages of water may be present when desired such as 75–80 percent or more by weight.

A very active catalyst is produced when using sulfated castor oil as the micelle-forming surfactant (Turkey Red oil.) Sulfated castor oil which has been purified sufficiently to be of U.S.P. or medicinal grade produces an exceptionally active catalyst. For the best results, the castor oil is reacted with about an equal weight of concentrated sulfuric acid (e.g., 20 percent by weight) at a temperature of approximately 25°–30°C. The mixture may be reacted for about two hours with stirring and is then neutralized with sodium hydroxide solution. The reaction mixture separates into three layers, i.e., an upper layer which is a water solution, an intermediate or oily layer, and a white curdy precipitate. The intermediate oily layer is separated from the upper and lower layers, and may be added to the colloidal suspension as the micelle forming surfactant in an amount, for example of 0.001–0.1 mole per liter and preferably about 0.005 mole per liter.

The activity of the catalyst may be increased very markedly by cooling the aqueous catalyst suspension to a temperature approaching the freezing point such as about 0°–10°C., and then warming over one or more cycles. For best results, the aqueous catalyst suspension should be frozen and thawed over one or more cycles. The reason for the increased catalytic activity is not fully understood at the present time but cooling and then warming the aqueous catalyst suspension seems to increase the concentration of the catalyst-containing micelles and/or increases the catalytic activity thereof.

The aqueous suspension of the catalyst contains a relatively small percentage by weight of the active catalyst as produced. When desired, it may be concentrated by evaporating a portion of the water to produce a concentrated liquid catalyst suspension which may be stored and used more conveniently. It is also possible to prepare a dry catalyst concentrate by evaporating substantially all of the water. The preferred method of producing the dry catalyst concentrate is by flash evaporation using a technique analogous to that employed in preparing powdered milk. The catalyst concentrates produced upon partial or complete evaporation of the water content of the initially prepared aqueous suspension may be reconstituted by addition of water with little or no loss of catalytic activity. Preferably, the water

is added to the dry catalyst concentrate under sufficiently vigorous conditions of agitation to assure that the catalyst micelles are resuspended and uniformly distributed.

The aqueous catalyst suspension may be used as produced in practicing the invention, but preferably it is diluted with approximately 100–10,000 parts by weight

of water and then used. For better results, the catalyst suspension should be diluted with about 250–2,000 parts by weight of water before use, and for best results it should be diluted with about 500–1,000 parts by weight of water before use. The surface active agent may be added thereto when desired as previously discussed. Alternatively the dry catalyst or liquid catalyst concentrate may be admixed with water and/or the surface active agent to provide an effective catalyst concentration in the quantities previously discussed. The weight of the catalyst is calculated on a dry solids basis, i.e., the weight of the catalyst ingredients in the aqueous suspension as produced after removal of the water.

The invention is further illustrated by the following specific examples.

EXAMPLE I

This example illustrates one presently preferred process for preparing the novel catalyst used in practicing the invention.

Anhydrous calcium chloride in an amount of 0.66 gram and magnesium sulfate heptahydrate in an amount of 1.32 grams were dissolved in two liters or deionized water with stirring and warming until solution was complete. Then 95 grams of sodium silicate pentahydrate having a molecular ratio of sodium oxide to silicon dioxide of 1:1 were added to the solution with stirring and continued warming to produce a white colloidal suspension of the reaction product.

After setting for 10 minutes, the colloidal suspension was heated to 80°C. and sulfated castor oil in an amount of 201 grams was added with stirring. The average molecular weight of the sulfated castor oil was 940 and it contained 50 percent of water. The turbidity lessened somewhat as the colloidal suspension was heated at 80°–90°C. for one hour with vigorous stirring to produce catalyst micelles. The aqueous suspension of catalyst micelles thus prepared had a viscosity similar to that of water and it was used as the catalyst in certain Examples as noted hereinafter.

A dry or solid catalyst concentrate was prepared in a further run by evaporating water from the initially prepared aqueous catalyst suspension. The resulting dry catalyst concentrate was resuspended in water and there was no substantial loss of catalytic activity. In still other runs, the catalytic activity of the aqueous suspension of catalyst as initially prepared, the diluted aqueous suspension of catalyst, and the reconstituted aqueous catalyst suspension was enhanced by freezing and thawing.

EXAMPLE II.

This example illustrates the preparation of additional catalyst suspensions.

Five suspensions of the catalyst were prepared from the same ingredients as used in Example I and following the general procedure of Example I. The ratios of ingredients were varied as follows:

Ingredient	Amount of Ingredient				
	Run 1	Run 2	Run 3	Run 4	Run 5
Deionized water	2 l	1.5 l	1.5 l	1.5 l	0.25 l
CaCl ₂	0.66 g	0.5 g	0.5 g	1.0 g	0.5 g
MgSO ₄ · 7H ₂ O	1.32 g	1.0 g	1.0 g	2.0 g	1.0 g
Na ₂ SiO ₃ · 5H ₂ O	165 g	132 g	71 g	185 g	71 g
Sulfated Castor oil (approximately 50% by weight H ₂ O)	100 ml	150 ml	150 ml	200 ml	150 ml

The catalyst suspensions prepared by the above five runs were used in certain examples as noted hereinafter.

EXAMPLE III

This example illustrates the use of a catalyst prepared in accordance with Example I in treating hair following shampooing.

The hair was shampooed using tap water and a commercially available shampoo (Prell), rinsed thoroughly with tap water to remove the residual shampoo, and dried. Following drying, the hair was intimately contacted with tap water containing 0.2 percent by volume of the aqueous catalyst suspension prepared in accordance with Example I. The water containing the catalyst was allowed to stay in contact with the hair for approximately ten minutes, and then the hair was dried. The dried hair was noticeably softer and more easily controlled following treatment with the diluted aqueous catalyst and also had a higher luster and sheen.

The above treatment of the hair with the diluted aqueous catalyst was repeated two days later, at which time the hair had lost some of its initial luster and sheen and was becoming more difficult to control. The beneficial results noted above were obtained and the hair was restored to its original condition immediately following shampooing and treatment with the diluted aqueous catalyst. It was not necessary to shampoo the hair immediately prior to the second treatment with the diluted aqueous catalyst suspension in order to obtain the beneficial results.

EXAMPLE IV

This example illustrates the shampooing of hair with commercial shampoo and water containing a catalytic amount of the catalyst of the invention.

The aqueous suspension of catalyst as prepared in accordance with Example I was diluted with 1,000 volumes of tap water to produce a diluted aqueous suspension of the catalyst. The hair was shampooed with a commercial shampoo (Prell) and the diluted aqueous catalyst, rinsed with tap water to remove the excess shampoo, and then dried.

The hair was noticeably softer and more controllable than when substituting tap water for the diluted aqueous catalyst in shampooing. The hair also had more luster and sheen and was improved in appearance.

EXAMPLE V

The general procedures of Examples III and IV were repeated with the exception of using the catalyst prepared in accordance with Example II. The catalyst of

Example II were found to be active and produced comparable results.

EXAMPLE VI

A catalyst suspension was prepared in accordance with the procedure of Example I. The water content was removed by evaporation to produce a dried catalyst concentrate.

The dried catalyst concentrate and a commercially available shampoo (Prell) are admixed in proportions to provide one weight percent of the dried catalyst in the resulting catalyzed shampoo composition. A portion of the mixture thus prepared was used shampooing following the general procedure of Example I. The admixture of catalyst and shampoo is as effective in shampooing the hair as the separate additions of catalyst and shampoo used in Example I.

EXAMPLE VII

The general procedures of Examples I through VI were repeated with the exception of substituting a castile soap for the commercial shampoo (Prell). The castile soap produced comparable results when used to shampoo hair in the presence of water containing a catalytic amount of the catalyst of the invention. In each instance, the appearance of the hair was improved markedly, and it was possible to control the same much easier immediately following shampooing and drying.

I claim:

1. A method of treating human hair to enhance softness and improve the general appearance thereof comprising

intimately contacting the hair with an effective amount of an aqueous treating composition containing water and an effective amount of a catalyst until the softness of the hair is enhanced and the general appearance thereof is improved,

the catalyst being prepared by a process comprising admixing a water soluble alkali metal silicate with an aqueous medium containing a dissolved substance which is a source of calcium ion and a dissolved substance which is a source of magnesium ion,

the aqueous medium containing said dissolved substances in amounts to provide between about 1×10^{-4} and 1×10^{-1} mole per liter each of calcium ion and magnesium ion,

the aqueous medium containing said dissolved substances in amounts to provide a molar ratio of calcium ion to magnesium ion between about 2.0:1.0 and 1.0:2.0;

the alkali metal silicate having an alkali metal oxide to silicon dioxide ratio between about 0.0:1.0 and less than 2.0:1.0 and being admixed with the aqueous medium in an amount of about 0.05–2 moles per liter,

reacting the alkali metal silicate with said dissolved substances providing calcium ion and magnesium ion to produce an aqueous suspension of finely divided particles of the reaction product,

admixing a micelle-forming surfactant with the aqueous medium in an amount to form catalyst micelles comprising said finely divided particles of the reaction product upon agitating the aqueous medium, and

agitating the aqueous medium containing said finely divided particles of the reaction product and sur-

factant to form said catalyst micelles.

2. The method of claim 1 wherein in the process for preparing the catalyst, said ratio of calcium ion to magnesium ion is between about 1.5:1.0 and 1.0:1.5.

3. The method of claim 1 wherein in the process for preparing the catalyst, said ratio of calcium ion to magnesium ion is about 1.0:1.0.

4. The method of claim 1 wherein in the process for preparing the catalyst, the alkali metal silicate is admixed with an aqueous medium containing said dissolved substances in amounts to provide between about 1×10^{-3} and 6×10^{-3} mole per liter each of calcium ion and magnesium ion.

5. The method of claim 1 wherein in the process for preparing the catalyst, the alkali metal silicate is admixed with an aqueous medium containing said dissolved substances in amounts to provide between about 2.5×10^{-3} and 3.0×10^{-3} mole per liter each of calcium ion and magnesium ion.

6. The method of claim 1 wherein in the process for preparing the catalyst, about 0.2–0.5 mole per liter of the alkali metal silicate is admixed with the aqueous medium.

7. The method of claim 1 wherein in the process for preparing the catalyst, the alkali metal silicate has an alkali metal oxide to silicon dioxide ratio between about 0.9:1.0 and 1.2:1.0.

8. The method of claim 1 wherein in the process for preparing the catalyst, the alkali metal silicate is alkali metal metasilicate having an alkali metal oxide to silicon dioxide ratio of about 1.0:1.0.

9. The method of claim 1 wherein the process for preparing the catalyst, about 0.01–0.1 mole per liter of the surfactant is admixed with the aqueous medium.

10. The method of claim 1 wherein in the process for preparing the catalyst, the surfactant is sulfated castor oil.

11. A method of shampooing human hair having at least one undesirable substance thereon comprising intimately contacting the hair with an effective amount of a shampooing composition containing (a) water, (b) an effective amount of an active ingredient which is at least one material selected from the group consisting of (1) a surface active agent, and (2) a combination of a surface active agent and a water softening agent, and (c) an effective amount of a catalyst under conditions whereby at least a portion of the said undesirable substance is removed,

the catalyst being prepared by a process comprising admixing a water soluble alkali metal silicate with an aqueous medium containing a dissolved substance which is a source of calcium ion and a dissolved substance which is a source of magnesium ion,

the aqueous medium containing said dissolved substances in amounts to provide between about 1×10^{-4} and 1×10^{-1} mole per liter each of calcium ion and magnesium ion,

the aqueous medium containing said dissolved substances in amounts to provide a molar ratio of calcium ion to magnesium ion between about 2.0:1.0 and 1.0:2.0;

the alkali metal silicate having an alkali metal oxide to silicon dioxide ratio between about 0.9:1.0 and less than 2.0:1.0 and being admixed with the aqueous medium in an amount of about 0.05–2 mole

per liter,
 reacting the alkali metal silicate with said dissolved
 substances providing calcium ion and magnesium
 ion to produce an aqueous suspension of finely di-
 vided particles of the reaction product,
 admixing a micelle-forming surfactant with the aque-
 ous medium in an amount to form catalyst micelles
 comprising said finely divided particles upon agi-
 tating the aqueous medium, and
 agitating the aqueous medium containing the said
 finely divided particles and surfactant to form said
 catalyst micelles.

12. The method of claim 11 wherein the said active
 ingredient of the aqueous shampooing composition
 comprises a major proportion of a mixture of (a) at
 least one surface active agent, and (b) at least one
 water softening agent.

13. The method of claim 11 wherein in the process
 for preparing the catalyst, said ratio of calcium ion to
 magnesium ion is between about 1.5:1.0 and 1.0:1.5.

14. The method of claim 11 wherein in the process
 for preparing the catalyst, said ratio of calcium ion to
 magnesium ion is about 1.0:1.0.

15. The method of claim 11 wherein in the process
 for preparing the catalyst, the alkali metal silicate is ad-
 mixed with an aqueous medium containing said dis-
 solved substances in amounts to provide between about

1×10^{-3} and 6×10^{-3} mole per liter each of calcium
 ion and magnesium ion.

16. The method of claim 11 wherein in the process
 for preparing the catalyst, the alkali metal silicate is ad-
 mixed with an aqueous medium containing said dis-
 solved substances in amounts to provide between about
 2.5×10^{-3} and 3.0×10^{-3} mole per liter each of cal-
 cium ion and magnesium ion.

17. The method of claim 11 wherein in the process
 for preparing the catalyst, about 0.2-0.5 mole per liter
 of the alkali metal silicate is admixed with the aqueous
 medium.

18. The method of claim 11 wherein in the process
 for preparing the catalyst, the alkali metal silicate has
 an alkali metal oxide to silicon dioxide ratio between
 about 0.9:1.0 and 1.2:1.0.

19. The method of claim 11 wherein in the process
 for preparing the catalyst, the alkali metal silicate is al-
 kali metal metasilicate having an alkali metal oxide to
 silicon dioxide ratio of about 1.0:1.0.

20. The method of claim 11 wherein in the process
 for preparing the catalyst, about 0.01-0.1 mole per liter
 of the surfactant is admixed with the aqueous medium.

21. The method of claim 11 wherein in the process
 for preparing the catalyst, the surfactant is sulfated cas-
 tor oil.

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UNITED STATES PATENT OFFICE
CERTIFICATE OF CORRECTION

PATENT NO. : 3,864,475
DATED : February 4, 1975
INVENTOR(S) : JOHN W. WILLARD, SR.

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

- Col. 3, L 11, Change "other" to -- Other --.
- Col. 5, L 1, Change "2.0:1.0," to --2.0:1.0, --.
- Col. 5, L 26, Change "dissolvwd" to -- dissolved --.
- Col. 5, L 28, Change "ionizAtion" to -- ionization --.
- Col. 7, L 1, Change "miscellees" to -- miscelles --.
- Col. 7, L 36, Change "know" to -- known --.
- Col. 7, L 39, Change "Compunds" to -- Compounds --.
- Col. 10, L 16, in the table under "Ingredient", delete -- s --.
- Col. 11, L 13, after "used" insert -- in --.
- Col. 11, L 54, Change "0.0:1.0" to -- 0.9:1.0 --.
- Col. 12, L 33, after "wherein" insert -- in --.

Signed and Sealed this

twenty-second Day of July 1975

[SEAL]

Attest:

RUTH C. MASON
Attesting Officer

C. MARSHALL DANN
Commissioner of Patents and Trademarks