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2,840,087

HAIR SETTING COMPOSITION AND METHOD FOR USE OF SAME

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No Drawing. Application December 19, 1952
Serial No. 327,030

4 Claims. (Cl. 132-7)

This invention relates to the treatment of hair on the human head and to compositions for use in same. It relates more particularly to compositions and methods for straightening kinky hair or for curling hair in the event that it is straight or has become straightened and to provide improvements in the characteristics and in the appearance of the treated hair.

In the copending application of Virgil A. Gant, Ser. No. 263,509, filed on December 26, 1951, now Patent No. 2,643,375, and in the copending application of Virgil A. Gant, Ser. No. 285,330, filed on April 30, 1952, now Patent No. 2,750,947, description is made of a new and improved composition formulated to contain an organo-silicon resinous material in an intermediate or in an advanced stage of polymeric growth, alone or in combination with an organo-silicon fluid or other lubricant for application in solvent solutions or from aqueous dispersions onto the hair fibers to improve the condition and the characteristics thereof. The organo-silicon resinous material is set on the hair fibers by thermal reaction in the event that the organo-silicon is in an intermediate stage of polymeric growth or else set by reaction in the presence of a catalyst alone or in combination with heat, or else the organo-silicon resinous material may be set on the hair fibers merely by elimination of the diluent in the event that the compound is incorporated in an advanced stage of polymeric growth.

The formed organo-silicon compounds are stable at elevated temperatures and provide a substantially colorless component on the hair fibers which effectively hold the hair fibers in position upon setting of the organo-silicon material. It has been found that the organo-silicon compounds are incapable of harmfully affecting the hair structure or the scalp skin associated therewith and are therefore easy and safe to handle for purposes of hair conditioning. When the organo-silicon resinous materials are used in combination with an organo-silicon fluid or other lubricant, the latter functions effectively to improve the softness and sheen of the hair fibers thereby markedly to improve the appearance of the hair and to lessen the stiffness of the hair with the result that the hair fibers soon lose their natural kinky tendencies and ultimately acquire a soft and silky feel. Quite unexpectedly, the organo-silicon compounds, whether in the form of a resinous component alone or in combination with the organo-silicon fluids, seem to impart marked life and vigor to the hair fibers without imparting any of the oily and greasy characteristics resembling constituents which have heretofore been used for hair straightening.

In the copending application of Hersh et al., Ser. No. 289,908, filed on May 25, 1952, now Patent No. 2,782,790, description is made of an improvement in a hair straightening composition of the type described wherein the organo-silicon compound is provided in the form of a silane having groups which hydrolyze readily upon addition of moisture to cause the immediate formation of polysiloxanes on the hair fibers whereby hair setting and

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conditioning is achieved. The improvement described and claimed in said copending application makes it possible to achieve hair conditioning without the use of heat for hair setting. The formation of the polysiloxanes on the hair fibers occurs as an incidence to the reactions which take place upon the addition of moisture for hydrolyzation of the silane followed substantially immediately by polymerization to form the polysiloxane on the hair fibers while the hair is being combed or has been set to a desired condition.

One of the objectionable features of compositions of the types previously described resides in the use of solvent medium for solution of the silanes or polysiloxanes. Such solvent substances constitute a hazard in the use of the material for hair setting and present a barrier to the shipment of such materials from station to station. In addition, the solvent component, present in high proportion in the hair treating composition, represents a major item in the cost of the hair conditioner and provides an odor which is difficult sometimes completely to eliminate.

Therefore, it is an object of this invention to provide a hair treating composition of the type described in which the organo-silicon compounds are contained in a stable aqueous system and it is a related object to provide a method for the use of same in the treatment of hair.

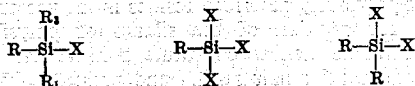
Another object is to provide a hair treating composition of the type described in which water constitutes the major diluent; which is stable in storage and use so as to prevent separation or deterioration of the hair treating composition upon storage; which is easy to prepare and which is safe and easy to apply onto the hair fibers for hair conditioning; which is safe to handle and ship and which is substantially free of odor and the ability to impart color.

In accordance with the practice of this invention, a silane of the type described in the copending application of Hersh et al., Ser. No. 289,908, and having from 1 to 3 hydrolyzable groups attached directly to the silicon atom and at least one organo group also attached to the silicon atom may be incorporated to form a stable hair treating composition when the aqueous medium is formulated to contain a base that is capable of maintaining the solubility or stability of the silanes or derivatives thereof and which functions also to neutralize the hydrolyzation and volatilization reaction whereby a polysiloxane is formed and deposited on the hair fibers upon drying.

As the base it is preferred to make use of ammonia in amounts to adjust the pH of the composition to between 5-10. For purposes of stability, it is preferred to adjust the pH of the composition to the acid side between 5-7 but for better performance on the hair fibers it is preferred to adjust the pH to the alkaline side between 7-10. Ammonia or other weak acid salts such as sodium acetate, sodium formate or the like apparently provide for a greater stability of the silane in the aqueous treating composition and it causes polymerization to take place in a manner to form lower molecular weight polymers capable of imparting some of the characteristics of an organo-silicon resin and some of the characteristics of an organo-silicon fluid thereby to impart the desired combination of hair setting and lubricity to the hair fibers. Stronger bases, such as sodium hydroxide or other alkali metal hydroxide may be used but to less advantage than ammonia. The alkali metal hydroxides react with the hydrolyzable groups of the silane to form the corresponding salts which, after the composition is applied to the hair fibers and set as the polysiloxane, should be removed from the hair fibers by acidification to form a salt which can be dissolved from the hair by water rinse or the like. Although the alkali metal hydroxides catalyze the reactions to pro-

duce polymers of larger molecular weight, they also act as end blocking units of the hydroxy groups necessary for polymerization. On the other hand, with ammonia, various polysiloxanols of lower molecular weight are formed into what is believed to be a stable colloidal dispersion in aqueous medium. When using an ammonia system, it becomes unnecessary to make use of an acid wash followed by a water rinse because the ammonia group can be removed by volatilization by drying preferably at elevated temperatures to deposit an insoluble polysiloxane on the hair fibers.

Silanes which may be used in accordance with the concepts of this invention may be represented by the formulae:



wherein X is a halogen of the type chlorine or bromine or an oxy group of low molecular weight capable of ready hydrolyzation from the organo-silicon molecule such as methoxy, ethoxy and the like oxy groups. R may be represented by hydrogen or alkyl groups of the type ethyl, methyl, propyl, butyl, isobutyl and the like, aryl or alkaryl groups of the type benzyl, phenyl, toluyl, naphthyl and the like. At least one of the R groups should be an organic group containing less than 8 carbon atoms in straight chain arrangement and having an unsaturated carbon to carbon linkage through which addition polymerization may take place, such for example as allyl, vinyl, methallyl, styryl and the like. Silanes of the type described include vinyltrichloro silane, methallyldichloro silane, diallyldiethoxy silane, methallyltriethoxy silane, styryltrichloro silane, and vinylchloro silane.

It has also been found that instability tends to result in the manufacture of the treating composition and inferior conditioning of the hair is secured when formulation of the silane in aqueous medium adjusted to the desired pH with ammonia is made at elevated temperature. However, when formulated at a temperature below 80° F. and preferably at a temperature below 55° F., a composition is formed which is stable under the conditions of storage for use in the treatment of hair fibers.

When the organo-silicon monomer of the hair treating composition contains two available hydrolyzable groups, hydrolyzation and substantially simultaneous condensation occurs in aqueous medium to form relatively straight chain polymers of a relatively soft and resilient character capable of setting the hair in positions held during organo-silicon polymerization while permitting resetting of the hair to desired configuration by subsequent application of heat or the like. When the organo-silanes in the treating composition are formed of a mixture of compounds having two hydrolyzable groups with a few compounds having three available hydrolyzable groups, a polymer is formed upon hydrolyzation and condensation having the characteristics of an elastomer whereby the hair particles remain highly flexible and embody a soft and silky appearance but are capable of maintaining hair setting conditions even in the presence of high humidity. As with the resinous materials formed with silanes having two hydrolyzable groups, the formed polymer permits resetting of the hair into pin curls or other configurations by the use of heat soon after resin formation. In the event that the organo-silane of which the hair treating composition is formed contains three available hydrolyzable groups, then cross-linked polymers are formed which are thermosetting in nature to provide for hair setting in a substantially more permanent manner.

Of the organo-silicon polymers formed in situ on the hair fibers by hydrolyzation and polymerization while holding the hair in position for setting, some of the polymers are liquids formed by the combination of a

smaller number of building units while others are solids of a waxy, rubber-like or resinous nature formed by considerably larger numbers of building units before polymerization is blocked. Those which are completely blocked against further polymerization are relatively heat stable but others are capable of further condensation and hardening upon the application of heat and/or catalyst but, in any event, sufficient polymers of solid character are produced by the initial hydrolyzation and polymerization to set the hair fibers. The polymers of low molecular weight function as lubricants in the final product to impart lubricity to the hair fibers and improve their luster and sheen and general overall appearance.

The character of the organo-silicon polymer formed depends not only upon the number of hydrolyzable groups available in the organo-silane but also on the character of the organic groups. Where the organic groups are of the low aliphatic series or aromatic groups, a harder product is formed which requires some lubricant to enhance the lubricity and softness as well as to improve the feel of the hair fibers while organic groups of the higher fatty acid radicals tend to produce polymers which are softer and more flexible in nature and therefore require less lubricant to achieve the desired characteristics. Thus in the practice of this invention, it is desirable to balance the silanes with respect to the number of hydrolyzable groups to control the polymer structure between liquid polymers of low molecular weight and harder polymers of high molecular weight and cross linking and also preferably to provide for a balance between silanes of long chain fatty acid groups such as dioctadecyl diethoxy silane which form soft and flexible lubricating polymers and silanes of short chained organic radicals such as with methyl, ethyl, and phenyl groups which produce harder solids of resinous nature.

As previously pointed out, by the proper selection of base and treating conditions, it is possible to regulate the type of polysiloxane which is formed on the hair fibers so as to impart the desired characteristics of the polysiloxane fluid and a polysiloxane resin. It is also possible by proper selection of the organo silane component itself to provide for the formation of a certain proportion of polysiloxanes of low molecular weight which correspond to the organo-silicon fluids and a certain proportion of polysiloxanes of high molecular weight which correspond to the polysiloxane resins whereby desired balance of lubricity and setting may be achieved by the treating composition. Where the amount of low molecular substance formed is insufficient to prevent matting or to impart the desired lubricity and silky softness to the hair, additional lubricants in the form of organo-silicon fluids in aqueous dispersion may be used, such for example as dimethyl polysiloxane (DC-200 fluid), diethyl polysiloxane (DC-400 fluid) and the like.

Generally it is desirable to formulate the treating composition of silanes calculated to produce polymers by hydrolyzation and polymerization having the desired balance of lubricity and bonding or which are adapted to form a maximum of about 5 percent by weight of the polymer which is solid in nature and a maximum of about 5 percent by weight of a polymer which is fluid in nature. When the solid polymers formed exceed 5 percent, hair stiffening occurs with the possibility of experiencing hair breakage and the hair becomes matted in a manner to prevent combining. Certain hairs will tolerate not more than 5 percent by weight polymeric solids especially when the polymer is able to impart some lubricity to the hair fibers but it is seldom desirable to exceed 10 percent by weight polymeric solids when calculated on the basis of the treating composition. When additional lubricant is required to provide the desired characteristics, instead of the organo-silicon fluids of the type described, other lubricants soluble in

the aqueous medium or which may be incorporated as dispersions in the aqueous medium may be used, such for example as mineral oils, palm oil, olive oil, vegetable oils, fatty acids, fatty acid amines and the like.

It is desirable to secure uniform distribution of the treating composition over the entire lengths of hair in order to avoid undesirable concentrations in certain areas which might cause matting or stiffening. Most hair acquires a film of oil on the outer surfaces thereof which often presents a barrier to the desired wetting out of the fibers and it is desirable, on that account, to incorporate a small amount of wetting agent or surface active agent in the treating composition. In the event that the hair is thoroughly washed prior to treatment, such wetting agents may be eliminated. As a wetting agent it is desirable to select a compound that is stable and which has little, if any, color and which is compatible with the system in which the organo-silane is contained.

It is preferred to make use of aryl alkyl polyethyl alcohols such as are marketed by Rohm & Haas under the trade name Triton X-100 or marketed by Atlas Powder Company under the trade name Tween. Use may also be made of sorbitan monolaurate, monopalmitate, monostearate polyoxyethylene derivatives or other commercially available materials such as the polyglycol esters represented by dioctyl ester of sodium sulfosuccinate or the alkyl naphthalene sulfonates, sorbitan sulfoleates, fatty alcohol sulphates, aromatic polyglycol ethers and the like. The amount of wetting agent may range from 0.05 to 2 percent by weight of the treating composition. Although more can be used, the amount specified is sufficient.

The following will illustrate the formulation of compositions embodying features of this invention and the use thereof in the treatment of hair on the human head:

Example 1

4.0 percent by weight diethyl diethoxy silane
95.0 percent by weight water adjusted to a pH of 7-9 with ammonium hydroxide
1.0 percent by weight wetting agent

Example 2

3.0 percent by weight diphenyl diethoxy silane
1.0 percent by weight ethyl triethoxy silane
1.0 percent by weight sodium ethyl phenyl, ethyl and phenyl polysiloxanolate copolymer
1.0 percent by weight wetting agent
94.0 percent by weight water adjusted to a pH of 7.5

Example 3

1 molecular equivalent methyl triethoxy silane
4 molecular equivalents of a base (ammonium or sodium hydroxide)

The above ingredients of Example 3 are introduced in water in amounts to provide for a solids concentration of about 3-5 percent by weight and 0.5 percent by weight of a wetting agent is added.

Example 4

2.0 percent by weight vinyl triethoxy silane
2.0 percent by weight diphenyl diethoxy silane
1.0 percent by weight wetting agent
95.0 percent by weight water adjusted to a pH between 6 and 9 with ammonia and a small amount of sodium hydroxide

In each instance, the base is first incorporated with aqueous medium and then the organo-silicon compound is added with mixing preferably after the base solution has been cooled to a temperature below 75° C. Best results are secured and greater stability results when the addition of the organo-silicon compound is made after the solution has been reduced to a temperature below 50° C. A stable solution is formed which can

be maintained over an indefinite period of time for use as a hair treating composition. In use, it is best first to wash the hair thoroughly to remove any dirt, grease, oil or the like which might interfere with the conditioning of the hair. After the hair is dry, a treating composition of Examples 1-4 may be applied lightly to the hair merely sufficient to dampen the hair. Application can be made by an atomizing spray, by a cotton swab or by applying the material onto portions of the hair and then working the hair with the fingers to secure uniform distribution. In straightening hair, it is best often to wet separated shocks of hair as by means of a spray or wetting pad and to process the individual shocks to the desired arrangement. The amount applied is critical only in the sense that too much will deposit solids and cause matting while insufficient treating composition will be ineffective to set the hair in the desired arrangement and water-proof the hair for subsequent permanence in hair setting.

Upon elimination of the diluent, as by slow evaporation or more rapidly by use of a hot comb, heat lamp, curling iron or other heating means, the hair should be combed or set to the arrangement desired. For hair straightening, it is desirable to comb or brush the hair straight during the final stages of the elimination of diluent. When a hot comb is used, generally heated to a temperature of 250-350° F., the hair becomes straightened substantially completely within a few passes of the comb through the hair.

Polymerization occurs during elimination of the diluent or subsequent thereto with the result that an organo-silicon polymer in the form of a polysiloxane lubricant or resin is formed on the hair fibers whereby the hair fibers are held in the desired arrangement and the formed organo-silicon polymer is believed to impart sufficient water repellency to the hair so that the hair is unable to be returned to its kinky or former condition even in the presence of moisture or high humidity. The organo-silicon polymers also impart improved lubricity and sheen to the hair and increase the softness thereof thereby to improve the hair texture and appearance. Kinky hair which has been straightened by this process can be subsequently permanently waved simply by wrapping the straightened hair about curlers and then heating in the manner generally employed for permanent waving for a short length of time with or without the application of additional treating composition. Hair which has been straightened by this manner can subsequently be treated with present compositions for permanent waving as with the compositions relying upon reduction with thioglycolic acid and oxidation to reform the hair fibers after the hair has been set while in plastic condition. Hair which is naturally straight can be permanently waved in accordance with present technique by wrapping the hair about curlers in the manner of a permanent wave and then applying the treating compositions of Examples 1-4 substantially uniformly to wet out the hair fibers. Upon elimination of the diluent, the hair set in pin curls or the like will maintain its set condition upon setting of the organo-silicon polymer on the hair fibers.

By way of still further improvement, it has been found that an organo-silicon compound may be incorporated to form a stable aqueous composition for use in the treatment of hair when the organo-silicon compound in the form of a silane or a corresponding silanol contains an aryl or aromatic group attached directly to the silicon atom. It has been found that the aryl or aromatic group stabilizes the silanol that is formed against the customary immediate condensation reaction to form the polymer while in aqueous medium. In the absence of an aryl or aromatic group attached directly to the silicon atom, polymerization by condensation between hydroxy groups of the silanol would proceed rapidly and possibly in an uncontrolled manner and provide an unstable composition. The 1 to 3 hydroxyl groups stabilized against re-

action in the presence of the aromatic groups are capable of immediate polymerization upon elimination of the diluent and heating to an elevated temperature to form the corresponding polymer which, depending upon the number of hydrolyzable or hydroxy groups, the type of organic groups, the catalytic agent present in the environment in which the reaction takes place, may range from a liquid lubricant to a solid polymer. By proper control a mixture can be formed of polymers of intermediate molecular weight to impart the desired ratio of lubricity and setting.

For still greater stability of the treating composition, use may be made of a base of the type previously described to adjust the pH of the composition to within a range of 7-9 and to catalyze the polymerization reaction upon drying. The amount of organo-silicon compound in the treating composition will correspond to the conditions previously described in the use of a silane in a base solution. Modification to include wetting agents and additional lubricants may also be made when incorporated as a water soluble compound or else dispersed in aqueous medium.

The following will illustrate the practice of this invention in the concepts described above:

Example 5

3.5 percent by weight diphenyl silane diol
1.0 percent by weight wetting agent
95.5 percent by weight water

Example 6

4.0 percent by weight phenyl silane diol
1.0 percent by weight wetting agent
95.0 percent by weight water adjusted to a pH of about 8 with ammonia

Example 7

2.0 percent by weight diphenyl silane diol
2.0 percent by weight methyl triethoxy silane
96.0 percent by weight water adjusted to a pH of 7.5 with ammonia

Example 8

3.0 percent by weight naphthol silane triol
1.0 percent by weight diethyl polysiloxane in aqueous dispersion
96.0 percent by weight water adjusted to a pH of 7.5

Application may be made as described in connection with Examples 1-4. Because of the stability of the silanol, it is possible to allow the hair to dry after being wet with the compositions of Examples 5-8 and then achieve hair setting simply by arranging the hair and heating to cause polymerization and setting of the organo-silicon compound on the hair fibers. Thus the treating composition may be applied to the entire head of hair and, when dry or just about dry, the hair may be combed with a hot comb, brushed under a drying lamp or heat lamp or set with pin curls, or waved with a hot iron or the like to effect simultaneous polymerization of the organo-silicon whereby the hair becomes set in the desired arrangement. Thus the process of hair setting and conditioning is still further simplified and rendered more practical.

As with the compositions of the type set forth in Examples 1-4, the straightened hair may be further conditioned for curling or arranged in another coiffure with or without the addition of more of the treating composition but preferably with the application of heat to effect further polymerization or setting.

It has been found further that a stable hair treating composition embodying an organo-silicon compound in aqueous medium may be prepared when an organo silane or silanol is so completely dispersed in the aqueous medium that the molecules remain sufficiently protected and separated one from another to minimize contact necessary for reaction to form the polymer. Complete

emulsification of this character generally requires the use of an emulsifying agent and the formation of the emulsion by supersonic vibration. In the preferred concept of this phase of the invention, the silane is first dissolved in alcohol or other solvent medium to achieve substantial dilution and then the solution in alcohol or other solvent is dispersed in the manner described in the aqueous medium. Dilution of the organo silane or silanol in amounts up to 70 percent by weight solvent followed by emulsification or dispersion in water to a solids content of 1-5 percent as previously described offer the best results with respect to stability and use.

Example 9

4.0 percent by weight diethyl diethoxy silane
8.0 percent by weight alcohol
87.0 percent by weight water
1.0 percent by weight emulsifying agent

Example 10

2.0 percent by weight dimethyl dimethoxy silane
2.0 percent by weight diphenyl diethoxy silane
9.0 percent by weight benzol
86.0 percent by weight water
1.0 percent by weight emulsifying agent

In each of the above examples, the silane is first dissolved in the solvent medium and then the solution is dispersed by supersonic vibration in the aqueous medium containing the emulsifying agent dissolved therein. Application in use corresponds to that previously described in connection with Examples 1-8. Condensation reaction to form the polymer occurs as emulsion breaks to enable reaction between the molecules and therefore may be caused to take place immediately upon passage of a hot comb through the hair for straightening thereby to set the hair in advance of the elimination of the diluent. The emulsion may be broken by the use of other heating means such as a curling iron, hot pads, heat lamp or the like for setting the hair in various arrangements desired. As in Examples 5-8, the hair may be allowed almost to dry before setting to the desired arrangement in anticipation of the breaking of the emulsion to form the organo-silicon compound. As in the foregoing, application may be made to separate shocks of hair as by means of an atomizing spray or a wetting pad followed by processing of the individual shocks to effect the desired arrangement until the entire head of hair is covered. As in previous systems, hair setting in pin curls or with a curling iron or the like to form a permanent after hair straightening may be achieved. Such after treatment to set the hair may be made with or without the application of additional material.

An important concept of this invention resides in the new and improved method for securing a permanent. It is generally recognized that a permanent of the type which makes use of a reduction reaction with thioglycolic acid or salts thereof to sever the sulphur to sulphur bond in the cystine molecule of the hair followed by oxidation with a peroxide or the like to reform the sulphur bond after the hair has been set is not available for use with kinky hair.

It is therefore an object of this invention to provide a method embodying compositions of this invention for enabling kinky hair to be permanently waved with compositions of the type described.

In accordance with the practice of this invention, kinky hair is straightened by the use of organo-silicon compositions in this and in the related applications of which this application is an improvement. After the kinky hair has been straightened in the manner described, then the standard compositions may be applied for permanent waving based upon reduction reaction with the glycolic acids or their salts to render the hair plastic followed by oxidation to reform the hair molecules after it has been set to the desired arrangement.

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This application is a continuation-in-part of applicant's copending application Ser. No. 289,908, filed on May 25, 1952.

It will be understood that various changes may be made in the selection of materials, their ratio within the area defined and their method of application and treatment without departing from the spirit of the invention, especially as defined in the following claims.

I claim:

1. A stable aqueous composition for hair setting comprising water as the diluent and containing an amount ranging from 1-5 percent by weight of an organo silane in a dissolved state in the aqueous medium wherein from one to three of the valence bonds of the silicon atom of the silane are satisfied by highly hydrolyzable ions selected from the group consisting of chlorine, bromine, methoxy, ethoxy and hydroxy ions with the remainder of the valence bonds being satisfied by an organic radical selected from the group of radicals consisting of methyl, ethyl, propyl, butyl, benzyl, phenyl, tolyl and naphthyl nuclei and in which ammonia is present in an amount to adjust the pH of the composition to between 7-10.

2. A stable aqueous composition for hair setting comprising water as the diluent containing an organo silane compound in a dissolved state in the aqueous medium wherein the silane is dissolved in an organic solvent and the solution of silane in solvent is dispersed dilutely in an aqueous medium in an amount to provide a concentration of the silane within a range of 1-5 percent by weight of the composition and wherein from one to three

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of the valence bonds of the silicon atom of the silane are satisfied by highly hydrolyzable ions selected from the group consisting of chlorine, bromine, methoxy, ethoxy and hydroxy ions with the remainder of the valence bonds being satisfied by an organic radical selected from the group consisting of methyl, ethyl, propyl, butyl, benzyl, phenyl, tolyl and naphthyl nuclei.

3. The method of setting hair comprising the steps of wetting the hair with a composition as defined in claim 1, setting the hair while eliminating the ammonia leaving an insolubilized organo silicon compound set on the hair fibers.

4. The method of setting hair comprising the steps of wetting the hair with a composition as defined in claim 2, setting the hair while eliminating the diluent to leave an insolubilized organo silicon compound set on the hair fibers.

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