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METHOD OF MAKING A SHAMPOO

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ABSTRACT OF THE DISCLOSURE

A method for preparing a shampoo comprising 13.5 to 23 percent by weight of water-soluble anionic sulfonated or sulfated detergent, 1 to 6 percent by weight of an alkylolamide, 6 to 10 percent by weight of soap, .5 to 2 percent by weight of hexachlorophene, and 0.5 to 6 percent by weight of finely divided sulfur is described which comprises forming a soap in situ in a first vessel under reaction conditions wherein a temperature above about 120° F. and a pH below about 6.0 are employed; transferring the resulting mixture to a second vessel where it is admixed with the remaining components of the shampoo under conditions which exclude temperatures above about 135° F. and a pH of below about 6.0, the sulfur being present solely in said second vessel, completing the preparation of said shampoo and removing said product from said second vessel leaving thereing a heel of product which may become a part of a subsequent batching operation.

The present invention relates to a new improved shampoo composition having as detergent component a water-soluble sulfated or sulfonated detergent salt, and more particularly to an improved composition which in addition contains an alkylolamide, sulfur, and hexachlorophene.

In accordance with the present invention, the shampoo composition contains, as primary ingredients, a mixture of at least one water-soluble anionic sulfated or sulfonated detergent, an alkylolamide, hexachlorophene, and sulfur. Included among the suitable detergents are the aliphatic sulfated or sulfonated agents, such as the aliphatic acyl-containing compounds wherein the acyl radical has about 8 to about 22 carbon atoms, and more particularly, the aliphatic carboxylic ester type, containing at least about 10 and preferably about 12 to about 26 carbon atoms to the molecule. As suitable examples of aliphatic detergents may be found the sulfuric acid esters of polyhydric alcohols incompletely esterified with higher fatty acids, e.g. higher fatty acid monoglyceride monosulfate and tallow diglyceride monosulfate; the long chain pure or mixed higher alkyl sulfates, e.g. lauryl sulfate, cetyl sulfate, higher fatty alcohol sulfates derived from reduced coconut oil fatty acids; the hydroxy sulfonated higher fatty acid esters, e.g. higher fatty acid esters of 2,3-di-hydroxy propane sulfonic acid; the higher fatty acid esters of low molecular weight alkylol sulfonic acids; sulfated higher fatty acid alkylolamides such as, e.g. ethanolamide sulfates; the higher fatty acid amides of amino alkyl sulfonic acids, e.g. lauric amide of taurine, and the like.

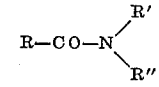
Also within the ambit of the invention are the alkyl aryl sulfonate detergents though these are not preferred generally because of their great drying power. These aromatic sulfonate detergents are also known in the art. They may be mononuclear or polynuclear in structure. More particularly, the aromatic nucleus may be drived from benzene, toluene, xylene, phenol, cresols, naphthalene, etc. The alkyl substituent on the aromatic nucleus may vary widely,

as long as the desired detergent power of the active ingredient is preserved. While the number of sulfonic acid groups present on the nucleus may vary, it is usual to have one such group present in order to preserve as much as possible a balance between the hydrophilic and hydrophobic portions of the molecule.

More specific examples of suitable alkyl aromatic sulfonate detergents are the higher alkyl aromatic sulfonates. The higher alkyl substituent on the aromatic nucleus may be branched or straight-chain in structure; it comprises moreover such groups as decyl, dodecyl, keryl, pentadecyl, hexadecyl, mixed long-chain fatty materials, cracked paraffin wax olefins, polymers of lower mono-olefins, and the like. Preferred examples of this class are the higher alkyl mononuclear aryl sulfonates wherein the alkyl group is about 8 to about 22, and preferably about 12 to 18, carbon atoms, e.g. n-dodecylbenzenesulfonate, sodium salt.

These various anionic detergents should be used in the form of their water-soluble salts such as the alkali metal and alkaline earth metal salts such as sodium or postassium salts, as well as in the form of salts of nitrogen containing bases, e.g. as salts of low molecular alkylolamines such as mono-, di- and triethanolamine, and as mixtures of various of these salts.

The alkylolamides of the present invention are characterized by their weakly polar nature and may be represented by the formula:



wherein R—CO represents a fatty acyl radical of 10–14 carbon atoms and R′ and R″ may be the same or different, wherein R′ may be a lower hydroxyalkyl group of preferably up to about 5 and, more particularly, from about 2 to about 5 carbon atoms, and R″ may be one of these radicals or hydrogen. The hydroxyalkyl groups may be mono- or polyhydroxy alkyl. Examples of suitable additives are N,N bis (2 hydroxyethyl) lauramide, N,N bis (2 hydroxyethyl) myristamide, N,N bis (2 hydroxyethyl) capramide, N,N bis (2,3 dihydroxypropyl) lauramide, N,N bis (2,3 dihydroxypropyl) myristamide, N,N bis (3-hydroxypropyl) lauramide, N,N bis (3 hydroxypropyl) capramide, N,N bis (2 hydroxypropyl) myristamide, N,N bis (4 hydroxybutyl) lauramide, etc., and the corresponding monoalkylolamides. Because of the excellent results obtained, it is preferred to use diethanolamide compounds possessing a fatty acyl radical of 12–14 carbon atoms.

These alkylolamides may be prepared in any suitable manner and numerous processes for their production are well known in the art. A convenient and economical mode of synthesis involves the condensation of the higher fatty acylating compounds (e.g. lauric acid, lauric acid halide, etc.) with a suitable amino compound to produce a reaction product having the desired amide structure.

The higher fatty acylating substances may be derived from pure, impure or commercial grades of capric, lauric, myristic acids and the like. More particularly, these acids may be produced from fatty oils, fats, greases, and other natural sources or be of synthetic origin as derived from the oxidation of hydrocarbons. According to its origin and the degree and manner of purification, capric, lauric and myristic acids may be commonly admixed or associated with other fatty acids of higher and lower molecular weight. It is within the scope of the invention that the caproyl, lauroyl and myristoyl compounds may be associated with other fatty substances and the like provided the character and amounts of such other materials are

not sufficient substantially to neutralize or materially to affect the enhancing power of the additives in the relationship set forth. Thus, there may be suitably employed for the preparation of the alkylolamide the commercially pure capric, lauric and myristic acids having a concentration of such acids of about 90% and above. A typical composition of commercially pure lauric acid may be about 1% C_{8-10}, about 71% C_{12}, about 28% C_{14}, and not more than about 1% C_{16} and higher. Another suitable fatty acid mixture is "topped" coconut oil fatty acids produced by the removal of a low-boiling fraction, e.g. 10–15%, from coconut oil in fatty acids.

Among the alkylolamines suitable for condensation are ethanolamine, diethanolamine, N,N bis (2,3 dihydroxypropyl) amine, propanolamine, dipropanolamine, N,N bis (2 hydroxypropyl) amine, dibutanolamine, etc. It is to be understood that the alkylolamines may be utilized in pure, impure or commercial form.

The present hair preparations contain as primary ingredients, in addition to the detergent and alkylolamide described above, hexachlorophene and finely divided sulfur. The sulfur and hexachlorophene components constitute a minor but important proportion, together on the order of about 1–8% of the instant hair preparations, the most suitable amount depending on the characteristics described. Thus, in a cream or lotion shampoo containing in combination a diethanolamide salt of coconut oil fatty acid, sodium lauryl sulfate, sulfur and hexachlorophene, the sulfur should be present in amounts on the order of about 0.5 to 6% by weight and the hexachlorophene should be present in amounts varying from about 0.5 to 2%.

It is important that the sulfur have a very small particle size wherein at least 90% and preferably 98% of the particles thereof have a diameter smaller than about 0.0017 inch. Preferably, the particles will have a size smaller than about 0.0004 inch. When the sulfur particles are larger than this size, or when the number of larger particles exceeds the stated limit, they tend to agglomerate and settle out even if a suspending agent is present. It is also important to maintain a predetermined viscosity. The viscosity of the shampoo composition at about 77° F. should be in excess of about 100 seconds, preferably in excess of about 150 seconds as measured by a No. 5 Raymond flowmeter. The test using a No. 5 Raymond flowmeter is performed by placing a sample at a specified temperature in a glass tube that has an inside diameter of one inch. At the bottom of this tube there is concentrically placed a stainless steel tube having an inside diameter of 0.252 ± 0.001 inch. The glass tube is marked so that the tube will read empty when there are two inches of material left in the glass tube above the upper end of the stainless steel tube. The apparatus is positioned so that both tubes are vertical. The tubes are filled and the material is allowed to flow freely from the bottom thereof. The time required for 50 milliliters of material to flow from a 50 milliliter mark to the empty mark is then measured in seconds. This method enables strict control of the viscosity. By control of the viscosity as described above, the ingredients of the composition are maintained in interrelated apposition whereby the cumulative effect of the various components is enhanced by the contiguity of the ingredients. In this manner, the hair preparations of the invention exhibit unusually excellent hair grooming properties. They render the hair soft and manageable, impart a high gloss and luster, and are particularly effective in controlling or preventing the formation of dry flaky scales of sloughed-off skin.

The present hair preparations may also contain as adjuvant materials various substances such as vitamins, hormones, cholesterol, lanolin, plant extracts, perfumes, colorants, buffers, and the like in compatible proportions, although the preparations should be free of substantial quantities of water-insoluble solid substances which may be deposited on and adversely affect the scalp. The instant composition should have a pH greater than about 6. Preferably the pH should be maintained above about 7.

The ingredients are proportioned and mixed with an aqueous base to form an opaque emulsion, generally of the oil-in-water type, wherein the detergent is partially dissolved in the aqueous phase and partially dispersed in fine particles throughout said emulsion, and the sulfur particles are also dispersed in said emulsion. The final product is a milky or creamy opaque lotion or cream. The amount of aqueous base is variable but will usually be above about 50% by weight and preferably about 60% for optimum results. The shampoos may be prepared in various forms. The composition should be opaque, have a high viscosity, and contain two or more mixed liquid and solid phases. For example, the compositions may be in the form of a cream or lotion shampoo.

The quantity of alkylolamide useful in the shampoos of this invention ranges from about 1.0 to 6% by weight. Preferably, there is employed an amount of about 4 to 6% of lauric-myristic diethanolamide when the final product is to be a lotion shampoo and about 1.0 to 2.5% if the final product is a cream shampoo. In this manner, the alkylolamide aids in maintaining the viscosity within a predetermined desired range, also controlling the consistency and the foaming properties of the final products. The amount of detergent to be used varies. Preferably, the detergent will be about 13.5 to about 21% of sodium lauryl sulfate for a lotion shampoo and 16 to 23% for a cream shampoo. The total quantity of primary ingredients, i.e., the detergent, alkylolamide, sulfur, and hexachlorophene should be maintained within a range of about 16 to 34% by weight in order to obtain a desirable product. If the total amount of primary ingredients falls below 16%, the cleansing effectiveness of the shampoo may be deleteriously affected. On the other hand, the total amount of primary ingredients should be maintained below about 34%. It has been found that by maintaining the primary ingredients within the ranges set forth above, the sudsing properties of the composition are excellent, and a pleasant fragrance can be imparted. The shampoo composition is cosmetically desirable and imparts a high gloss and luster thereby leaving hair soft and manageable. The instant compositions are mild to the skin yet have been found to be especially effective in removing grease and debris from areas adjacent the scalp. Furthermore, the instant compositions impart a lasting effect to the hair whereby subsequent accumulations of such debris tends to be lessened.

During preparation of this shampoo in a reaction vessel, the product tends to build up on the walls and floor of the vessel and on the mixer blades. This is especially true with the viscous products of this invention. There is also a dead space in the vessel below the product takeoff line. It has been found, however, that shampoo formulations produced in accordance with this invention may be adversely affected if a heel containing the sulfur is subjected to a heat treatment at the low pH employed in the beginning of the processing. The heel cannot be economically removed by cleaning because this would require considerable time and a special drain for diluted product. Furthermore, the product which would be removed by cleaning could represent a loss of a large enough percentage of material to make the difference between a profit or a loss where, as here, a viscous product is involved and the amount of the heel is relatively large. In accordance with this invention, it is possible to leave a heel of product for a subsequent batch, the initial processing of a new batch of material is carried out up to the point of addition of the sulfur in a first vessel, the batch is then transferred from the first vessel to a second vessel, the sulfur is added, and the processing is continued to completion. Upon removal of the final product, a heel of material is left in the second vessel for a subsequent batch, and the processing is repeated. This method has the additional advantage that it allows a second batch to be begun before the first batch is completed.

The combination of primary ingredients, namely the water-soluble anionic sulfated or sulfonated detergent salt, the alkylolamide, the sulfur, and the hexachlorophene affect the hair and scalp so that soil and oily secretions are readily displaced and eliminated, thus aiding in the grooming and care of the hair. The formulation of a cream shampoo in accordance with the procedure set forth herein will advantageously include the addition of a soap which may be formed in situ during the processing. The soap may be present in amounts of about 6.0 to 10.0 percent by weight. The use of about 8 percent by weight is preferretd.

Specific hair preparations according to this invention are illustrated by the following examples. All quantities indicated are by weight unless otherwise specified.

Example I.—Cream shampoo

The following composition is a typical cream shampoo of the invention:

	Percent
Sodium lauryl sulfate solution (30%)	60.8
Triethanolamine lauryl sulfate solution (42%)	4.5
Sodium chloride	1.4
Sodium benzoate	0.2
Methyl p-hydroxy benzoate	0.1
Anhydrous lanolin	0.5
Deionized water	15.6
Stearic acid	6.6
Caustic potash (34%)	4.9
Lauric-myristic diethanolamide (71:28)	1.5
Powdered sulfur [1]	2.0
Hexachlorophene (2,2'-dihydroxy-3,5,6,-3',5',6'-hexachloro diphenyl methane)	1.0
Monomethyloldimethyl hydantoin (25%)	0.4
Perfume	0.5
	100.0

[1] In excess of 98% of the sulfur particles have a size smaller than 0.0004 inch.

The sodium lauryl sulfate solution, triethanolamine lauryl sulfate solution, sodium chloride, sodium benzoate, methyl p-hydroxy benzoate, lanolin, and deionized water are mixed and heated to a temperature of about 160° F. The stearic acid is melted by separately heating it to a temperature of about 190° F. and the melted stearic acid is then added to the above-described mixture, whereby the pH falls to below 6. The caustic potash is then added and the mixture agitated. The caustic potash reacts with the stearic acid forming a potassium soap and the mixture under agitation becomes a smooth cream. At this time, the lauric-myristic diethanolamide is added, and the mixture is further agitated. The resulting mixture is then cooled to about 135° F. and transferred to a second reaction vessel, and the sulfur and hexachlorophene are then added. The mixture is agitated to disperse thoroughly these additives. The mixture is further cooled to a temperature of about 125° F. at which temperature there is added the monomethyloldimethyl hydantoin and the perfume. The complete product is then cooled to about 85° F. and allowed to cool to room temperature. The product is removed from the second vessel leaving a heel of material in the vessel. This procedure is then repeated without deleteriously affecting the product. The final product is a cream shampoo having an opaque, mother-of-pearl appearance. Cream shampoos of this type may advantageously have a pH of about 8 to 9 varying slightly during agitation, and a high viscosity. The shampoo of this example had a pH of about 8.6 to 8.8. The product is a mild cream, pleasantly fragrant, which, upon use, imparts a high gloss and luster, leaving the hair soft and manageable after removing the grease and debris from the scalp.

Example II.—Cream shampoo

The following describes a second method of forming a cream shampoo in accordance with this invention:

	Percent
Part I	
Stearic acid	6.58
Caustic potash (34% aqueous solution)	4.90
Deionized water	30.23
Part II	
Sodium lauryl sulfate paste [1]	41.73
Triethanolamine lauryl sulfate (42% aqueous solution)	4.50
Deionized water	2.54
Sodium chloride	1.17
Methyl p-hydroxybenzoate	0.14
Sodium betnzoate	0.24
Anhydrous lanolin	0.47
Part III	
Lauric-myristic diethanolamide (71:28)	1.50
Sulfur [2]	4.00
Monomethyloldimethyl hydantoin (25% solution)	0.40
Color	0.10
Hexachlorophene	1.00
Part IV	
Perfume	0.50

[1] The sodium lauryl sulfate paste comprised a mixture of about 40% sodium lauryl sulfate and 60% water.
[2] 98% of the sulfur particles have a size smaller than 0.0004 inch, and the sulfur was dispersed in a 1.5% aqueous solution of sodium lauryl sulfate in the ratio of 1 part sulfur to 1 part of said solution.

The water and potash solution are heated to 145° F. in a first vessel, and simultaneously the stearic acid is separately heated to 160° F. When the stated temperatures are reached, the stearic acid is added to the first vessel.

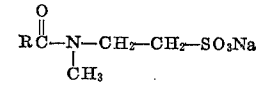
The Part II ingredients are placed in a second vessel and agitated. The agitation is performed at a relatively low speed in order to minimize aeration of the composition. Although it is common practice to use a 30% solution of sodium lauryl sulfate, in this example it is used in the form of a 40% paste to allow the water not present in the paste to be used partially for slurrying the sulfur and partially for increasing the amount of water present in Part I so as to liquefy the soap formed by the reaction of the stearic acid and the potash. Part I is then slowly added, under agitation, to the second vessel, and care is taken during this addition to ensure that the addition of ingredients from the first vessel does not cause the temperature to rise above 120° F. Part III is then added to the second vessel and the batch is cooled to 102° F., at which temperature Part IV is added. The batch is then cooled to 75° F. The final product is a soft, high-quality, desirable cream shampoo having excellent cleaning and foaming properties.

Example III.—Cream shampoo

Another embodiment of a cream shampoo prepared in accordance with the invention is as follows:

	Percent
Fatty acid methyl taurate (34% aqueous solution) [1]	73.1
Sodium lauryl sulfate (50% aqueous solution)	12.2
Lauric-myristic diethanolamide (71:28)	3.0
Anhydrous lanolin	0.5
Polyethylene glycol 600 distearate	1.0
Hexachlorophene	1.0
Sodium chloride	5.0
Deionized water	1.6
Powdered sulfur [2]	2.0
Formaldehyde	0.1
Perfume	0.5

[1] A mixture of compounds of the formula



wherein R is a mixture of the commercial fatty acids obtained from coconut oil and commercial stearic acid in the ratio of about 66 :7 respectively.
[2] In excess of 98% of the particles have a size smaller than 0.0004 inch.

The prepartion of this embodiment of a cream shampoo comprises the steps of mixing the fatty acid methyl taurate, sodium lauryl sulfate, lauric-myristic diethanolamine, anhydrous lanolin, polyethylene glycol 600 distearate, hexachlorophene, and water in a vessel and heating the mixture while under agitation at slow speed to a temperature of between 160–165° F. The sulfur is then added and the mixing speed increased. The composition is thoroughly mixed to ensure adequate dispersion of the sulfur, and the sodium chloride is then added and the composition cooled while stirring. When the composition reaches a temperature of 127° F., the formaldehyde is added; and the perfume is added at 110° F. The agitation is continued until the mixture cools to 90° F. The final product has a pH of 7.1, excellent foaming properties, and is outstanding in its ability to remove foreign matter from the hair and scalp.

Example IV.—Lotion shampoo

An opaque lotion shampoo of desirable consistency is made from the following formula:

	Percent
Part I	
Sodium lauryl sulfate (30% solution)	48.9
Tetra sodium pyrophosphate	0.4
Lanolin	0.5
Polyethylene glycol (600) monostearate	1.0
Ethylene glycol monostearate	4.0
Deionized water	20.0
Part II	
Lauric-myristic diethanolamide (71:28)	5.0
Hexachlorophene	1.0
Part III	
Glycerine	5.0
Deionized water	11.6
Powdered sulfur [1]	2.0
Part IV	
Formalin	0.1
Perfume	0.5
	100.0

[1] In excess of 98% of the sulfur particles have a size smaller than 0.0004 inch.

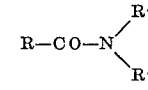
The ingredients of Part I are mixed and heated to a temperature of about 160° F. and this mixture is then cooled to a temperature of about 140° F. at which time Part II is added in the nature of a solution of hexachlorophene in the lauric-myristic diethanolamide. Part III is separately formed by mixing the glycerine and water and forming a slurry of this mixture with the sulfur. Part I is cooled from 140° F. to about 115° F. and transferred to a different reaction vessel at which time Parts III and IV are added. The resulting composition is then cooled to a temperature of about 90° and, if necessary, the pH is adjusted to about 7.5 with citric acid. The resulting shampoo is an opaque, mild, fragrant lotion having a viscosity of about 197 seconds as read on a No. 5 Raymond flowmeter at 77° F., and good sudsing ability in either hard or soft water. The shampoo is effective in cleaning the hair and in the removal of debris and oily deposits therefrom.

The term "soap" as used in the specification and claims refers to the reaction product of one or more higher fatty acids with an alkali. The soaps in the present compositions are water-soluble, and preferably the alkali metal salts of higher fatty acids will be used, such as potassium stearate, sodium stearate, potassium laurate, sodium laurate, sodium oleate, etc. Potassium and sodium salts of higher fatty acids having from 10 to 18 carbon atoms and mixtures thereof may be employed provided that the soap is of suitable solubility. The fatty acid portion of the soap may be a mixture of higher fatty acids of 10 to 18 carbon atoms either saturated or unsaturated or both such as those obtained from the usual soap-making fats such as coconut oil and tallow, or may be a relatively pure fatty acid or mixture such as commercial stearic acid.

Although the present invention has been described with reference to particular embodiments and examples, it will be apparent to those skilled in the art that variations and modifications can be substituted therefor without departing from the principles and true spirit of the invention.

We claim:

1. A method for preparing a shampoo comprising 13.5 to 23 percent by weight of water-soluble anionic sulfonated or sulfated detergent, 1 to 6 percent by weight of an alkylolamide, 6 to 10 percent by weight of water-soluble soap, .5 to 2 percent by weight of hexachlorophene, and 0.5 to 6 percent by weight of sulfur wherein at least 90% of the particles are less than 0.0017 inch, comprising the steps of: blending a higher fatty acid of 10 to 18 carbon atoms and sodium or potassium hydroxide to form a soap in situ in a first vessel under reaction conditions wherein a temperature above about 120° F. and a pH below about 6.0 are employed; transferring the resulting mixture to a second vessel wherein the contents of the first vessel are contacted with the remaining components of the shampoo under conditions which exclude temperatures above about 135° F. and a pH of below about 6.0, the sulfur being present solely in said second vessel, completing the preparation of said shampoo and removing said shampoo from said second vessel leaving therein a heel of product which may become a part of a subsequent batching operation, said water-soluble detergent being selected from the group consisting of water-soluble salts of anionic sulfated or sulfonated detergent salts having an aliphatic chain of 8 to 22 carbon atoms, and said alkylolamide compound being represented by the formula:



wherein R—CO— is a fatty acyl radical of 10–14 carbon atoms, R′ is a hydroxy alkyl group of up to 5 carbon atoms, and R″ is a member selected from the group consisting of R′ and hydrogen.

2. The method of claim 1 wherein said sulfated or sulfonated detergent is initially added to said first vessel.

3. The method of claim 1 wherein said higher fatty acid is stearic acid and said alkali is potassium hydroxide.

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