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PERSONAL USE DETERGENT LOTION

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This invention relates to opaque liquid detergent compositions especially adapted for personal use and free of soaps of fatty acids.

High quality bar soaps have been favored for years as personal cleansing agents. They are mild to the skin and copiously provide thick, dense lather in use. However, they have the inherent disadvantage of reacting with hard water to form soap curd which deposits in an unsightly form on the wash bowl or bath tub and which leaves a dulling film on hair. Furthermore, soap often may be wasted by the formation of unsightly smears of softened soap on inadequately drained soap dishes. This latter disadvantage can be overcome by employing a dispenser containing a clear liquid soap, usually potassium or sodium-potassium coconut or oleate soap. However, it is difficult to work up a dense thick lather, like that obtainable with bar soaps, with the solutions usually provided; and the problem of soap curd still remains.

The problem of soap curd can be overcome by using a synthetic detergent in bar or liquid form, but new problems arise. Synthetic detergents do not lather on the skin as soaps do; the foam is less dense and thick and has less lubricity. This is particularly noticeable when the synthetic detergent is used on a wash cloth in the shower or bath; for it is difficult to work up a dense soap-like lather under these conditions.

Synthetic detergents do not leave the skin of some people with the same feel as soaps do; after use of a detergent, the skin may tend to feel scroopy. In the properties of the lather and the feel of the skin after washing, the accepted standard of quality, for most people, is the behavior of soap.

Liquid soaps distributed for personal use are usually clear solutions and often contain a little dye added to make them appear more attractive. The opaque creamy appearance of hand lotions and cold creams is taken by many consumers as characteristic of mildness and of luxury products. The chief problems in formulating an opaque creamy product are to avoid excessive dilution by an inert opacifying agent and to achieve a stable dispersion of the opacifying agent.

An object of this invention is to provide a non-soap detergent composition which has at least the mildness to skin of soap and which gives the thick, dense, lubricous lather of soap.

A further object of this invention is to provide a detergent composition with the performance characteristics of a toilet soap but which will not form soap curd-like precipitates in hard water.

A further object is to provide this detergent composition in the form of an opaque, pearlescent, pourable lotion, without the use of an inert diluent.

An additional object is to provide a detergent lotion which leaves the skin feeling lubricous—but not oily—and not scroopy.

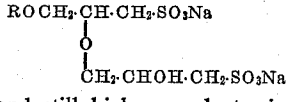
These and other objects can be achieved by a composition containing the following essential active ingredients: (1) a mixture of alkyl glyceryl ether sulfonate salts; (2) a long chain tertiary amine oxide; (3) an auxiliary anionic sulfated or sulfonated detergent salt; and (4) a water soluble polymer, as hereinafter specified. The essential active ingredients, inorganic salts, and other desirable ingredients are dissolved or suspended in an aqueous vehicle. The use of the hereinafter specified

quantity of each essential ingredient achieves the objects of the invention.

The identities and concentrations of the ingredients will now be specified in greater detail. Weight percent will be used throughout in giving the concentrations of the various ingredients. The basis for the percentages is the total formulated detergent composition unless otherwise indicated. It should be pointed out that in an aqueous solution it is not possible to state which cations are associated with which anions; for all constituents are dissolved in the manufacture, and there is an interchange of ions among the ingredients. Thus, the overall composition can be achieved in many ways by using different combinations of the same sets of cations and anions.

Sodium ion can be used as the sole metallic cation in this invention; but it is preferable to use a mixture of sodium and potassium. Potassium has the effect of increasing the viscosity of the product which contributes to the stability of the dispersion of opacifier. Low molecular weight alkanolamines, such as ethanolamine, diethanolamine, and triethanolamine, can be used as total or partial replacements for potassium. The mole ratio of potassium (or alkanolammonium) to sodium should not exceed 0.8 and preferably should not exceed about 0.75 so that the opacity of the formula can be maintained up to 100 to 100° F. At higher ratios and elevated temperatures the opacifier tends to dissolve.

The alkyl glyceryl ether sulfonate salts employed in the compositions of this invention as the chief lathering and cleaning agent can be prepared by reacting fatty alcohols with a slight excess of epichlorohydrin and then sulfonating the resulting chloroglyceryl ethers by means of the Streckerization reaction. The primary product is alkyl monoglyceryl ether sulfonate, using the sodium salt as an example, $ROCH_2 \cdot CHOH \cdot CH_2 \cdot SO_3Na$. Secondary reaction products, such as alkyl diglyceryl ether disulfonate,



are formed; and still higher products, in amounts less than about 3% of the total alkyl glyceryl ether sulfonate, are formed. Other isomers of the diglyceryl ether disulfonate than the one illustrated are also formed and may be mono- or disulfonated. A further disclosure of these detergents and methods for their production is given in co-pending application Serial No. 659,038, filed May 14, 1951 in the name of D. D. Whyte et al. For the purposes of this invention, the sodium or potassium alkyl glyceryl ether sulfonate should preferably contain less than 30% of the diglyceryl ether sulfonate and higher products. As a practical matter it is often more expensive to make a product containing no diglyceryl ether sulfonate, and alkyl glyceryl ether sulfonate containing about 23% diglyceryl ether sulfonate is entirely suitable. Note that the use of "alkyl glyceryl ether sulfonate" is intended to include monoglyceryl, diglyceryl (mono- and disulfonated), and the traces of higher glyceryl compounds.

The alkyl groups of the alkyl glyceryl ether sulfonates should be substantially saturated and should contain from about 10 to about 18 carbon atoms. A substantial fraction of the alkyl glyceryl ether sulfonates should contain from 12 to 14 carbon atoms in the alkyl radical, for such detergents are quite soluble; a smaller fraction should contain from 16 to 18 carbon atoms in the alkyl radical in order to provide, by virtue of their insolubility in the formulated detergent, the opaque pearlescent lotion-like appearance. The alkyl radicals of both fractions can each consist of a single chain length or a mixture of chain lengths. When the formula is diluted by water in use, the

opacifier is at least in part dissolved and contributes to lathering.

Middle cut coconut fatty alcohols are a convenient source of a mixture consisting predominantly of C_{12} and C_{14} alkyl radicals; a typical analysis is 2% C_{10}, 66% C_{12}, 23% C_{14}, and 9% C_{16}. Hydrogenated tallow fatty alcohols are a convenient source of a mixture consisting predominantly of C_{16} and C_{18} alcohols; a typical analysis is: 2% C_{14}, 32% C_{16}, and 66% C_{18}. The higher cut of coconut fatty alcohols, called the Stenol cut, is another convenient source of predominantly C_{16} and C_{18} alcohols. There are, of course, other natural and synthetic sources of lauryl, myristyl, cetyl and stearyl alcohols which can provide the alkyl radicals of the alkyl glyceryl ether sulfonates used in the formulas of this invention. From about 6% to about 15% (of the total formulation) of sodium or potassium alkyl glyceryl ether sulfonate, wherein the alkyl radicals range from 10 to 14 and contain predominantly from 12 to 14 carbon atoms, and from about 2% to about 4% of sodium or potassium alkyl glyceryl sulfonate, wherein the alkyl radicals contain predominantly from 16 to 18 carbon atoms are used in the compositions of this invention. The preferred compositions contain about 6% to about 10% of predominantly from C_{12} to C_{14} compounds and approximately 3% of from C_{16} to C_{18} compounds.

For example, from about 6% to about 15% sodium or potassium middle cut coconut glyceryl ether sulfonate mixed with from about 2% to about 4% of sodium or potassium tallow glyceryl ether sulfonate is suitable for the formulations of this invention. Below the lower limit of 2% of the C_{16} and C_{18} alkyl glyceryl ether sulfonates, a desirable pearlescent appearance is not developed; above the 4% upper limit no further advantage in appearance is obtained, and the less soluble alkyl glyceryl ether sulfonates become too large a fraction of the total.

It is immaterial whether the fatty alcohols are mixed before preparation of the alkyl glyceryl ether sulfonates or whether the C_{12}—C_{14} alkyl glyceryl ether sulfonates and the C_{16}—C_{18} alkyl glyceryl ether sulfonates are prepared separately and then are mixed. It is preferable to control the sodium-potassium ratio in the formulas of this invention by adding part of the alkyl glyceryl ether sulfonate as the sodium salt and part as the potassium salt. An especially convenient way to control the sodium-potassium ratio is to add not more than 0.7 by weight of the form C_{12} to C_{14} alkyl glyceryl ether sulfonate as the potassium salt, and, as is hereinafter described, to add pyrophosphate as the potassium salt and to add all the other ionic ingredients as sodium salts.

Long chain tertiary amine oxides are employed in the formulas of this invention because they were discovered to have a beneficial effect in increasing the volume of lather and in enhancing the soap-like character of the lather. They correspond to the general formula

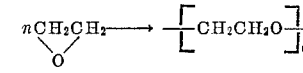
$$R_1R_2R_3N\rightarrow O$$

R_1 is an alkyl radical of from about 8 to 18 carbon atoms, and R_2 and R_3 are each methyl or ethyl radicals. More than about 50% of the amine oxide should have R_1 12 carbon atoms in chain length. The arrow in the formula is a conventional representation of a semi-polar bond. Examples of amine oxides suitable for use in this invention include mixtures of dimethyldodecylamine oxide with dimethyloctylamine oxide, dimethyldecylamine oxide, dimethyltetradecylamine oxide, dimethylhexadecylamine oxide and dimethyloctadecylamine oxide. Whole cut coconut dimethylamine oxide is suitable for use in this invention. The approximate chain length composition is: C_6—C_{10} 15%, C_{12} 50%, C_{14} 17%, C_{16} 7%, and C_{18} 11%. Especially preferred are amine oxides in which R_1 is predominantly of chain length from C_{12} to C_{14}, for example, dimethyldodecylamine oxide or middle cut coconut dimethylamine oxide. Additional specific examples of suitable amine oxides are the compounds just listed, in which one or both of the methyl radicals are replaced by ethyl radicals. For example, methylethyldodecylamine oxide and diethyldodecylamine oxide are suitable for use in the formulas of this invention.

More than about 1% amine oxide, preferably about 2%, is required to achieve the desired effect on lathering; generally there is little further enhancement of lathering when more than 5% is used. Also higher levels of amine oxide cause undue solubilization of the portion of the alkyl glyceryl ether sulfonate which is intended to be insoluble and act as an opacifier in the formulas of this invention. This solubilization can be counteracted by increasing the level of inorganic salt in the formulas.

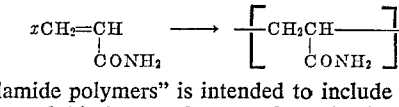
Polymeric materials are employed in the compositions of this invention to provide lubricity to the lather and to leave the skin feeling smooth and not scroopy after use. They must meet requirements of water solubility, mildness, low toxicity, and paleness of color as well as producing the desired effects. Many polymeric materials have some effect on the feel of the lather and skin, for example carboxymethylcellulose, but two types of polymers are outstanding in this respect and are preferred for incorporation into the compositions of this invention. They will be referred to as "ethylene oxide polymers" and "acrylamide polymers." They are more fully described hereinafter.

By "ethylene oxide polymers" is meant polymers of ethylene oxide of very high molecular weight. Their preparation can be represented in a formal way:



The value of n will be of the order of 10,000 to 100,000. It is convenient, as is often done in polymer chemistry, to specify viscosity of a solution of a given concentration and thus specify indirectly the approximate magnitude of n or the size of the polymer. For the purposes of this invention, the ethylene oxide polymers when dissolved in distilled water at the level of 1% should have a viscosity of greater than 10 centipoises at 25°. Ethylene oxide polymers effective in the practice of this invention may have a viscosity higher than 5500 centipoises under the conditions specified, but they must be soluble in water. These viscosity values are as determined with a Brookfield Viscometer RVF at 2 r.p.m. using the largest spindle for the desired scale.

Preparation of the acrylamide homopolymer, or polyacrylamide, can be represented in a formal way:



"Acrylamide polymers" is intended to include in addition to water-soluble homopolymers of acrylamide, water-soluble copolymers of acrylamide with: (1) up to about 15% by weight of the total polymer of one of the following, methacrylamide, acrylonitrile, vinylchloride, vinylalkyl ethers, lower alkyl esters of acrylic acid, and lower alkyl esters of methacrylic acid; or (2) up to 50 mole percent of one of the following, acrylic acid, methacrylic acid, alkali metal salts of acrylic acid, or alkali metal salts of methacrylic acids. The copolymers are suitable for the practice of this invention, and indeed pure acrylamide homopolymer is difficult to obtain, there usually being some acrylic acid present in commercial acrylamide and some hydrolysis being likely to occur in storage and handling of the polymer. The monomer content of the acrylamide polymers or copolymers is held to less than 1% and preferably less than 0.05% of acrylamide monomer. These polymers and copolymers are of such a molecular weight that they are characterized by a viscosity of at least 4 centipoises when they are dissolved at the level of 0.5% in a 4% sodium chloride solution at pH 5 to 6. A salt solution is employed rather than water to eliminate the effect of charge on viscosity encountered

with ionic polymers. While it is not necessary to measure the viscosity in salt solution when the polyacrylamide is less than about 10% hydrolyzed, i.e. when it is substantially nonionic, it is convenient to do so. The viscosities of acrylamide polymers are as determined by measuring the kinematic viscosity by ASTM Method D445–53T (ASTM Standards, 1955, part 5, pp. 192–202) and converting it to absolute viscosity. The absolute viscosity in centipoises is obtained from the kinematic viscosity in centistokes by multiplying by the specific gravity of the test solution which is taken as one. The requirement of water solubility places an upper limit on the molecular weight of the polymers which are desirably employed. Some specific examples of acrylamide polymers suitable for use in this invention and characterized by a viscosity of 0.5% solutions as described above are: a polymer of 97 mole percent acrylamide and 3 mole percent acrylic acid—viscosity about 5 centipoises; a polymer of 50 mole percent acrylamide and 50 mole percent acrylic acid—viscosity about 15 centipoises; a polymer of 90 mole percent acrylamide and 10 mole percent acrylic acid—viscosity about 10 centipoises; a polymer of 95 weight percent acrylamide and 5% acrylonitrile—viscosity about 5 centipoises; and a polymer of 95 weight percent acrylamide and 5% methacrylamide—viscosity about 9 centipoises.

About 0.05% of acrylamide polymer or ethyleneoxide polymer is required to give a noticeable effect and more than about 0.2% tends to give an undesirably slippery or oily effect. Preferably about 0.1% is employed.

While alkyl glyceryl ether sulfonate could be used as the sole anionic detergent, it is preferable to include from about 1% to about 5% of a supplementary anionic nonsoap synthetic detergent. The supplementary detergent is selected to promote rapid lathering, to increase the concentration of active that can be incorporated in a liquid of satisfactory physical properties, and in some circumstances to reduce the cost of the formulation. Alkali metal salts, such as sodium and potassium salts, of the supplementary detergent are suitable and if alkanolammonium cations (e.g. ethanolammonium, diethanolammonium, and triethanolammonium) are to be introduced, it is often convenient to do so by means of an alkanolammonium salt of the supplementary detergent. The supplementary detergent salt should be soluble in the formulas of the invention and should have pronounced sudsing characteristics. Suitable detergents include those having in their molecular structure an alkyl radical of from about 8 to about 18 carbon atoms with the ionic character being supplied by a sulfonate or sulfate radical. The salts which find greatest utility, with exceptions to be pointed out, are those which have from 10 to 16 carbon atoms and predominantly about 12 carbon atoms in the alkyl radical; but since other constituents of the detergent molecule can be varied to increase or decrease the degree of solubility, the number of carbon atoms in the alkyl radical can also be adjusted upwardly in such instances to as high as 18 or downward to as low as 8, for example. Examples of the synthetics which can be used in the composition of the present invention are sodium or potassium alkyl benzene sulfonates in which the alkyl groups contain from about 9 to about 15 carbon atoms; sodium, potassium or alkanolammonium (mono-, di-, and triethanolammonium) alkyl sulfates, especially those derived by sulfation of higher alcohols produced by reduction of glycerides of coconut oil; sodium coconut oil fatty acid monoglyceride sulfates and sulfonates; sodium, potassium and mono-, di-, and triethanolammonium salts of sulfuric acid esters of the reaction product of one mole of coconut oil fatty alcohol and about 1 to 5 moles of ethylene oxide or of one mole of tallow fatty alcohol and about 10 moles of ethylene oxide; the reaction product of fatty acids esterified with isethionic acid and neutralized with sodium hydroxide where, for example, the fatty acids are derived from coconut oil; sodium or potassium salts of fatty acid amide of a methyl taurine in which the fatty acids, for example, are derived from coconut oil; sodium, potassium, and mono-, di-, and triethanolammonium salts of the sulfated condensation product of nonylphenol with 2 to 6 moles of ethylene oxide; and others known in the art.

The anionic synthetic detergents preferred for use as supplementary detergents in the formulas of this invention are: sodium and potassium alkyl sulfate wherein the alkyl radical contains predominantly 12 to 14 carbon atoms—as an example potassium middle cut coconut alkyl sulfate; sodium and potassium alkyl benzene sulfonate wherein the alkyl radical contains from 9 to 15 carbon atoms—as an example potassium tetrapropylene benzene sulfonate; the sodium salts of the sulfated condensation product of predominantly C_{12}, lauryl, and C_{14}, myristyl, alcohols with an average of three moles of ethylene oxide per mole of alcohol—as an example the sodium salts of sulfated condensation product of one mole of middle cut coconut fatty alcohol with about 3 moles of ethylene oxide. Especially preferred because of its mildness to the skin and its high solubility is the sodium, potassium or triethanolammonium salt of the sulfated condensation product of one mole of nonylphenol with about four moles of ethylene oxide.

The anionic synthetic detergents bring inorganic salts such as sodium or potassium chloride or sulfate into the composition. When these are dissolved, they tend to serve a useful purpose by increasing the density of the solution and thereby increasing the stability of the dispersion of the opacifier. They also tend to salt-out a portion of alkyl glyceryl ether sulfonate of longer chain length, keeping it in the solid state and available as opacifier. The compositions of this invention will ordinarily contain from about 1% to about 3% of sodium and potassium chlorides and sulfates introduced with the anionic detergents. In order to exercise more positive control, it is preferable to add additional alkali metal salts of inorganic or low molecular weight organic acids. Among those particularly suitable may be mentioned sodium or potassium chloride, sulfate, orthophosphate, pyrophosphate, acetate and mixtures thereof. The salt should be soluble, at the level used, in the formulation and should not be objectionable as to odor, toxicity, and mildness to the skin.

Potassium pyrophosphate is especially preferred; for in addition to the other effects, it appears to improve the quantity and quality of the lather. Potassium pyrophosphate can be included whether or not other salts are added. About 1% $K_4P_2O_7$ is preferably used; below about 1% there is a tendency for the opacifier to settle, and above about 3% there is a tendency for the product to gel at higher temperatures.

When the amine oxide content is relatively high, e.g. 3–5%, another salt, in addition to potassium pyrophosphate is preferably added. Up to about 5% (total of that brought in with the detergent and that added) of one of the other salts listed above serves to counteract the solubilizing effect of the amine oxide on the opacifier. The total of potassium pyrophosphate and the additional salt is preferably kept under about 8%.

Small quantities of minor ingredients can be added to the formulas of this invention to enhance its performance, physical properties, or esthetic appeal. Sodium carboxymethylcellulose is preferably added at levels of about 0.1% to about 0.4%. The water soluble, high or medium viscosity types are employed and help to control viscosity of the formulation in order to reduce settling and in order to control the flow characteristics of the product as it is poured or otherwise dispensed. Sodium carboxymethylcelluloses are conveniently characterized by degree of substitution (average number of carboxymethyl groups per glucose unit) and by viscosity of a solution of specified concentration. The materials preferred for use in this invention will have a degree of substitution between about 0.65 and 1.4. The preferred viscosity grades will be from about 200 centipoises for a 2% solution at 25° C. to about

3000 centipoises at 25° C. Carboxymethylcellulose is also thought to have some beneficial effect in improving the feel of the lather. The product may be colored by a trace of a soluble dye and preferably will be perfumed. Sodium or potassium silicate can be added in quantities up to about 3% to inhibit corrosion of metal containers. Preferably the SiO_2/Na_2O weight ratio is above 2.8, for example about 3.2.

Small quantities of alkali metal hydrotrope salts, such as potassium or sodium benzene sulfonate, toluene sulfonate and xylene sulfonate can be added to thin the product. While not essential, preferably about 0.5% to about 3% will be added depending on the levels of the other ingredients and the viscosity desired.

The usual process of manufacture is to mix all ingredients together and to dissolve them by heating the mixture to about 160° F. The solution is then quickly cooled, for example by pumping through a heat exchanger, to about 70° F. Rapid cooling produces fine crystals of the C_{16}—C_{18} alkyl glyceryl ether sulfonate which have less tendency to settle out than coarser crystals do.

The Saybolt furol viscosity of the product is from 150 to 750 sec. at 75° F.; viscosities around the middle of this range are preferred.

Specific formulas illustrative of the invention will now be given.

While properties of lather worked up in hand washing or in the bath are difficult to measure quantitatively, as is the feel of the skin after washing, the effects are real. They are best evaluated by a panel of judges who rate their impressions on an arbitrary scale. The resulting grades then can be averaged for comparison of products. The formulas of the examples were evaluated by a panel in this fashion; each member of the panel graded his impression of (1) speed of lathering and volume, (2) creaminess, lubricity, and feel of lather, and (3) skin feel after washing.

Example I

Several formulations of this invention were prepared as hereinbefore described and were examined and tested.

	A, percent	B, percent
Potassium coconut[1] glycerol ether sulfonate (about 23% diglyceryl and the balance substantially all monoglyceryl)	4.0	5.0
Sodium coconut[1] glyceryl ether sulfonate (diglyceryl and monoglyceryl content as above)	4.0	4.0
Sodium tallow glyceryl ether sulfonate (diglyceryl and monoglyceryl content as above; the tallow alkyl radicals correspond to those of substantially saturated tallow alcohols containing approximately 2% C_{14}, 32% C_{16}, and 60% C_{18})	3.0	3.0
Coconut[1] dimethylamine oxide	2.0	2.0
Sodium salt of sulfated condensation product of one mole of nonylphenol with 4 moles ethyleneoxide	2.0	2.0
Potassium pyrophosphate	1.0	1.0
Sodium toluene sulfonate	1.5	2.0
Sodium carboxymethylcellulose (degree of substitution, 0.65–0.95; viscosity of 1% soln., 1,000–2,800 cps. at 25° C.)	0.2	0.2
Acrylamide polymer (contains 5–10 mole percent acrylic acid radicals; monomer is about 0.02%; viscosity of a 0.5% soln. is 6–10 centipoises at 25° C.)	0.1	0.1
Color	0.0001	0.0001
Perfume	0.5	0.5
Salts (sodium and potassium chloride and sulfate from detergents)	1–2	1–2
Water	Balance	Balance

[1] Coconut indicates alkyl radicals corresponding to those of middle cut coconut fatty alcohol containing approximately 2% C_{12}, 66% C_{12}, 23% C_{14}, and 9% C_{16}.

"A" was varied by using 0.15% and 0.20% acrylamide polymer as well as 0.10%. "B" was varied by omitting the sulfated nonylphenolethyleneoxide detergent. "A" was varied by using dimethyldodecylamine oxide instead of coconut dimethylamine oxide.

The above formulas were stable pourable liquids having an attractive opaque pearlescent appearance.

When portions of 1 ml. to 3 ml. were used for hand washing, a thick lubricous lather at least equivalent to that obtained from a quality soap bar was obtained. After the hands were dried they felt smooth and not scroopy. No soap curd or other objectionable precipitate was formed in hard water. The formulas were substantially as mild to the skin as high quality bar soap.

The formulas also proved suitable for working up a lather when used on a wash cloth in the bath and left no ring of soap curd on the tub. Also they performed well as a bubble bath, that is when put in the tub while the water was being run in, the water then produced copious suds, sufficiently long lasting for the purpose. The formulas proved to be useful as a shampoo and left the hair free of dulling soap films.

These formulas are effective cleaners for heavily soiled greasy or oily hands. The liquid is applied undiluted to solubilize and emulsify the oil and then is washed off.

Example II

The three formulas set forth below were prepared and were found to be at least the substantial equivalent in appearance and performance of the formulas of Example I.

	A, Percent	B, Percent	C, Percent
Potassium coconut[1] glyceryl ether sulfonate (about 23% diglyceryl and the balance substantially all monoglyceryl)	5.0	8.0	10.0
Sodium coconut[1] glyceryl ether sulfonate (diglyceryl and monoglyceryl content as above)	4.0	4.0	4.0
Sodium tallow glyceryl ether sulfonate (diglyceryl and monoglyceryl content as above; the tallow alkyl radicals correspond to those of substantially saturated tallow alcohols containing approximately 2% C_{14}, 32% C_{16}, and 60% C_{18})	3.0	3.0	3.0
Dimethyldodecylamine oxide	2.0	2.0	2.0
Sodium salt of sulfated condensation product of one mole of nonylphenol with 4 moles ethyleneoxide	2.0	2.0	2.0
Potassium pyrophosphate	1.0	1.0	1.0
Hydrotrope[2]	2.0	1.0	2.0
Sodium carboxymethylcellulose (degree of substitution 0.65–0.95; viscosity of 1% soln., 1,000–2,800 cps. at 25° C.)	0.2	0.2	0.2
Acrylamide polymer (contains 5–10 mole percent acrylic acid radicals; monomer is about 0.02%; viscosity of a 0.5% soln. is 6–10 centipoises at 25° C.)	0.1	0.1	0.1
Color	0.0001	0.0001	0.0001
Perfume	0.5	0.5	0.5
Salts (sodium and potassium chloride and sulfate from detergents)	1–2	1–2	1–2
Water	Balance	Balance	Balance

[1] Coconut indicates alkyl radicals corresponding to those of middle cut coconut fatty alcohol containing approximately 2% C_{10}, 66% C_{12}, 23% C_{14}, and 9% C_{16}.

[2] "A" contained sodium toluene sulfonate; "B" and "C" contained sodium xylene sulfonate.

Whole cut coconut dimethylamine oxide can be substituted for the dimethyldodecylamine oxide without substantially adversely affecting the appearance or properties of these products.

Example III

The following formulas containing different supplementary detergents were prepared and evaluated.

Sodium coconut[1] glyceryl ether sulfonate (about 23% diglyceryl and the balance substantially all monoglyceryl)	7.0
Sodium tallow glyceryl ether sulfonate (diglyceryl and monoglyceryl content as above)	3.0
Dimethyldodecylamine oxide	2.0
Supplementary detergent[2]	2.0
Potassium pyrophosphate	1.0
Acrylamide polymer (contains 5–10 mole percent acrylic acid radicals; monomer is less than 0.15%; viscosity of a 0.5% soln. is 6–10 centipoises at 25° C.)	0.05
Sodium carboxymethylcellulose (degree of substitution 0.65–0.95; viscosity of 1% soln., 1000–2800 cps. at 25° C.)	0.3

Example III—Continued

Sodium carboxymethylcellulose (degree of substitution 0.65–0.95; viscosity of 2% soln., 200–800 cps. at 25° C.)	0.5
Color	0.0001
Perfume	0.2
Salts (sodium chloride and sulfate from detergents)	1–2
Water	Balance

[1] Coconut indicates alkyl radicals corresponding to those of middle cut coconut fatty alcohol containing approximately 2% C_{10}, 66% C_{12}, 23% C_{14} and 9% C_{16}.

[2] The supplementary detergents which were employed were: (A) sodium salt of the sulfated condensation product of nonylphenol with four moles of ethylene oxide, (B) sodium salt of the sulfated condensation product of one mole of middle cut coconut fatty alcohol with three moles ethylene oxide, and (C) sodium middle cut coconut alkyl sulfate.

These formulas gave products of as good appearance as those of Example I. In performance A and C were substantially equivalent to the formulas of Example I, Formula B, with alkyl ethylene oxide sulfate as a supplementary detergent, was equivalent in appearance and satisfactory in performance. Sodium N-methyl-N coconut oil fatty acid taurate or the coconut oil fatty acid of sodium isethionate can be used as the "supplementary detergent" without substantially adversely affecting the appearance or performance of the product.

Example IV

The following is set forth as an example of a formula with a high amine oxide content. Salt is added to counteract the solubilizing effect of the amine oxide on the tallow alkyl glyceryl sulfonate opacifier.

Potassium coconut [1] glyceryl ether sulfonate (about 23% diglyceryl and the balance substantially all monoglyceryl)	3.0
Sodium coconut [1] glyceryl ether sulfonate (diglyceryl and monoglyceryl content as above)	4.0
Sodium tallow glyceryl ether sulfonate (diglyceryl and monoglyceryl content as above; the tallow alkyl radicals correspond to those of substantially saturated tallow alcohols containing approximately 2%–C_{14}, 32%–C_{16}, and 60%–C_{18})	3.0
Coconut [1] dimethylamine oxide	5.0
Sodium salt of sulfated condensation product of one mole of nonylphenol with 4 moles ethylene oxide	2.0
Potassium pyrophosphate	1.0
Sodium chloride (in addition to that from detergents)	3.0
Sodium toluene sulfonate	2.5
Sodium carboxymethylcellulose (degree of substitution 0.65–0.95; viscosity of 1% soln., 1000–2800 cps. at 25° C.)	0.3
Acrylamide polymer (contains 5–10 mole percent acrylic acid radicals; monomer is less than 0.05%; viscosity of a 0.5% soln. is about 10–15 centipoises at 25° C.)	0.1
Salts (sodium and potassium chloride and sulfate from detergents)	1–2
Water	Balance

[1] Coconut indicates alkyl radicals corresponding to those of middle cut coconut fatty alcohol containing approximately 2% C_{10}, 66% C_{12}, 23% C_{14} and 9% C_{16}.

This formula gives an opaque pearlescent liquid of pleasing appearance. In use it gives a thick dense lather and leaves the skin feeling smooth. It is eminently suitable for personal use.

Acrylamide polymers containing (*a*) 40 mole percent acrylic acid radicals; (*b*) containing 10 mole percent methacrylic acid radicals; or (*c*) 10 mole percent sodium salt of methacrylic acid radicals; but otherwise similar, can be substituted for the acrylamide polymer of this example without substantially affecting the appearance or performance of this composition.

Example V

Compositions of this invention with levels and varying viscosity grades of ethyleneoxide polymers were prepared and evaluated. The basic formula was as follows:

Potassium coconut [1] glyceryl ether sulfonate (about 23% diglyceryl and the balance substantially all monoglyceryl)	3.0
Sodium coconut [1] glyceryl ether sulfonate (diglyceryl and monoglyceryl content as above)	4.0
Sodium tallow glyceryl ether sulfonate (diglyceryl and monoglyceryl content as above; the tallow alkyl radicals correspond to those of substantially saturated tallow alcohols containing approximately 2% C_{14}, 32% C_{16}, and 60% C_{18})	3.0
Sodium salt of sulfated condensation product of one mole of nonylphenol with 4 moles of ethylene oxide	2.0
Coconut [1] dimethylamine oxide	2.0
Potassium pyrophosphate	1.0
Sodium toluene sulfonate	1.0
Sodium carboxymethyl cellulose (degree of substitution 0.65–0.95; viscosity of 1% soln., 1000–2800 cps. at 25° C.)	0.3
Eethylene oxide polymer (as indicated below)	0.05, 0.1
Color	0.0001
Salts (sodium and potassium chloride and sulfate from detergents)	1–2
Perfume	0.5
Water	Balance

[1] Coconut indicates alkyl radicals corresponding to those of middle cut coconut fatty alcohol containing approximately 2% C_{10}, 66% C_{12}, 23% C_{14} and 9% C_{16}.

At both levels of ethylene oxide polymer, 0.05% and 0.1%, each of the three viscosity grades of ethylene oxide polymer were tested. They were:

	Approx. viscosity of 1% soln. at 25° C., centipoises
A	[1] 15
B	3000
C	5500

[1] (About 2000 in 5% soln.)

Each of six products were stable pourable liquids having an attractive opaque pearlescent appearance.

When used for hand washing, a thick lubricous lather at least equivalent to that obtained from bar soap was obtained. After the hands were dried they had a pleasant smooth feeling and were not scroopy.

The liquid form of this product adapts it for automatic dispensers and for various types of deformable plastic containers which can be squeezed to discharge the product. It is also well adapted for being dispensed from a pressurized package. The product can be discharged as a liquid or as a lather. The pressure generating agent can be the type of fluorinated and chlorinated hydrocarbons usually employed for such purposes.

What is claimed is:

1. An opaque pearlescent liquid detergent composition having a lotion-like consistency consisting essentially of: (1) from about 6% to about 15% of a detergent selected from the group consisting of sodium and potassium alkyl glyceryl ether sulfonates having an alkyl radical ranging in chain length from 10 to 14 carbon atoms; (2) from about 2% to about 4% of said sulfonates having an alkyl radical ranging in chain length from 16 to 18 carbon atoms; (3) from about 1% to about 5% of an amine oxide having the formula $R_1R_2R_3N\rightarrow O$ in which R_1 is an alkyl radical ranging in chain length from 8 to 18 carbon atoms and R_2 and R_3 are each selected from the group consisting of methyl and ethyl radicals, at least about 50% of said amine oxide havin R_1 12

carbon atoms in chain length; and (4) from about 0.05% to about 0.2% of a water soluble polymer selected from the group consisting of acrylamide polymers having a viscosity of at least 4 centipoises at 25° C. when dissolved at the 0.5% level in 4% sodium chloride solution adjusted to a pH of 5 to 6, and ethylene oxide polymers having a viscosity of at least 10 centipoises at 25° C. when dissolved in water at the 1% level, the ingredients being in an aqueous vehicle and the mole ratio of potassium to sodium being less than about 0.8.

2. An opaque pearlescent liquid detergent composition having a lotion-like consistency consisting essentially of: (1) from about 6% to about 15% of a detergent selected from the group consisting of sodium and potassium alkyl glyceryl ether sulfonates having an alkyl radical ranging in chain length from 10 to 14 carbon atoms; (2) from about 2% to about 4% of said sulfonates having an alkyl redical ranging in chain length from 16 to 18 carbon atoms; (3) from about 1% to about 5% of an amine oxide having the formula $R_1R_2R_3N\rightarrow O$ in which R_1 is an alkyl radical ranging in chain length from 8 to 18 carbon atoms and R_2 and R_3 are each selected from the group consisting of methyl and ethyl radicals, at least about 50% of said amine oxide having R_1 12 carbon atoms in chain length; (4) from about 0.05% to about 0.2% of a water soluble polymer selected from the group consisting of acrylamide polymers having a viscosity of at least 4 centipoises at 25° C. when dissolved at the 0.5% level in 4% sodium chloride solution adjusted to a pH of 5 to 6, and ethyleneoxide polymers having a viscosity of at least 10 centipoises at 25° C. when dissolved in water at the 1% level; (5) from about 1% to about 5% of a supplementary non-soap anionic synthetic detergent having in its molecular structure an alkyl radical of 8 to 18 carbon atoms, the said detergent having pronounced sudsing characteristics and being soluble; and (6) not more than 8% of a soluble alkali metal salt, the ingredients being in an aqueous vehicle and the mole ratio of potassium to sodium being less than about 0.8.

3. The liquid detergent composition of claim 2 wherein the supplementary detergent is selected from the group consisting of alkali metal salts of: alkyl sulfate wherein the alkyl radical contains predominantly 12 and 14 carbon atoms; alkyl benzene sulfonate wherein the alkyl radical contains 9 to 15 carbon atoms; sulfated condensation product of predominantly lauryl and myristyl alcohols condensed with about 3 moles of ethylene oxide per mole of alcohol; a sulfated condensation of one mole of nonylphenol with about four moles of ethylene oxide.

4. The liquid detergent composition of claim 2 wherein the alkali metal salt is selected from the group consisting of sodium and potassium salts of chloride, sulfate, orthophosphate, pyrophosphate, acetate and mixtures thereof.

5. An opaque pearlescent liquid detergent composition having a lotion-like consistence and consisting essentially of: (1) from about 6% to about 10% of a mixture of sodium and potassium alkyl glyceryl ether sulfonates wherein the alkyl radicals contain predominantly from 12 to 14 carbon atoms, the potassium alkyl glyceryl ether sulfonate constituting not more than about 0.7 by weight of said mixture; (2) from about 2% to about 4% sodium alkyl glyceryl ether sulfonate wherein the alkyl radicals contain predominantly from 16 to 18 carbon atoms; (3) from about 1% to about 5% of amine oxides having the formula $R_1R_2R_3N\rightarrow O$ wherein R_1 is an alkyl radical containing predominantly 12 to 14 carbon atoms and R_2 and R_3 are each selected from the group consisting of methyl and ethyl radicals; (4) from about 0.05% to about 0.2% of a water soluble acrylamide polymer having a viscosity of at least 4 centipoises at 25° C. when dissolved at the 0.5% level in 4% sodium chloride solution adjused to a pH of 5 to 6; (5) from about 1% to about 5% of a supplementary detergent selected from the group consisting of sodium salts of sulfated condensation product of one mole of nonylphenol and about 4 moles of ethylene oxide, sulfated condensation product of predominantly lauryl and myristyl alcohols with about 3 moles of ethylene oxide per mole of alcohol, alkyl sulfate wherein the alkyl radical contains predominantly 12 to 14 carbon atoms, alkyl benzene sulfonate wherein the alkyl radical contains predominantly 9 to 15 carbon atoms; (6) from about 1% to about 3% potassium pyrophosphate; (7) from about 0.1% to about 0.4% water soluble sodium carboxymethylcellulose; (8) from about 1% to about 5% of a sodium salt selected from the group consisting of acetate, chloride, sulfate and orthophosphate, and mixtures thereof; (9) from about 0.5% to about 3% of a sodium salt selected from the group consisting of benzene sulfonate, toluene sulfonate, and xylene sulfonate; and (10) water.

6. An opaque pearlescent liquid detergent composition having a lotion-like consistence and consisting essentially of (1) from about 6% to about 10% of a mixture of sodium and potassium alkyl glyceryl ether sulfonates wherein the alkyl radicals contain predominantly from 12 to 14 carbon atoms, the potassium alkyl glyceryl ether sulfonate consituting not more than about 0.7 by weight of said mixture; (2) from about 2% to about 4% sodium alkyl glyceryl ether sulfonate wherein the alkyl radicals contain predominantly from 16 to 18 carbon atoms; (3) from about 1% to about 5% of amine oxides having the formula $R_1R_2R_3N\rightarrow O$ wherein R_1 is an alkyl radical containing predominantly 12 to 14 carbon atoms and R_2 and R_3 are each selected from the group consisting of methyl and ethyl radicals; (4) from about 0.05% to about 0.2% of an ethylene oxide polymer having a viscosity of at least 10 centipoises at 25° C. when dissolved in water at the 1% level; (5) from about 1% to about 5% supplementary detergent selected from the group consisting of sodium salts of sulfated condensation product of one mole of nonylphenol and about 4 moles of ethylene oxide, sulfated condensation product of predominantly lauryl and myristyl alcohols with about 3 moles of ethylene oxide per mole of alcohol, alkyl sulfate wherein the alkyl radical contains predominantly 12 and 14 carbon atoms, alkyl benzene sulfonate wherein the alkyl radicals contain predominantly 10 to 15 carbon atoms; (6) from about 1% to about 3% potassium pyrophosphate; (7) from about 0.1% to about 0.4% water soluble sodium carboxymethylcellulose; (8) from about 1% to about 5% of a sodium salt selected from the group consisting of acetate, chloride, sulfate and orthophosphate, and mixtures thereof; (9) from about 0.5% to about 3% of a sodium salt selected from the group consisting of benzene sulfonate, toluene sulfonate, and xylene sulfonate: and (10) water.

References Cited in the file of this patent

UNITED STATES PATENTS

2,060,568	Graenacher et al.	Nov. 10, 1936
2,877,185	Krumrei et al.	Mar. 10, 1959

FOREIGN PATENTS

437,566	Great Britain	Oct. 31, 1935

OTHER REFERENCES

"The Chemistry of Fatty Amines," 1948, pub. by Armour and Co., p. 13.

"Synthetic Organic Chemicals," 1952, pub. by Carbide and Carbon Chemicals Co., pp. 52–54.

Harris: "Shampoo Formulation," in the American Perfumer and Essential Oil Review, vol. 48, No. 12, December 1946, p. 71.

UNITED STATES PATENT OFFICE

CERTIFICATE OF CORRECTION

Patent No. 2,999,068 September 5, 1961

Will Pilcher et al.

It is hereby certified that error appears in the above numbered patent requiring correction and that the said Letters Patent should read as corrected below.

Column 2, line 26, for "100° F." read -- 110° F. --; column 3, line 47, for "form" read -- from --; column 4, line 41, for "25°." read -- 25° C. --; column 7, table of Example I, column 1, line 1 thereof, for "glycerol" read -- glyceryl --; column 10, line 2, after "with" insert -- varying --; line 27, for "Eethylene" read -- Ethylene --; column 11, line 15, for "either" read -- ether --; line 18, for "redical" read -- radical --; column 12, line 25, for "consituting" read -- constituting --.

Signed and sealed this 6th day of February 1962.

(SEAL)

Attest:

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