

1

2,988,511

NONSMEARING DETERGENT BAR

Victor Mills, Wyoming, Ohio (The Procter & Gamble Company, P.O. Box 175, Cincinnati 31, Ohio), and Edwin O. Korpi, The Procter & Gamble Company, MA & R Bldg., Cincinnati 17, Ohio
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This application is a continuation-in-part of application Serial Number 498,433, filed March 31, 1955, and now abandoned.

The present invention relates to detergent compositions. More particularly, this invention relates to synthetic detergent-containing detergent compositions in bar form, characterized by a unique combination of superior qualities.

A synthetic detergent, to be usable in a bar, must normally be solid at room temperature. Consequently, the anionic sulfuric reaction products which have this characteristic have been considered to possess the greatest utility for detergent bar applications. However, most of the anionic sulfuric reaction products are also characterized by the undesirable tendency to absorb water from a soap dish, and become smeary. This condition can be remedied by combining the synthetic detergent with a soluble soap and an inert binder material such as starch, freshly precipitated calcium or magnesium soap, or a waxy material, for example, the superglycerinated hard fat described in U.S. Letters Patent 2,175,285, issued to R. A. Duncan and assigned to the present applicants' assignee. British Patent 689,569 describes the use of a solid polyethylene glycol of molecular weight 1500-4000, and high molecular ester (both mono- and di-esters) of polyhydric alcohols as a binder substance with synthetic detergents. Many other binder materials equivalent in physical behavior to any one or more of the foregoing may also be used. For example, U.S. Letters Patent 2,356,903, issued to Richard C. Wood, discloses a binder material comprising paraffin with superglycerinated hard fat. In general, any normally solid waxy material which will become plastic under the conditions encountered in the milling of soap may be used.

The use of soluble soap, which has been found to be a very desirable ingredient in a bar containing an anionic synthetic detergent, presents a further problem. In hard water, whenever the foaming and dispensing power of the soap has been destroyed by dilution of the soap, the soluble soap will interact with the calcium and magnesium salts present in the water to form an insoluble soap curd. Accordingly, the synthetic detergent should desirably have good curd dispersing characteristics. In addition, any bar product must lather well in hard as well as soft water, it should not crack unduly when, as in normal use, it is alternately wetted and allowed to dry out, and it should be mild to the skin.

A primary object of the present invention is to provide a synthetic detergent composition in a toilet bar form which will be mild to the skin.

Another object is to provide a synthetic detergent composition which will have good curd dispersing characteristics.

It is also an object to provide a detergent bar which will lather well in hard as well as soft water.

A further object is to provide a detergent bar which will not crack unduly when, as in normal use, it is alternately wetted and allowed to dry out.

The foregoing and other objects can be accomplished by providing a detergent bar containing, as the major anionic organic sulfuric reaction product constituent, alkyl

2

glyceryl ether sulfonate, as more fully defined herein-after, in combination with soluble soap and a binder material.

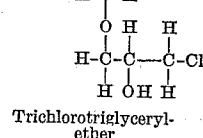
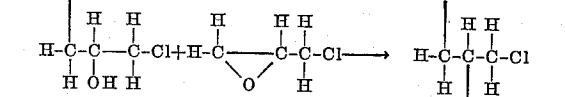
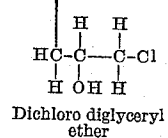
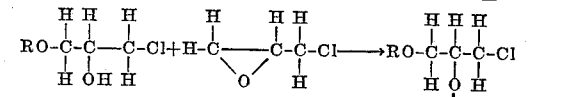
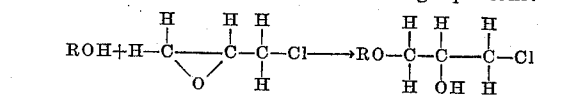
The sodium and potassium alkyl glyceryl ether sulfonates are outstanding in their ability to disperse soap curd and in their mildness toward the skin of the user. The sodium and potassium alkyl glyceryl ether sulfonates are somewhat crystalline materials at room temperature, hence they do not swell and slime unduly when in contact with water. They do, however, dissolve readily in water and form a copious lather. This combination of properties permits the formulation of a toilet bar having all of the desirable characteristics set forth above.

Alkyl glyceryl ether sulfonates are disclosed in U.S. Letters Patent 2,094,489, granted to Richard Hueter, September 28, 1937. Hueter describes the production of these compounds by reacting epichlorohydrin with an excess of fatty alcohols in the presence of an acid catalyst. The catalyst is removed by water washing and the product is purified by fractional distillation. The resulting alkyl chloroglyceryl ether is then reacted with sodium sulfite (the Streckerization reaction) to form the sodium salt of alkyl glyceryl ether sulfonic acid and sodium chloride.

The alkyl glyceryl ether sulfonate constituent of the detergent compositions of this invention are prepared in a manner different from that disclosed in the Hueter patent.

It has been found that these alkyl glyceryl ether sulfonates, which are characterized by good detergent properties, may be prepared by reacting high molecular weight fatty alcohols with an amount of epichlorohydrin which is in excess of that required to react with the alcohol to produce the chloromonoglyceryl ether and then sulfonating the resulting chloroglyceryl ethers by means of the Streckerization reaction.

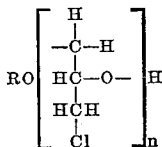
The use of an excess of epichlorohydrin results in the production of chloroglyceryl ethers in which the glyceryl radical is replaced in part with polyglyceryl radicals e.g. with 2 or 3 condensed glyceryl radicals. The formulation of such polyglyceryl ethers may be said to progress step-wise in accordance with the following equations:



The higher polymers, such as the tetrachloro tetraglyceryl ethers may also be formed, the amount depending upon the excess of epichlorohydrin which is used.

In any event, the reaction product will comprise a mixture of monomer and various proportions of the polymers.

The mixture of chloroglyceryl ethers which is formed in the process can be represented by the following general formula,



wherein R is an alkyl radical containing from about 10 to about 20 carbon atoms and n is an integer from 1 to 4, said mixture comprising at least 10% of such ethers wherein n is 2. For convenience, the chloroglyceryl ethers wherein n is 1, 2, 3 or 4 will hereinafter be referred to respectively as the monomer, dimer, trimer and tetramer.

In preparing the alkyl glyceryl ether sulfonate of the invention, the foregoing procedure, utilizing an excess of epichlorohydrin, was employed. For example, a fatty alcohol having from about 10 to about 20 carbon atoms was reacted with at least a 5% molar excess of epichlorohydrin in the presence of stannic chloride as a catalyst. The reaction product was washed free of catalyst and was then reacted with sodium or potassium sulfite (Streckerization).

When the alkyl glyceryl ether sulfonates of the invention are prepared using the "middle" cut coconut alcohol, to be discussed hereinafter, it is preferred that the amount of excess epichlorohydrin be adjusted so as to produce a chloroglyceryl ether product, which, upon sulfonation, will be characterized by a dimer content in the range from about 10% to about 30%. Adjustment of the excess epichlorohydrin to produce a product having a dimer content within the aforementioned limits will insure the presence, in the chloroglyceryl ether intermediate, of a minimum of unreacted alcohol and of trimer and tetramer fractions.

The presence of large amounts of trimer and tetramer sulfonates in the alkyl glyceryl ether sulfonates prepared from middle cut coconut alcohols is to be avoided because of the reduced deterative activity of such trimer- and tetramer-containing compounds in detergent compositions.

It has been found that the amount of dimer specified above, after subsequent sulfonation, is sufficient to impart to the products of this invention the advantages associated with the presence of the dimer. In addition, it has been found that the amount of unreacted alcohol after subsequent sulfonation does not significantly modify the advantageous properties of the alkyl glyceryl ether sulfonates.

During the Streckerization reaction referred to above, sodium or potassium chloride is formed depending upon whether sodium or potassium sulfite is used as the Streckerization agent. Normally, the sodium or potassium chloride is left in the product. Any excess sodium or potassium sulfite which may be present in the product as a result of the Streckerization reaction is partially converted to the sulfate by oxidation. All of these salts will be referred to hereinafter generically as "secondary Streckerization products."

If desired, salts other than the sodium or potassium salts of the alkyl glyceryl ether sulfonic acid can be present in the composition, but the amount present should not have adverse effect on the desired normally solid character of the ultimate composition. Such other salts include the calcium, magnesium, ammonium or alkylol substituted ammonium salts which can be prepared from the sodium salts for example, by passing a solution of same over an ion exchange resin to replace the sodium ion with a hydrogen ion, the resulting acid being neutralized

with calcium or magnesium hydroxide, ammonia or alkylol-substituted ammonia (alkylol amines).

For the purposes of this invention, saturated fatty alcohols having from about 10 to 20 carbon atoms in the alkyl chain, or mixtures thereof, derived from naturally occurring fats and oils, which may include minor amounts of unsaturated alcohols, as well as the oxo alcohols, can be readily used in the preparation of the alkyl glyceryl chloro ether products. The "middle cut" of fatty alcohol mixtures derived from oils of the coconut oil group, i.e., the fraction consisting almost entirely of C_{12} and C_{14} fatty alcohols, has been found particularly suitable. For example, "middle cut" coconut alcohol is definitive of the alcohol derived from fractionally distilling the alcohols made by the reduction of coconut oil, the separated middle fraction having the following approximate composition:

	Percent
C_{10}^1 -----	2
C_{12} -----	66
C_{14} -----	23
C_{16} -----	9

¹The subscript denotes the number of carbon atoms in the alkyl chain.

The advantages of alkyl glycerol ether sulfonates in the detergent bar of this invention begins to be apparent at about 25% of the total of the bar's anionic synthetic detergent content. However, it is preferred that the alkyl glyceryl ether content be from about 50% to 100% by weight of the organic anionic sulfuric reaction product and that at least 50% of the alkyl radicals contain about 12 carbon atoms.

Many anionic organic sulfuric reaction products may be advantageously used in conjunction with the alkyl glyceryl ether sulfonates. However, these ancillary anionic detergent agents should be selected with some discretion since they must be adequately water soluble, normally solid at room temperature and should not hydrolyze unduly under conditions wherein they are alternately wetted and dried. The anionic organic sulfuric reaction products which are preferred are the sodium and potassium salts. Ammonium and substituted ammonium salts tend to produce pasty, less-solid materials and should therefore be used only in minor proportions. In addition, it is advisable that the alkyl groups of the active detergent ingredients be substantially saturated since it has been found that unsaturation in these groups will tend to promote a softer product.

Typical of the large group of water soluble anionic sulfuric organic reaction products which can satisfactorily be used along with alkyl glyceryl ether sulfonate are:

The alkyl sulfates, typified by sodium dodecyl sulfate. The monoglyceride sulfates, such as coconut oil monoglyceride sodium sulfate. (These are preferably not used with alkaline builders.)

The alkyl ether glycol sulfates, such as coconut alcohol ether ethylene glycol sodium sulfate.

The alkyl ether monoglyceryl sulfate such as coconut alcohol monoglycerol sodium sulfate.

The isethionates—for example, $\text{RCOOC}_2\text{H}_4\text{SO}_3\text{Na}$, where the RCO radical is derived from an oil of the coconut oil group.

By "coconut oil group" as used herein is meant the group of tropical nut oils characterized by a predominant content of combined fatty acids having 10-14 carbon atoms.

The taurines, for example, $\text{RONHC}_2\text{H}_4\text{SO}_3\text{Na}$, where the RCO radical is derived from an oil of the coconut oil group.

The sulfo acetates $\text{ROOCCH}_2\text{SO}_3\text{Na}$, where R is an alkyl radical of an alcohol derived from an oil of the coconut oil group.

Detergents made by sulfating and neutralizing the compounds made by reacting a fatty amine with hydroxy acetic acid.

The alkyl benzene sulfonates where the alkyl radical has a chain length of about 9 to about 15 carbon atoms can also be employed provided their tendency to exhibit a sticky feel is not objectionable. Mixtures of such sulfonates with alkyl sulfates wherein at least 50% of the alkyl radicals have a chain length of 12 carbon atoms can also be employed.

The water soluble soaps preferably used in the present invention are the sodium salts of fatty acids having a chain length of from about 10 to about 18 carbon atoms.

In order to determine the performance characteristics of various detergent bar formulations, the following tests, which have been standardized for such evaluations, were used. Grading was estimated on an arbitrary scale of 1 to 10, in which 1 is the poorest grade and 10 is the best grade. It should be noted that this scale has an opposite connotations for the overnight smear test only, wherein 1 is considered the best grade and 10 is considered the poorest grade.

In the tests described below, reference to a water of a certain "grain" hardness, means the grains per U.S. gallon expressed as calcium carbonate equivalent.

(1) *Bathub ring and curd test.*—1 gram of the detergent composition being tested is dispersed in 100 ml. of 21 grain water at 100° F. in a Waring type blender. This dispersion is then transferred to a 5 liter glass funnel-like device and 1500 ml. of 21 grain water at 100° F. are run into the funnel under standard conditions of rate and location of inlet. After 1 minute, the funnel-like device is drained to a volume of 100 ml. in 50 seconds. The amount of curd remaining on the walls of the funnel and in the last 100 ml. of the "bath" is estimated and given a grade according to the beforementioned arbitrary 1-10 scale.

(2) *Hand lather technique.*—1 to 3 drops of standard soil (1 part lanolin-2 parts light mineral oil) is applied to the hands. The hands are lathered under running tap water (80-100° F. controlled within $\pm 1^\circ$ F.) with the test bar. The speed, amount, and character of lather are visually estimated.

(3) *Overnight smear test.*—A $\frac{1}{4}$ " slice is cut from the face of the bar to be tested by wire cutting or planing. The bar is then weighed. The cut face is placed on two $\frac{1}{16}$ " rods in a dish to which water at 70° F. has been added. The dish and bar are kept at 70° F. for 16 hours. The bar is then removed and all softened material is wiped from the bar by holding the forefinger as tightly as possible against the softened surface of the bar and moving the finger across the face of the bar with a swiping motion. The bar is then reweighed and the difference in weight in grams is reported as grams of smear. It should be noted that contrary to the rating of the other characteristics of the bar, where 10 is "perfect" and 1 is very poor, the smear values increase as the bar becomes poorer. Hence, the lower the smear value the more desirable the smear characteristics of the bar being tested.

(4) *Wet cracks test.*—A $\frac{1}{4}$ " slice is removed from the bar. The cut surface is then washed until smooth. The washed bar is placed cut side up in water at 70° F. such that the bar surface is submerged in the water. The bar is allowed to soak for 30 minutes and is then removed from the water and dried at 70° F. for 16 hours, with a baffle 1" above the cut surface and parallel to it. The surface is coated with a carbon black slurry which is then washed off leaving the black in any cracks. The bar is graded by comparison with a graded series of ten photos of the hereinafter defined standard bar.

The relationship of the synthetic detergent, of binder, and of soluble soap in the compositions of the following examples illustrates generally the useful and practical limits in the practice of the instant invention. The preferred products of the present invention are those having the following composition limits and which have been

processed into a toilet bar form by the operations normal to the production of milled soap in the industry.

15 to 55% anionic sulfuric reaction product of which at least 50% is alkyl glyceryl ether sulfonate
20 to 70% binder
5 to 50% soluble soap

These figures are based on an assumed total of 100%, and the actual total percentage of synthetic detergent, of binder, and of soluble soap in the bar will normally be not less than about 75% by weight of the bar. The remainder of the bar will comprise moisture, perfume, secondary Streckerization products with or without other ingredients normally identified with a detergent toilet bar.

In the examples, in which all parts are by weight, the following definitions apply.

"*Neat soap.*"—The final phase obtained in the kettle boiling method for preparing soap containing approximately 30% water.

Amalgamator.—A dry-material type mixer which distributes coloring matter, perfume and other materials, which it may be desired to add, uniformly throughout partially dried soap in the flake or chip form.

Crutcher.—A vessel equipped with a strong agitator used for mixing neat soap with other materials.

In order to judge the performance characteristics of the bars of the examples, the various numerical grading values set forth can be compared with those values appearing below which represent the values obtained with a "standard" soap bar marketed by The Procter & Gamble Company under the trademark "Camay."

Test	Grade
Bathub Ring and Curd Dispersion.....	4.
Hand Lather.....	6.
Wet Cracks.....	9.
Overnight Smear.....	7-8 grams.

Example 1

Parts shown are by weight.

A paste of 12 parts water and 8 parts of sodium alkyl glyceryl ether sulfonate containing about 20% of diglyceryl ether sulfonates, the alkyl radical being derived from middle cut coconut alcohols, are combined in a crutcher with a paste of 30 parts water and 20 parts of potassium alkyl sulfate derived from middle cut coconut alcohols. To the resultant mixture is added 25½ parts of "neat" soda soap obtained from a mixture of 80% tallow and 20% coconut oil (i.e. about 18 parts real soap and 7½ parts water). After these materials are well mixed, there is added slowly with agitation 7.61 parts of Epsom salts ($MgSO_4 \cdot 7H_2O$), whereby the sodium soap is converted to the magnesium soap which precipitates in hydrated form. The hydrated magnesium soap possesses superior characteristics as a binder material. The resultant mix is roll dried and the dried product is mixed in an amalgamator with 41 parts of sodium soap derived from a mixture of 80% tallow and 20% coconut oil. This latter soap is added in the form of pellets containing about 15% moisture. 2.7 parts of sodium chloride is also added to the mix to repress the solution of the alkyl sulfate. The amalgamated mix is then milled, extruded and stamped in conventional soap making equipment.

This bar was an improvement over a soap-synthetic bar in which all of the active anionic synthetic was alkyl sulfate, and wherein at least 50% of the alkyl groups contained 12 carbon atoms. With alkyl sulfate as the sole active synthetic detergent ingredient, it is necessary to have about 40% or more of alkyl sulfate in the bar in order for the bar to have good curd dispersion properties. These high alkyl sulfate bars were drying to the skin, i.e. they exhibited the tendency to remove the natural skin oils, especially when repeatedly used at relatively

7

short time intervals such as is encountered in wash stand use. By using 8% of alkyl glyceryl ether sulfonate in such a toilet bar, the alkyl sulfate content of the bar can be cut to 20% without loss of curd dispersion. Such a 20% alkyl sulfate-8% alkyl glyceryl ether sulfonate bar is mild to the skin of the user, and is distinctly superior in this respect to bars containing alkyl sulfate or alkyl benzene sulfonate (averaging about 12 carbon atoms in the alkyl group) as sole active anionic synthetic detergent ingredient.

In the preferred products of this invention, which are outstandingly mild and are comparable in this respect to toilet soaps, at least 50% of the anionic synthetic detergent should be the alkyl glyceryl ether sulfonate, and the alkyl sulfate and alkyl benzene sulfonate should constitute less than 50% of the anionic synthetic detergent components. A consideration of all performance factors, indicates that bars in which alkyl glyceryl ether sulfonates predominate over the other anionic synthetic detergent components are outstandingly superior.

In the following examples, the materials, such as "alkyl glyceryl sulfonate," and "soda soap," are to be understood to connote the pure ingredient unless otherwise noted, that is, the water and inorganic salts will be listed as such.

Example 2

The following constituents are combined as indicated to produce a superior toilet bar.

Sodium alkyl glyceryl ether sulfonate derived from middle cut coconut alcohols and containing about 10% of diglyceryl ether sulfonates.....	36
Soda soap, 50-50 tallow-coconut soaps.....	32
Superglycerinated hardened cottonseed oil ¹	18
Secondary Streckerization products.....	14

¹This constituent had an iodine value of 23 and contained about 38% monoglycerides, 43% diglycerides, and 19% triglycerides.

The above components are placed in an amalgamator, water is added in an amount sufficient upon subsequent mixing, milling, plodding and stamping to give a final product with 9% moisture.

Example 3

A toilet bar having the following formulation is prepared as indicated.

Sodium alkyl glyceryl ether sulfonate derived from middle cut coconut alcohols and containing about 20% of diglyceryl ether sulfonates.....	20
Sodium soap of coconut oil.....	35
Sodium soap of 80% tallow, 20% coconut oil.....	20
Secondary Streckerization products and sodium chloride from calcium soap preparation.....	10
Moisture	15

The alkyl glyceryl ether sulfonate is brought into the crutcher in the form of a paste containing about 50% water. The 80% tallow, 20% coconut soap is then added as kettle soap. Calcium chloride in an amount equivalent to that required to convert the 80% tallow, 20% coconut soap wholly into calcium soap is added. During the subsequent crutching operation calcium soap and sodium chloride are formed. The resultant crutcher mix is then roll dried. The dried mix is then put in an amalgamator and the coconut soap, as kettle soap, is added and well mixed with the dried crutcher mix. The amalgamated mix is milled, extruded, and stamped in conventional equipment. The resulting detergent bar contained about 15% moisture and exhibited the following performance characteristics.

Hand lather	9
Bathtub ring and curd dispersion.....	10
Wet cracks	9.6

8

Example 4

A superior toilet bar having the following formulation was prepared as indicated. Parts are by weight.

5 Sodium alkyl glyceryl ether sulfonate derived from middle cut coconut alcohols and containing about 20% of diglyceryl ether sulfonate.....	10
Sodium alkyl sulfate from middle cut coconut alcohols	10
10 Sodium soap of coconut oil.....	35
Magnesium soap of 80% tallow, 20% coconut oil....	20
Secondary Streckerization products and sodium sulfate from magnesium soap preparation.....	10
15 Moisture	15

This was prepared by the method used in Example 3, substituting magnesium sulfate (Epsom salts) for the calcium chloride. The resultant bar had the following performance characteristics:

20 Hand lather	9
Bathtub ring and curd dispersion.....	10
Overnight smear	2.4
Wet cracks	9.5

25 One part of the alkyl sulfate in this example can be replaced with polypropylene benzene sodium sulfonate, in which the polypropylene radical averages 12 carbon atoms, to give a comparable bar.

Example 5

30 A toilet bar of the following formulation was prepared as indicated.

35 Sodium alkyl glyceryl ether sulfonate derived from middle cut coconut alcohols and containing about 20% of diglyceryl ether sulfonates.....	10
Sodium alkyl sulfate from middle cut coconut alcohols	10
Sodium coconut soap.....	35
40 Calcium soap of 80% tallow, 20% coconut oil....	25
Secondary Streckerization products and sodium chloride from calcium soap preparation.....	10
Moisture	10

Made as in Example 3, that is, the 80% tallow-20% coconut sodium soap was converted to calcium soap by using calcium chloride. This bar tested as follows:

Hand lather.....	9
Bathtub ring and curd dispersion.....	9.3
Overnight smear.....grams..	4.5

50 Instead of the freshly precipitated calcium or magnesium soap as the binder material, starch can be used to replace all or part of the calcium or magnesium soaps with comparable results.

Example 6

A toilet bar of the following formulation is prepared as indicated.

60 Sodium alkyl glyceryl ether sulfonate derived from middle cut coconut alcohols and containing about 20% of diglyceryl ether sulfonates.....	20
Sodium coconut soap.....	15
Sodium soap of 80% tallow, 20% coconut oil.....	10
Oxidized starch.....	35
65 Secondary Streckerization products.....	10
Water	10

Enough hot water is added to a crutcher initially so that the ultimate crutcher mix will contain from 60 to 75% water. The alkyl glyceryl ether sulfonate is added to the crutcher with rapid agitation until there is thorough dispersion of the material. The starch is then added and also dispersed with rapid agitation. The crutcher is then operated at normal speed to achieve a good mix. 70 This mix is then dried on a roll heated with 60 lb. steam.

9

The roll dried mix is added to an amalgamator, the sodium 80% tallow, 20% coconut oil soap is added as pellets and the sodium coconut soap is added as kettle soap—about 30% water. The amalgamator charge is thoroughly mixed and is then milled, extruded and stamped in conventional milled soap equipment. The bar tested as follows:

Hand lather.....	8
Overnight smear.....grams..	9.3
Wet cracks.....	10

Example 7

A toilet bar having the following formulation is prepared as indicated.

	Parts
Sodium alkyl glyceryl ether sulfonate derived from middle cut coconut alcohols and containing about 20% of diglyceryl ether sulfonates.....	20
Sodium soap of 80% tallow, 20% coconut oil.....	10
Sodium coconut oil soap.....	15
Calcium soap of 80% tallow, 20% coconut oil.....	10
Oxidized starch.....	25
Water.....	10
Secondary Streckerization products and sodium chloride from calcium soap preparation.....	10

The sodium alkyl glyceryl ether sulfonate is dissolved in enough water to constitute 60 to 75% of the final crutcher mixture. The 80% tallow, 20% coconut soap to be made into calcium soap is then dissolved in the soap crutcher. Calcium chloride in an amount equivalent to that required to convert this soap to the calcium soap is added and agitation is continued until dispersion takes place. The starch is then added and dispersed with agitation, which is continued until all the ingredients are well mixed. The crutcher mix is roll dried. The dried crutcher mix is then amalgamated with pellets of sodium soap of 80% tallow, 20% coconut oil and with sodium coconut oil soap as kettle soap. The combined components are milled, plodded and stamped. The hand lather grade of the resulting detergent bar was 8—.

Example 8

A toilet bar having the following formulation is prepared as indicated.

	Parts
Sodium alkyl glyceryl ether sulfonate derived from middle cut coconut alcohols and containing about 20% of diglyceryl ether sulfonates.....	20
Sodium soap of coconut oil.....	35
Oxidized starch.....	25
Secondary Streckerization products.....	10
Water.....	10

Made as in the case of Example 6, this bar tested as follows:

Hand lather.....	9
Bathtub ring and curd dispersion.....	9

Example 9

A toilet bar having the following formulation is prepared as indicated.

	Parts
Potassium alkyl glyceryl ether sulfonates derived from middle cut coconut alcohols and containing about 20% of diglyceryl ether sulfonates.....	40
20% sodium soap of 80% tallow, 20% coconut oil; 80% magnesium soap of 80% tallow, 20% coconut oil.....	42
Moisture.....	8
Secondary Streckerization products and sodium chloride from calcium soap preparation.....	10

The potassium alkyl glyceryl ether sulfonate is transferred to the crutcher as a paste, containing about 8% water. The 80% tallow, 20% coconut oil is added as

10

kettle soap containing 35 lbs. of real sodium soap of 80% tallow, 20% coconut oil, and 17.5 lbs. of Epsom salts dissolved in water is added. After thorough agitation to secure complete reaction, the mix is roll dried, milled, extruded and stamped. This bar tested as follows:

Hand lather.....	8
Bathtub ring and curd dispersion.....	9
Overnight smear.....grams..	10

Example 10

	Parts
Sodium alkyl glyceryl ether sulfonates derived from middle cut coconut alcohols and containing about 20% of diglyceryl ether sulfonates.....	25.7
Calcium hardened tallow soap.....	45.3
Sodium coconut soap.....	5.0
Coconut fatty acid monoethanol amide.....	2.0
Water and secondary Streckerization products and sodium chloride from calcium soap preparation.....	22.0

This bar tested as follows:

Hand lather grade.....	7—
Bathtub ring and curd dispersion.....	10
Overnight smear.....grams..	8+
Wet cracks.....	10

In this example it will be noted that all of the insoluble soap is made from tallow and all of the soluble soap is made from coconut oil. This is a desirable procedure in a composition with a high level of insoluble soap. The soluble soap being of the quick lathering coconut type, tends to overcome the lather repressing effect of the large amount of insoluble soap. Moreover, there is a price advantage in using the less expensive tallow for the insoluble soap. The tallow fatty acids are substantially all of 16 and 18 carbons in chain length and have a titer between 38 and 45.

In the above formulation three parts of the indicated sodium alkyl glyceryl ether sulfonates are replaced with sodium alkyl glyceryl ether sulfonates prepared from substantially completely hydrogenated tallow alcohols and containing about 30% dimer. A synthetic-soap toilet bar with outstanding characteristics is obtained.

Having thus described our invention, what we claim is:

1. A milled detergent bar at least 75% by weight of which consists essentially of (1) from about 15% to about 55% of normally solid detergent salts of anionic organic sulfuric reaction products which do not hydrolyze unduly under conditions of alternate wetting and drying, said salts being selected from the group consisting of the sodium and potassium salts, and said anionic organic sulfuric reaction products containing at least 50% alkyl glyceryl ether sulfonates from about 10% to about 30% of which alkyl glyceryl ether sulfonates are alkyl diglyceryl ether sulfonates, the alkyl radicals containing from about 10 to about 20 carbon atoms; (2) from about 5% to about 50% of a water-soluble soap of fatty acids having from about 10 to about 18 carbon atoms; and (3) from about 20% to about 70% of a binder material selected from the group consisting of freshly precipitated calcium soaps of fatty acids having from about 10 to about 18 carbon atoms, freshly precipitated magnesium soap of fatty acids having from about 10 to about 18 carbon atoms, starch, normally solid waxy materials which will become plastic under the conditions encountered in the milling of soap and mixtures thereof.

2. The detergent bar of claim 1 wherein the anionic organic sulfuric reaction product is substantially completely alkyl glyceryl ether sulfonate the alkyl radical of which is derived from middle cut coconut alcohols.

3. The detergent bar of claim 1 wherein the anionic organic sulfuric reaction product is a mixture of alkyl glyceryl ether sulfonate, the alkyl radical of which is derived from middle cut coconut alcohols and alkyl sulfate, the said alkyl glyceryl ether sulfonate and alkyl sulfate

11

each containing about 50% of alkyl radicals having 12 carbon atoms.

4. The detergent bar of claim 1 wherein the binder is starch.

5. The detergent bar of claim 1 wherein the binder is magnesium soap.

6. The detergent bar of claim 1 wherein the binder is calcium soap.

7. The detergent bar of claim 1 wherein the soluble soap is derived from the fatty acids of coconut oil and the binder is an insoluble soap averaging from about 16 to 18 carbon atoms in the fatty acid chain and has a titer ranging from 38 to 45.

8. The detergent bar of claim 1 wherein the anionic organic sulfuric reaction product comprises in combination alkyl glyceryl ether sulfonates and an organic sul-

12

furic reaction product selected from the group consisting of the water-soluble salts of alkyl sulfuric acid, wherein at least 50% of the alkyl radicals contain 12 carbon atoms, water-soluble salts of alkyl benzene sulfonic acid wherein the alkyl radical contains from 9 to 15 carbon atoms, and mixtures thereof.

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443,559	Great Britain	Feb. 17, 1936
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UNITED STATES PATENT OFFICE
CERTIFICATE OF CORRECTION

Patent No. 2,988,511

June 13, 1961

Victor Mills 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 1, line 48, for "dispensing" read -- dispersing --; column 4, line 25, for "glycerol" read -- glyceryl --; line 67, for "RONHC₂H₄SO₃Na" read -- RCONHC₂H₄SO₃Na --; column 5, line 17, for "connotations" read -- connotation --; column 10, line 64, for "plasic" read -- plastic --.

Signed and sealed this 14th day of November 1961.

(SEAL)

Attest:

ERNEST W. SWIDER

Attesting Officer

DAVID L. LADD

Commissioner of Patents

USCOMM-DC