METHOD OF CLEANING HAIR

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Related U.S. Application Data
Continuation-in-part of Ser. No. 90,835, Nov. 18, 1970, abandoned.

U.S. Cl. .......... 252/545, 252/555, 252/DIG. 13
Int. Cl. ......................... C11d 1/18
Field of Search .......... 252/555, 554, 173, 545,

References Cited
UNITED STATES PATENTS
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3,776,861 12/1973 Mausner et al. 252/555 X

Primary Examiner—Stephen J. Lechert, Jr.

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 alkyl alcohol 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 alkyl alcohol 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 alkyl alcohol. Thus the composition containing the higher primary alkyl alcohol 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 alkyl alcohol to the alpha-olefin sulfonate, which tends to precipitate from solution on standing in the absence of the higher primary alkyl alcohol. 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 vinylidene. 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 5 to 10 percent excess over that amount which would stoichiometrically be needed to neutralize the SO₃ fed to the tube reactor. This serves to neutralize the free sulfonic acid present and provides sufficient alkali for the subsequent saponification of the sulfone 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 alkyl alcohol 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 alkyl alcohols can also be used. These primary alkyl alcohols 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 alkyl alcohol. 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 alkyl alcohol 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 alkyl alcohols having less than 10 carbon atoms are not satisfactory, while the use of primary alkyl alcohols 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 pretreating 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 ml. 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 CaCO₃. 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-tetradecane predominating and made by E. I. du Pont 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>
<thead>
<tr>
<th>Surfactant</th>
<th>Alcohol</th>
<th>Weight Percent</th>
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<td>C₁₄-C₁₆</td>
<td>—</td>
<td>2.5</td>
<td>335</td>
<td>35</td>
<td>418</td>
<td>57</td>
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<tr>
<td>C₁₄-C₁₆</td>
<td>n-Deanol</td>
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<td>485</td>
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**Table I**

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</tr>
</tbody>
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**Table I**
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 thorough
ly 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 sec-
ond composition was used in shampooing the same
hair in the same manner as with the first shampoo com-
position. 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
managable and did not have as good a feel as was ob-
gained 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 \(n\)-\(\alpha\)-tetradecene and
\(n\)-\(\alpha\)-hexadecene was tested with the higher alcohol ad-
ditive. With no higher alcohol a clear solution was ini-
tially produced which slowly formed a precipitate that
settled out upon standing. A series of solutions were
made up identical to this solution and additionally con-
taining 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 \(n\)-\(\alpha\)-tetradecene and \(n\)-\(\alpha\)-hexadecene was demonstrated by adding varying
amounts of higher primary alkanols to the olefin sulfon-
ate solution. The results are set forth in Table II in
which the amount of alcohol is base upon the olefin sul-
fonate.

**Table II**

<table>
<thead>
<tr>
<th>Alcohol, Wt.%</th>
<th>Viscosity, CS. at 25°C.</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>0</td>
</tr>
<tr>
<td>(n)-Decanol</td>
<td>14</td>
</tr>
<tr>
<td>(n)-Octadecanol</td>
<td>14</td>
</tr>
<tr>
<td>(n)-Dodecanol</td>
<td>14</td>
</tr>
</tbody>
</table>

\(30 \text{ 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, oth-
erv things being equal.

In addition to the olefin sulfonate, higher primary al-
kanol and water described above, a commercial sham-
poo 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 interfe-
re with the advantageous shampoo properties. For ex-
ample, 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 an-
ti-dandruff agent can be added to the formulation pro-
vided 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 modifica-
tions 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 add-
ing to the hair sufficient water and a shampoo compos-
tion 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 al-
pha-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 manipula-
tion; manipulating said hair containing the shampoo
composition and water to produce a wet, creamy, sta-
ble 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 composi-
tion.

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-
decleryl 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.

* * * * *