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[54] **METHOD OF CLEANING HAIR**

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Related U.S. Application Data

[63] Continuation-in-part of Ser. No. 90,835, Nov. 18, 1970, abandoned.

[52] **U.S. Cl.**..... **252/545,** 252/555, 252/DIG. 13

[51] **Int. Cl.**..... **C11d 1/18**

[58] **Field of Search** 252/555, 554, 173, 545, 252/DIG. 13

[56] **References Cited**

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[57] **ABSTRACT**

Hair cleansing with a 14 to 18 carbon olefin sulfonate and a 10 to 15 carbon primary alkanol composition.

6 Claims, No Drawings

METHOD OF CLEANING HAIR

This application is a continuation-in-part of Ser. No. 90,835, filed Nov. 18, 1970, now abandoned.

This invention relates to the cleansing of hair commonly called shampooing in which a composition containing a 14 to 18 carbon alpha-olefin sulfonate and a 10 to 15 carbon primary alkanol is used to produce a rich, stable and creamy wet lather.

A suitable shampoo is different from most commonly used household detergents and must fulfill special requirements, and therefore, information regarding a surfactant's properties in dilute solutions is not generally applicable with regard to its potential as a shampoo. Thus, a shampoo detergent in use must be effective as a rather concentrated solution in water while an ordinary cleansing detergent is used as a very dilute solution in water. Furthermore, the user requires a shampoo which produces a rich, stable foam in copious quantities. Also, although not directly observable, a good shampoo foam must be wet, that is, it must hold a substantial amount of surfactant solution in the foam, since a relatively dry foam has low cleansing properties and poor workability. This is indicated by a creamy lather having small bubbles.

When the composition containing the 14 to 18 carbon alpha-olefin sulfonate and the 10 to 15 carbon primary alkanol composition is used in hair cleansing according to this invention, it produces a creamy wet lather which is stable during the shampooing operation to effectuate good cleansing of the hair. The user is esthetically pleased as he shampoos his hair. Hair which is cleansed in this manner is very manageable and has a good feel to the hand after it has dried in contrast with an alpha-olefin sulfonate composition which does not contain the primary alkanol. Thus the composition containing the higher primary alkanol makes a larger volume of more stable foam, containing a larger amount of entrained surfactant solution. The shampoo composition also is storage stabilized by the addition of the higher primary alkanol to the alpha-olefin sulfonate, which tends to precipitate from solution on standing in the absence of the higher primary alkanol. This storage stability improves its salability and therefore increases the likelihood that this highly effective shampoo will be used to the ultimate benefit of the public.

The alpha-olefin sulfonate surfactant which is used is produced from a straight chain alpha-olefin having 14 to 18 carbon atoms or a mixture of these. The alpha-olefins that are produced by Ziegler telomerization of ethylene are preferred but other alpha-olefins can be used such as those produced by the cracking of paraffin wax. The alpha-olefin should have a purity of at least about 85 percent and preferably at least about 90 percent. Primary impurities are generally internal olefins and vinylidenes. The sulfonation is carried out in a continuous manner in a tube reactor using gaseous sulfur trioxide. A thin, flowing film of alpha-olefin flows down the interior wall of the tube into which the gaseous sulfur trioxide is injected. This gaseous sulfur trioxide is diluted with an inert gas, such as air, to provide a concentration of about two to eight volume percent sulfur trioxide. The sulfur trioxide to alpha-olefin molar ratio is advantageously between about 1:1 and about 1.1:1. The reaction temperature is moderated by passing a coolant at about 40° F. to about 140° F. through cooling jackets appropriately placed about the tube reactor.

A coolant temperature is selected which permits a minimum sulfonation temperature but which allows the sulfonation product to remain in a fluid condition.

The sulfonation product is separated from the gaseous effluent and is generally allowed to age at about 50° F. to about 150° F. This aging is carried out for a sufficient time, usually about 15 minutes to an hour, to permit an undesirable sulfonation species to isomerize to a more desirable species. The sulfonation product is then neutralized by combining it with a potassium, sodium, ammonium or mono, di, or triethanolamine caustic solution in about a 5 to 10 percent excess over that amount which would stoichiometrically be needed to neutralize the SO_3 fed to the tube reactor. This serves to neutralize the free sulfonic acid present and provides sufficient alkali for the subsequent saponification of the sultone co-product. The saponification is effected by heating the aqueous caustic mixture to a temperature of about 285° F. to about 340° F. under autogenous pressure for a period of about 10 to 30 minutes. The aqueous product is cooled and adjusted to a pH of about 8 with aqueous sulfuric acid. The resulting product is an aqueous solution containing about 25 to about 40 weight percent of the alpha-olefin sulfonate. This alpha-olefin sulfonate product is a mixture of the alkali metal, ammonium or ethanolamine salts of alkene sulfonates and hydroxyalkane sulfonates. The alpha-olefin surfactants that were used and are described in the following examples were prepared by this procedure. Alpha-olefin sulfonate as used herein means the product resulting from the above-described procedure.

The primary alkanol which is an essential component of the shampoo composition contains 10 to 15 carbon atoms and has the normal or 2-methyl configuration. Mixtures of two or more of this class of alkanols can also be used. These primary alkanols include 1-decanol, 1-undecanol, 1-dodecanol, 1-tridecanol, 1-tetradecanol, 1-pentadecanol, 2-methyl-1-nonanol, 2-methyl-1-decanol, 2-methyl-1-undecanol, 2-methyl-1-dodecanol, 2-methyl-1-tridecanol, 2-methyl-1-tetradecanol, and the like.

The shampoo composition comprises, as its major components, an aqueous solution of the alpha-olefin sulfonate and the primary alkanol. The amount of the alpha-olefin sulfonate used is from about five to about 40 weight percent and preferably from about 10 to about 35 percent of the solution. The primary alkanol having from 10 to 15 carbon atoms is added as a minor constituent in an amount from about two to about 15 percent based on the alpha-olefin sulfonate, more preferably about 5 to about 10 percent. The primary alkanols having less than 10 carbon atoms are not satisfactory, while the use of primary alkanols having greater than 15 carbon atoms does not exhibit any improvement in foam volume or foam wetness.

Shampoos are a mixture of a suitable surfactant, water and various additives. In using the shampoo solution on the hair it is mixed with an additional amount of water, usually at least in part by prewetting the hair, such that the concentration of the surfactant is generally from about one to about seven percent of the total amount of water present, usually about two to about six percent. Thus, the nature of a shampoo is significantly different from a general cleansing surfactant which is used in solutions many times more dilute. Users of shampoos expect a good shampoo to produce a large quantity of foam which does not break down before the

shampooing is completed. However, it is also necessary for the foam to entrain a substantial amount of the liquid to provide good cleansing. The quality of a shampoo can, in part, be evaluated by a measure of the foam volume that is produced according to standardized procedures together with a measure of the foam wetness. The foam wetness is determined by a measure of the foam drainage time, that is, the time it takes for a predetermined amount of the surfactant solution to drain out of the foam into a separate liquid phase.

The storage stability of the shampoo is evaluated by observing the liquid shampoo composition after prolonged standing for precipitation or a cloudy appearance. In addition to the above characteristics, a good salable shampoo must have appreciable viscosity which is designated as body to those working in the shampoo art. A thin, watery shampoo possesses poor body and is virtually non-salable according to present standards regardless of its shampoo qualities. A viscous shampoo when poured out into the hand is easier to apply to the hair than a water-thin shampoo. The higher primary alkanols having from 10 to 15 carbon atoms possess the additional advantage of substantially increasing the viscosity of the alpha-olefin sulfonate-water solution such that a rich shampoo solution can be formulated therefrom. Unexpectedly, I have discovered that the lower the molecular weight of the primary alkanol, the greater the effect on the viscosity of the shampoo solution, that is, a given quantity of n-decanol will increase the viscosity of the shampoo solution more than the same quantity of n-dodecanol.

The following examples are set out to illustrate the novel shampoo compositions of my invention and to provide a better understanding of its details and advantages.

EXAMPLE 1

In an experimental program a series of determinations were made according to procedures standardized for evaluating shampoo properties to determine the effect of higher primary alkanols on the volume of foam produced by various alpha-olefin sulfonates as well as the foam wetness as determined by the foam drainage time. Three grams of synthetic sebum, a defoaming agent used to simulate hair oils, were added to 100 mil. of an alpha-olefin sulfonate-water solution. In each instance the sodium salt of the alpha-olefin sulfonate was used.

This solution contained sufficient calcium and magnesium salts to constitute 250 p.p.m. of hardness expressed as $CaCO_3$. Various higher alcohols (5 percent based on the sodium salt of the alpha-olefin sulfonate) were added to many of the solutions. Each solution was heated to 100° F. and placed in a 500 ml. graduated cylinder. The solution was agitated for 40 seconds by rotating the cylinder 180° per second after which the volume of foam produced by this agitation was noted. The foam was then permitted to stand until 85 ml. of liquid solution collected at the bottom of the graduated cylinder. The time that was required for this liquid to collect was designated the foam drainage time (F.D.T.). The results of these determinations are set out in Table I. The various alpha-Olefin sulfonate formulations were also compared with formulations of a triethanolamine salt of lauryl sulfate (TLS) which is commonly used in shampoos. The data for the three solutions which do not contain any primary alkanol are the average of two runs.

EXAMPLE 2

Two aqueous shampoo compositions were made up, each one containing 25 weight percent of the sodium salt of an alpha-olefin sulfonate made from an equimolar mixture of 1-tetradecene and 1-hexadecene. The first composition contained no other component while the second also contained five weight percent based on the sulfonate of Lorol 5, a mixture of higher primary alkanols with 1-dodecanol and 1-tetradecanol predominating and made by E. I. duPont de Nemours & Co. Each shampoo was used in the cleansing of hair in a conventional manner as will now be described. The first composition was used in the cleansing of dirty hair by adding a sufficient amount of shampoo composition

Table I

		Surfactant, Weight Percent			
		2.5		6.5	
Surfactant	Alcohol	Foam,ml	F.D.T., sec.	Foam,ml	F.D.T., sec.
C_{14}—C_{16}	—	335	35	418	57
C_{14}—C_{16}	n-Decanol	—	—	485	85
C_{14}—C_{16}	n-Undecanol	—	—	510	93
C_{14}—C_{16}	n-Dodecanol	—	—	460	94
C_{14}—C_{16}	Oxo-undecanol	390	40	510	86
C_{14}—C_{16}	n-Decanol-dodecanol	—	—	495	106
C_{14}—C_{16}	Lauryl	350	37	458	92
C_{14}—C_{16}	Oxo-dodecanol-tridecanol	375	42	500	84
C_{14}—C_{16}	Cetyl	—	—	450	60
C_{14}—C_{16}—C_{18}	—	323	40	428	60
C_{14}—C_{16}—C_{18}	Lauryl	395	69	525	96
TLS	—	290	23	340	45

C_{14}—C_{16} — Olefin sulfonate from a 1/1 molar mixture of n-α-tetradecene and n-α-hexadecene.

C_{14}—C_{16}—C_{18} — Olefin sulfonate from a 1.6/1.4/1 mixture of n-α-tetradecene, n-α-hexadecene and n-α-octadecene.

Oxo-undecanol — 60 percent 1-undecanol and 40 percent 2-methyl-1-decanol.

TLS — Triethanolamine salt of lauryl sulfate.

to the prewetted hair to produce a foam with gentle manipulation of the hair. The hair and scalp were gently manipulated in a manner which is conventional in shampooing hair to produce a foam and to thoroughly work it into and through the hair. After the hair was thoroughly shampooed, it was rinsed, partially dried and then allowed to air dry.

After the same hair had again become dirty, the second composition was used in shampooing the same hair in the same manner as with the first shampoo composition. The foam produced by the second composition was more voluminous, more creamy and of smaller bubble size than the foam produced with the first composition. The dried hair after the second shampooing was very manageable and had a good feel to the hand. The dried hair after the first shampooing was much less manageable and did not have as good a feel as was obtained with the second shampooing.

EXAMPLE 3

The storage stability of a 25 weight percent aqueous solution of the sodium salt of an olefin sulfonate prepared from a 1/1 molar mixture of n-α-tetradecene and n-α-hexadecene was tested with the higher alcohol additive. With no higher alcohol a clear solution was initially produced which slowly formed a precipitate that settled out upon standing. A series of solutions were made up identical to this solution and additionally containing 2.5, 5.0 and 10.0 weight percent n-decanol or n-dodecanol based on the olefin sulfonate. These six solutions were initially clear and were clear without precipitate after standing for one month at room temperature.

EXAMPLE 4

The effect upon viscosity of a 25 percent aqueous solution of the sodium salt of an olefin sulfonate prepared from a 1/1 molar mixture of n-α-tetradecene and n-α-hexadecene was demonstrated by adding varying amounts of higher primary alkanols to the olefin sulfonate solution. The results are set forth in Table II in which the amount of alcohol is base upon the olefin sulfonate.

Table II

	Viscosity, CS. at 25° C.			
Alcohol, Wt.%	0	2.5	5.0	10.0
n-Decanol	14	32	240	8,100
Oxo-undecanol	14	32	165	6,340
Oxo-undecanol[a]	—	—	3,160	14,800
n-Dodecanol	14	32	65	204

[a]30 wt.% aqueous concentration of olefin sulfonate.

These results show that the viscosity of the shampoo product can be readily controlled by means of the amount of the olefin sulfonate and the type and amount of the higher alcohol. Additional experiments show that the higher the molecular weight of the alpha-olefin sulfonate, the greater the viscosity of the solution, other things being equal.

In addition to the olefin sulfonate, higher primary alkanol and water described above, a commercial shampoo incorporating the advantages described herein can include additional constituents as described which are conventional and well known to those of ordinary skill in the shampoo formulating art and do not interfere with the advantageous shampoo properties. For example, it is conventional to incorporate a mild perfume in shampoos and a buffering agent. Also a hair softening material such as lanolin, a hydrolyzed protein or an anti-dandruff agent can be added to the formulation provided that they do not substantially interfere with the desired properties of the finished product.

It is to be understood that the above disclosure is by way of specific example and that numerous modifications and variations are available to those of ordinary skill in the art without departing from the true spirit and scope of the invention.

We claim:

1. A method of cleansing hair which comprises adding to the hair sufficient water and a shampoo composition consisting essentially of an aqueous solution of from about five to about 40 weight percent of an alkali metal, ammonium, or mono, di, or triethanolamine alpha-olefin sulfonate prepared from at least one normal alpha-olefin in having from 14 to 18 carbon atoms and about two to about 15 weight percent based on the alpha-olefin sulfonate salt of a primary normal of 2-methyl alkanol having from 10 to 15 carbon atoms to result in a wet, creamy, stable lather upon manipulation; manipulating said hair containing the shampoo composition and water to produce a wet, creamy, stable lather and cleanse the hair of foreign matter; and water rinsing said foam and foreign matter from said hair.

2. A method of cleansing hair in accordance with claim **1** in which the alpha-olefin sulfonate is about 10 to about 35 weight percent of the shampoo composition.

3. A method of cleansing hair in accordance with claim **2** in which the alkanol is from about 5 to about 10 weight percent based on the alpha-olefin sulfonate.

4. A method of cleansing hair in accordance with claim **1** in which the alkanol is predominately 1-undecyl alcohol.

5. A method of cleansing hair in accordance with claim **1** in which the alkanol is 1-dodecyl alcohol.

6. A method of cleansing hair in accordance with claim **1** in which the alkanol is lauryl alcohol.

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