Detergent compositions having improved foam persistence

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No Drawing. Application August 2, 1951, Serial No. 240,056

6 Claims. (Cl. 252—117)

This invention relates to detergent compositions of matter. It is an object of this invention to provide improved detergents, characterized by their ability to produce and maintain copious foam in the presence of greasy soil and by improved ability to emulsify such soil and hold it in suspension. Additional important objects and effects of this invention will appear as the description proceeds.

Detergent compositions such as soap and synthetic detergents have for many years been admixed with various other chemical materials, which either improve the power of the former. Common examples of these materials are sodium sulfate, sodium carbonate, and various polyphosphate esters. These materials are generally referred to in the art as builders, and their principal effect is to improve the cleansing power of the detergent in so far as the power to remove solid soil particles from textile fibers is concerned. Thus, the test commonly applied to detergents containing such adjuvants is generally a measurement of the degree of whiteness imparted to (or light reflected by) a white piece of fabric which has been cleaned by a standard soil prior to testing and which has then been washed for a standard length of time by a standard bath containing the detergent being tested.

Our invention differs from the aforesaid field in that we are interested primarily in improving the capacity of a detergent to remove greasy soil from various articles, not necessarily textiles. A good example of the field in which we are interested is washing dishes by hand or in a dishwashing machine. It is known that the power of a detergent to emulsify greasy soil is related to its power to produce and maintain foam in the presence of greasy soil. The two practically go hand-in-hand, and the one may be taken as a convenient yardstick for the other. Thus, in household use, the housewife is accustomed to adding more detergent when more foam appears from the dishwasher or washing machine. To the detergent manufacturer, it is a persistency of foam in the presence of grease constitutes "mileage" per unit of active component.

Our invention is also not to be confused with the problem of preparing a composition of matter adapted to produce copious foam for its own value, as for instance in fire extinguishers. Such foams are frequently made from non-detergent materials and have no detergent power whatever in the usual sense. Also, in their field of application grease is not ordinarily present; and the problem, generally, is how to produce a maximum volume of foam from a given weight of the composition or in a given time limit, or how to produce a foam of certain qualities for instance as to bubble size, stability in time, etc.

We are not concerned with such problems. Our problem is to endow the composition with such materials that emulsify a maximum quantity of grease, and in so far as foaming is an index to this power, our problem is to produce a detergent which will continue to produce foam until a great many greasy dishes have passed through the washing bath containing the detergent.

Now, our invention is based on the observation that N-dodecylacetamide, i.e., the compound

\[ \text{C}_8\text{H}_{17}-\text{NHCOCH}_3 \]

has excellent properties for increasing the foam persistence of non-cationic detergents. Moreover, it is applicable successfully to a large variety of detergents, including soaps and synthetic detergents, and the percentage of it to be added to a given weight of detergent may be varied within reasonable wide limits.

Our discovery was particularly surprising to us when we found that many compounds of related structure are essentially ineffective and often detrimental to the purposes and objects of this invention. As examples of such ineffective compounds we may mention acetamide, N-hexacylacetamide, N-dodecyl propyramide, and N-dodecyl formamide. On the other hand, N-dodecylacetamide was found by us to give improved mileage with a large variety of non-cationic detergents. More particularly, we have successfully tested this invention with detergents taken from the following distinct classes:

1. Water-soluble salts of the alkyl sulfates (e.g., the sodium salts of the mixed fatty alcohols derived from coconut oil, and the triethanolamine salts of the sulfuric acid esters of alcohols derived from the reduction of sperm oil).

2. Water-soluble salts of the sulfuric acid esters of fatty acid monoglycerides (e.g., the sodium salt of the coconut oil fatty acid ester of 1,2-dihydroxy-propene-3-sulfonic acid).

3. Water-soluble salts of higher fatty acids of monooesters of lower molecular weight hydroxy alkyl and poly-hydroxy alkyl sulfonic acids (e.g., sodium salt of the oleic acid ester of isethionic acid).

4. Water-soluble salts of the higher fatty acid amides of low molecular weight amino-alkyl sulfonic acids (e.g., the sodium salt of the oleic acid amide of N-methyltaurine).

5. Water-soluble salts of alkyl benzene sulfonic acids (e.g., the sodium salt of the sulfonic acid derived from the condensation product of benzene and a propylene reteam, manufactured according to U. S. Patents 2,477,382 and 2,477,383).

6. Water-soluble salts of the higher alcohol esters of sulfo-carboxylic acids (e.g., the sodium salt of the lauryl alcohol ester of sulfoacetic acid).

7. Water-soluble salts of alkyl sulfonates (e.g., the sodium salt of a sulfonated mineral oil containing predominately 14-16 C-atoms).

8. C-substituted alkyl betaines (e.g., C-ethyl betaine made according to U. S. Patent 2,129,264).

9. The non-ionic detergent of the ethylene-oxide alkyl-phenol condensation-product type (e.g., an octyl phenol polyether alcohol containing about eleven ethylene oxide groups).

10. Water-soluble soaps (e.g., the sodium salt of a mixture of fatty acids which contain about 85% of the mixed fatty acids derived from tallow and about 15% of the mixed fatty acids derived from coconut oil). It will be noted that the above typical cationic or non-ionic surface-active agents which, even without the adjuvant of this invention, are capable of producing a persistent foam in the presence of greasy soil. For the purpose of measuring foaming persistence, a special test is detailed hereinbelow. A composition of matter is considered as having good mileage if a 0.1% concentration thereof (based on active ingredient) will produce a persistent foam when shaken in water containing 5 gms. per liter of greasy soil. By the adjuvant of our invention, this tendency is increased in the sense that the foaming power will continue in the presence of larger quantities of greasy soil than are otherwise tolerable by the detergent.

More particularly, we have found that when N-dodecylacetamide is added to a non-cationic detergent in quantity corresponding to about 20% or more by weight of the active ingredient of the detergent, the latter acquires an improved foam persistence in the presence of grease, which can be measured by standard tests as more fully described hereinbelow. These tests are based on the quantity of grease required to be added to a detergent bath containing a specified weight of the detergent to be tested before the bath, upon shaking, ceases to produce a stable foam. By such tests, we found that the quantity added of 20% or more, the foaming capacity of the detergent nearly doubles in most cases and may in some cases reach 12 times the foaming value of the original detergent. Smaller quantities than 20% produce...
a correspondingly decreased improvement, but are nevertheless in most cases effective to a measurable degree. Larger quantities of N-dodecylacetamide may be added, the upper limit being determined by the mutual solubility of the detergent and the active ingredient. It must be explained here that N-dodecylacetamide itself is not soluble in water. The detergent, however, usually exerts some hydrotopic action upon this adjunct and causes it to dissolve or disperse as the aqueous bath in quantities which may reach 60% by weight compared to the weight of the detergent in the case of non-ionic detergents (non ionics). The condensation products of alkylene oxides with alkylated phenols), and 100% or higher in the case of anionic detergents (for instance soaps or sulfates and sulfonates of various organic compounds). Without limiting our invention still, the following examples will serve to illustrate our preferred embodiments thereof. Parts mentioned are by weight, and where a commercial detergent is stated to contain a specified percentage of active ingredient, it is to be understood that the remainder is water and inorganic salts, primarily Na₂SO₄ and NaCl.

Example 1

To 330 parts of a commercial detergent containing as active ingredient 30% of the sodium salts of the sulfonic acid esters of the alcohols obtained by reduction of sperm oil were added 100 parts of N-dodecylacetamide. The compositions were mixed with the detergent at a ratio of 1:1 and washed by the method previously alluded to, it was found to have a foam persistence (or "mileage") about 3.20 times that of the same detergent when it was not admixed with N-dodecylacetamide.

Example 2

Example 1 was repeated except that the active ingredient was the sodium sulfate of the alcohols obtained by reduction of coconut oil, and that the proportion of adjunct to active ingredient was varied from one experiment to another. In one experiment, wherein this proportion was as low as 5:100 (by weight), the improvement in foam persistence was still measurable, and was found by the above test to be about 17% (that is, the treated detergent emulsified about 17% more grease than the untreated detergent).

Example 3

Example 2 was repeated except that 2 parts of a commercial non-ionic surface active agent (an alkylphenol-ethylenc oxide condensate, believed to be isooctyl phenol condensed with about 11 CH₂O units) were admixed to each 100 parts of the active ingredient therein specified. The quantity of N-dodecylacetamide added was varied from one experiment to the other. When this quantity was as low as 4 parts (per 100 parts of active ingredient) the improvement in "mileage" was found to be about 15 to 20%.

Example 4

20 parts of N-dodecylacetamide were added to 360 parts of a commercial detergent containing as active ingredient 30.1% of the sodium salts of the coconut oil mixed fatty acid esters of 1,2-dihydroxypropane-3-sulfuric acid. The mixture thus contained an adjunct:active-ingredient ratio of 20:100 and was found to have an improvement in foam persistence (over the untreated detergent) of about 80%.

Example 5

667 parts of a commercial detergent containing as active ingredient 15% of the sodium salt of the oleic acid amide of N-methyl taurine were mixed with 100 parts of the N-dodecylacetamide. A 1:1 mixture thus obtained was found by our test to be 12 times as great in foam persistence as the corresponding untreated detergent.

Other experiments wherein the adjunct:active-ingredient ratio was as low as 20:100 showed a lesser, but nevertheless marked, improvement.

Example 6

286 parts of a commercial detergent containing as active ingredient 35% of an alkane sodium sulfonate prepared according to U. S. Patent 2,339,974 were mixed with 100 parts of N-dodecylacetamide in quantities, varying over different experiments, from 5 to 100 parts. The composi-

Example 7

(a) A series of experiments as in Example 6 was carried out except that the active ingredient of the detergent employed was C-ethyl betaine (prepared according to U. S. P. 2,129,264). The improvements obtained varied from about 34% to about 217% compared to the untreated detergent.

Similar experiments were further carried out with detergents containing the following active ingredients:

(b) The sodium salt of the lauryl alcohol ester of soapsulfonic acid;

(c) The sodium salt of the oleic acid ester of isethion-

(d) Alkyl-aryl sodium sulfonates prepared according to U. S. P. 2,477,822-3.

Marked improvements in foam persistence were obtained in each case.

(e) Marked improvements were also obtained in a similar series of experiments using a detergent whose active principle was a non-ionic surface-active agent (an alkyl-phenol-ethylene oxide condensate, believed to be isooctyl phenol condensed with about 11 CH₂O units) without exception except that here the ratio of adjunct to active ingredient was varied within the limits of 5:100 to 100:1. It will be noted that the surface-active agent mentioned does not have such a high dispersing effect on N-dodecylacetamide as do the anionic surface-active agents containing sulfates or sulfonates. We found it impossible to increase the quantity of adjunct beyond that which the given quantity of surface-active agent is capable of dispersing (or solubilizing) in the aqueous bath. This limit, in the case of the surfactant agent under discussion, appears to be reached with a ratio of about 60:100.

Example 8

To 105 parts of a commercial flame-salt soap which contains 95% anhydrous sodium soap (made from a mixture of fatty acids comprising 85% fatty acids derived from tallow and 15% fatty acids derived from coconut oil), N-dodecylacetamide was added in quantities varying from 20 to 100 parts. The mixtures (whose ratios thus ranged from 20:100 to 100:100) exhibited marked improvement in foam persistency, the improvements ranging from about 20% to about 100% compared to the untreated soap.

The standard test hereinabove alluded to and by which the foam persisteny of the compositions produced in the above examples was measured is as follows:

A standard greasy soil is prepared as follows:

<table>
<thead>
<tr>
<th>Ingredient:</th>
<th>Parts by weight</th>
</tr>
</thead>
<tbody>
<tr>
<td>Evaporated milk</td>
<td>60</td>
</tr>
<tr>
<td>Flour</td>
<td>40</td>
</tr>
<tr>
<td>Fresh egg yolk</td>
<td>40</td>
</tr>
<tr>
<td>Distilled water</td>
<td>200</td>
</tr>
<tr>
<td>Black India ink</td>
<td>16</td>
</tr>
<tr>
<td>Butter</td>
<td>40</td>
</tr>
<tr>
<td>Lead</td>
<td>40</td>
</tr>
<tr>
<td>Peanut butter</td>
<td>40</td>
</tr>
<tr>
<td>Black printing ink</td>
<td>16</td>
</tr>
<tr>
<td>Salicylanilide</td>
<td>4</td>
</tr>
<tr>
<td>Ammonium hydroxide (28%)</td>
<td>16</td>
</tr>
</tbody>
</table>

A paste of the milk and flour is mixed thoroughly into a blend of the egg yolk and water. The India ink is added, and then the mixture is blended with a melt of the peanut butter, lard and butter containing the printing ink. Finally the salicylanilide in the ammonium hydroxide is added.

B. The detergent composition to be tested is made up into a solution containing 0.1 gm. of the active ingredient per liter of water. The solution is adjusted to a temperature of 24° to 26° C., and 5 gm. portions of the standard greasy soil are added successively, each followed by agitation for 2 minutes. This addition is continued until the foam formed fails to cover the surface of the liquid 30 seconds after agitation has been stopped.

The weight of the soil in grams which can be added to the solution of the detergent before the foam disappears or is broken constitutes a convenient measure of...
the ability of the detergent in question to form a persistent foam in the presence of greasy soil. This property, commonly referred to as "mileage" can be correlated with practice, for example, in the hand washing of dishes. Thus, the detergent product which requires the greatest amount of soil to break the foam in this test will also wash the greatest number of soiled dishes. The N-dodecylacetamide used in this invention can be made by any of the conventional syntheses for amides. For example, it may be prepared by heating dodecyl amine with acetic acid, the water formed by the reaction being distilled off. The N-dodecylacetamide used in the example hereinabove was made in this manner from two commercial grades of dodecyl amine, which contained 85% and 97% of C12 amine, respectively. The C12 portion of each was primarily normal dodecyl (i.e., primary, straight chain).

While a high degree of purity of dodecylacetamide is desirable, a satisfactory quality for the purpose of this invention can also be obtained by use of a grade of amide containing as much as 15% to 20% of other alkyl acetamides in which the alkyl chains may vary down to 10 carbon atoms or up to 18 carbon atoms. Small amounts of the analogous alkyl amides of other fatty acids are also permissible.

The incorporation of our novel adjuvant in the detergent may be done in a variety of optional methods, according to convenience. For instance, the N-dodecylacetamide may be mechanically mixed with or kneaded into the solid detergent; or it may be crutched (mixed) into the detergent in the form of a slurry; or, again, it may be dissolved in an aqueous solution of the detergent. Furthermore, while such ready-for-use mixtures may be manufactured and may be preferable for many purposes, it is also within the scope of our invention to add the N-dodecylacetamide to the aqueous cleansing bath prior to adding the detergent, or vice versa, or to add both N-dodecylacetamide and detergent simultaneously.

Mixtures of several types of the hereinabove indicated detergent compounds may be employed to produce a single detergent composition containing our novel adjuvant, and the customary builders may be added. Other permissible variations will be readily apparent to those skilled in the art.

We claim as our invention:

1. A composition of matter for cleansing greasy articles, comprising a non-ionic, foaming, organic detergent and N-dodecylacetamide, the quantity of the latter being not less than 20% by weight of said detergent and not greater than the quantity capable of being rendered water-soluble through the hydrotropic action of said detergent.

2. A composition of matter adapted for emulsifying greasy soil, comprising an intimate admixture of a non-ionic, foaming detergent and N-dodecylacetamide, the quantity of the latter being not less than 20% and not more than 60% by weight based on the weight of the active ingredient of the detergent, and the entire composition being characterized by improved foaming properties in an aqueous bath containing greasy soil.

3. A composition of matter adapted for emulsifying greasy soil, comprising an intimate admixture of an anionic, foaming detergent and N-dodecylacetamide, the quantity of the latter being not less than 20% and not more than 100% by weight based on the weight of the active ingredient of the detergent, and the entire composition being characterized by improved foaming properties in an aqueous bath containing greasy soil.

4. A composition of matter adapted for emulsifying greasy soil, comprising an intimate admixture of a water-soluble soap and N-dodecylacetamide, the quantity of the latter being not less than 20% and not more than 100% by weight based on the weight of the active ingredient of the soap, and the entire composition being characterized by improved foaming properties in an aqueous bath containing greasy soil.

5. A composition of matter adapted for emulsifying greasy soil, comprising an intimate admixture of a synthetic organic detergent of the alkyl sulfate class and N-dodecylacetamide, the quantity of the latter being not less than 20% and not more than 100% by weight based on the weight of the active ingredient of the detergent, and the entire composition being characterized by improved foaming properties in an aqueous bath containing greasy soil.

6. A composition of matter adapted for emulsifying greasy soil, comprising an intimate admixture of a synthetic organic detergent of the aryl sulfonate class and N-dodecylacetamide, the quantity of the latter being not less than 20% and not more than 100% by weight based on the weight of the active ingredient of the detergent, and the entire composition being characterized by improved foaming properties in an aqueous bath containing greasy soil.

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