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Gatti et al.

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[54] **DETERGENT BAR MADE FROM MIXED FATTY ACID DERIVATIVES**

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[51] **Int. Cl.** **C11d 13/00**

[58] **Field of Search** ..... 252/121, 132, 182, 557, 252/117, 142, DIG. 16; 260/398, 400, 413

[56] **References Cited**

UNITED STATES PATENTS

3,730,912 5/1973 Inamorato..... 252/121 X

OTHER PUBLICATIONS

McBride, Chemical & Metallurgical Engineering, Dec. 1940, pp. 830 to 833 (TN 1 M5).

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[57] **ABSTRACT**

Mixtures of acyl isethionates of selected critical chain lengths exhibit superior lathering properties when incorporated into a detergent toilet bar.

4 Claims, No Drawings

DETERGENT BAR MADE FROM MIXED FATTY ACID DERIVATIVES

BACKGROUND OF THE INVENTION

1. Field of the Invention

Detergent toilet bars containing acyl isethionates have been known for many years. For example Nuesslein in U.S. Pat. No. 1,906,484 discloses an alkali-metal soap, and specifically sodium palmitate, containing admixed therewith from 5 to 1000%, soap basis, of an acyl isethionate of vegetable origin.

A commercially successful bar is described and claimed in U.S. Pat. No. 2,894,912 to Geitz. However this bar is relatively expensive to manufacture, and it is evident that the discovery of a procedure for making a less expensive bar without sacrificing quality would be highly desirable.

2. The Prior Art

U.S. Pat. No. 1,906,484 to Nuesslein

The most pertinent disclosure in this patent is Example 3, wherein there is set forth a mixture of 80 parts oleic isethionate, 15 parts of sodium palmitate, and 5 parts of sodium perborate.

U.S. Pat. No. 2,749,315 to Faier

Discloses a process for making a bar having a major amount of anhydrous, water-soluble soap, and a minor amount of mixed water-soluble synthetic detergents comprising a mixture of higher fatty acid monoglyceride monosulfates and higher fatty acid amide of aminomethanesulfonates.

U.S. Pat. No. 2,781,321 to Mayhew et al diisopropyl

Relates to a bar containing a detergent, e.g., Na oleic isethionate, a plasticizer, e.g., calcium stearate, and a lathering agent, e.g. Na diisorpropyl naphthalenesulfonate.

U.S. Pat. No. 2,894,912 to Geitz

Discloses a bar having 30 to 70% of an acyl isethionate, at least 75% of the acyl groups having 12–18 carbon atoms and up to 25% having 6–10 carbon atoms, up to 25% soap, 10% to 40% fatty acid, up to 9% water, 2 –10% of a suds-boosting detergent, the bar having a pH of 6 to 8 measured as a 10% aqueous solution at 35°C.

U.S Pat. No. 3,420,857 to Holland et al

Relates to a step in the process for reacting a fatty acid with sodium isethionate comprising continuously replacing the fatty acids volatilized during the course of the reaction with fatty acids having a composition corresponding to that of the volatilized fatty acids.

U.S. Pat. No. 3,420,858 to McCrimlisk

Relates to a two-step vacuum stripping operation in a process for reacting a fatty acid with sodium isethionate.

U.S. Pat. No. 3,607,761 to Feighner et al

Relates to soaps containing soaps prepared from C_{12}–C_{16} acids derived from the oxidation of alcohols produced by the Guerbet reaction.

SUMMARY OF THE INVENTION

It has now been discovered that the lathering properties of a toilet detergent bar, wherein the major surface-active detergent component is an acyl isethionate, are improved, or the same lathering as heretofore can be obtained with less active detergent, when the acyl portion of the acyl isethionate is a mixture derived from fatty acids of critical chain length.

Accordingly it is an object of the present invention to provide a toilet detergent bar having improved lathering properties.

It is another object of the invention to provide a fatty acid mixture adapted to be esterified with an alkali-metal isethionate to produce a mixture of alkali-metal acyl isethionates having the chain-length distribution of said mixture, and having superior lathering properties when formulated into a toilet bar for cleansing the skin.

Therefore the invention concerns a toilet detergent bar having as its major detergent active a mixture of acyl isethionates wherein the acyl portion is derived from fatty acids of mixed selected critical chain lengths and has substantially the chain-length distribution of said fatty acids.

In a broader aspect the invention relates to a fatty acid mixture having critical proportions of selected chain lengths and being capable of esterification with isethionic acid to form a mixture of acyl isethionates having superior lathering properties.

In another aspect the invention relates to a mixture of acyl isethionates having superior lathering properties when formulated into a toilet detergent bar.

By the term "superior lathering properties" is meant the ability to produce at a lower acyl isethionate content the same lather volume as prior-art acyl isethionate bars, as well as more lather at the same active detergent content.

The fatty acyl portion of the fatty acyl isethionate must be selected critically to obtain the improved sudsing characteristics of the products of the present invention. The invention contemplates the use of acyl isethionates wherein the fatty acyl portion comprises about 28–37% C_{10}, about 18–24% C_{12}, about 16–21% C_{14}, about 9–20% C_{16}, and about 10–17% C_{18} chain lengths.

The preferred ranges of acyl chain lengths are about 32–34% C_{10}(decanoyl), about 20–23% C_{12}(dodecanoyl), about 18–20% C_{14}(tetradecanoyl), about 12–16% C_{16}(hexadecanoyl), and about 11–14% C_{18} (octadecanoyl), chain lengths. The foregoing proportions are based on the total fatty acids combined as isethionate.

The fatty acids contemplated for use in the practice of this invention are unbranched and saturated, and may be derived from natural fats, such as coconut oil, palm kernel oil, tallow, hydrogenated vegetable oils, etc., or may be prepared synthetically.

MANUFACTURE OF THE ACYL ISETHIONATE

The acyl isethionate found useful in accordance with the present invention is prepared by the general procedure described in Holland et al, U.S. Pat. No. 3,420,857, and McCrimlisk U.S. Pat. No. 3,420,858, both assigned to the instant assignee. In these patents there are described procedures whereby, in the esterification of sodium isethionate with fatty acids having a range of chain lengths, there is avoided any substantial enrichment of the esters in the higher molecular weight acyl portion of the molecule due to volatilization of the lower molecular weight fatty acids. This is accomplished by continuously supplying to the reaction mass fatty acid reactants of a composition corresponding to fatty acids volatizlied during the course of the reaction. Moreover when excess fatty acid reactant is stripped from the reaction mass, stearic acid, which is a component of the bar to be formulated from the acyl isethionate, is added to the mass to retain fluidity. If the stearic acid is added at the start of the stripping operation a

portion of this acid reacts with free sodium isethionate since at this point the esterification reaction is still proceeding. This results in lowering the proportion of the desirable low molecular weight acyl isethionates. To minimize this, the early stage of the step is conducted under vacuum to remove as much of the lights as possible and when lack of fluidity due to depletion of the free fatty content becomes a problem, stearic acid is added to restore fluidity. By delaying the addition of stearic acid, less of the undesirable high molecular weight acyl isethionates are formed than would be formed if the stearic acid were added at the start of the stripping step.

The use of the critical fatty acid mixture for preparing the more efficient acyl isethionate in accordance with the invention has the advantage of economy of manufacture due primarily to the fact that the reaction between sodium isethionate and fatty acids need proceed only to the point of attaining 67% active detergent acyl isethionate in the reaction mass rather than 75–85% as in the Holland et al process described in U.S. Pat. No. 3,420,857, this process being representative of the prior-art commercial process. Moreover the absence of the lower molecular weight fatty acids in the process of the instant invention lessens the stripping time. It is estimated that the reaction time for the preparation of acyl isethionate from the critical fatty acid mixture of the instant invention is only about one-half that by prior-art procedures.

THE LATHERING TEST

Twenty women constituting a test panel are provided with detergent or soap bars to be tested and requested to wash their hands in their own customary manner and to make a judgment of the comparative lathering properties of the bar. Only two bars are tested at one time.

THE WEAR-RATE TEST

This test determines the weight loss of a detergent or soap bar which has been washed in a standardized manner and allowed to remain in an undrained soap dish between uses.

To determine wear rate, initial and final weights of a bar are made in a partially wet state, the test being conducted in the following manner. A bar is submerged in the hands in ½ gallon of water at 105°F having a desired hardness in a 1-gallon container. The bar is removed and rotated in the hands 20 times through 180°. The bar is again submerged in the same water, removed, and rotated 20 times as before. The bar is submerged a third time in the water, removed, and carefully contacted with terry towelling to remove adhering water. The bar is then weighed, and placed in a conventional flat bottom soap dish containing water at 80°F; 6.5 ml of water for a regular size bar, and 7.5 ml for a bath-size bar. The foregoing is carried out as early in the day as is practical. At equally spaced-apart intervals throughout the day, the abovedescribed dual handwashing steps are repeated three times, the bar being placed in the water-containing dish after the second wash of each of the dual washing steps. On the second day, as early as practical, the bar is removed from the dish and a dual wash step made as before, following which the bar is again contacted with terry towelling to remove adhering water, and again weighed. The difference in weights divided by the number of washes conducted is the weight loss per wash.

The invention may be more readily understood by reference to the following Examples which are illustrative, but are not to be considered limitative, of the invention.

EXAMPLE 1

This example shows the effect of varying the chain-length distribution of the acyl isethionates on lathering and wear rate of toilet detergent bars prepared therefrom.

Sodium acyl isethionates are prepared from a mixture of 50% C_{10}, 25% C_{12} and 25% C_{14} saturated fatty acids. A separate batch is prepared from commercial triple pressed stearic acid, consisting of about 55% hexadecanoic and about 45% octadecanoic acids. A control batch is prepared from partially hydrogenated coconut oil fatty acids. Each batch is fluidized with the aforementioned triple pressed stearic acid, the batches being prepared in general by the process of Holland et al described in U.S. Pat. No. 3,420,857, to form a mixture of sodium acyl isethionates and free fatty acids.

The compositions of the bars are shown in Table I and the fatty acid distribution of the acyl isethionates and the free fatty acids in the mixture prepared as above are given in Table II.

TABLE I

	BAR COMPOSITIONS			
Composition No.	1.	2.	3.	4.
Na acyl isethionate from partially hydrogenated mixed coconut oil fatty acids	49.80	—	—	—
Na acyl isethionate from C_{10}, C_{12} and C_{14} fatty acids	—	31.00	33.00	35.00
Na acyl isethionate from C_{16} and C_{18} fatty acids	—	10.00	8.00	6.00
Na dodecylbenzenesulfonate	2.02	2.02	2.02	2.02
Na tallow soap	6.66	8.66	8.66	8.66
Na coconut oil soap	1.66	2.16	2.16	2.16
Na stearate	2.98	2.98	2.98	2.98
Na isethionate	4.78	4.78	4.78	4.78
Free fatty acid	23.23	30.73	30.73	30.73
NaCl	0.35	0.35	0.35	0.35
Misc. extraneous matter associated with the components	3.27	2.07	2.07	2.18
Water	5.25	5.25	5.25	5.14
	100.00	100.00	100.00	100.00
Fatty Acid Distribution				
C_8	3.0	none	none	none
C_{10}	5.0	31.0	33.0	35.0
C_{12}	45.0	20.0	21.2	22.5
C_{14}	18.0	18.2	19.4	20.6
C_{16}	14.0	16.7	14.2	11.7
C_{18}	10.0	14.1	12.2	10.2
$C_{18:1}$ (oleic acid)	5.0	none	none	none
	100.0	100.0	100.0	100.0

TABLE II

FATTY ACID DISTRIBUTION
ACYL ISETHIONATES

	From partially hydrogenated coconut fatty acid (control)	From C_{10}–C_{14} Fatty Acids	From Triple-Pressed Stearic Acid
C_6	—	—	—
C_8	3.2	—	—
C_{10}	5.0	41.0	—
C_{12}	45.0	26.4	—
C_{14}	18.0	24.1	—
C_{16}	14.0[a]	4.3[a]	55.0
C_{18}	10.2[a]	4.2[a]	45.0
$C_{18.1}$	5.0	—	—
C_{20}	—	—	—
	Free Fatty Acid		
C_6	—	—	—
C_8	0.6	—	—
C_{10}	2.1	13.0	—
C_{12}	29.0	15.0	—
C_{14}	15.0	20.3	—
C_{16}	25.0	25.3	55.0
C_{18}	23.0	25.3	45.0
$C_{18.1}$	4.2	0.5	—
C_{20}	0.7	0.1	—

[a]introduced into the acyl isethionate molecule by reaction with the C_{16}–C_{18} fatty acids used for fluidizing.

Wear rate and lathering properties of the bars are determined by methods described elsewhere herein.

A slight decrease in wear rate and a slight decrease in lather volume are noted as the proportion of C_{16} and C_{18} isethionate is increased in the foregoing series. All three compositions however by statistical analysis are judged to be substantially equal to control bar No. 1 in these properties.

The evaluation clearly shows the advantages of employing the selected mixture of acyl isethionate chain lengths within the instant invention, i.e., substantially the same lathering and rate of wear as are characteristic of acyl isethionate bars heretofore known can be obtained at a lower acyl isethionate content with the bars of the invention, along with the processing advantages hereinbefore described. Within the broader percentage ranges of acyl isethionate chain lengths set forth herein there are insignificant decreases in lather volume and improvements in wearing quality as the proportions of the higher molecular weight acyl isethionates are increased.

EXAMPLE 2

Mixtures of fatty acids of varying chain lengths are prepared and reacted with sodium isethionate in accordance with the procedures of Holland et al. and McCrimlisk described hereinbefore. The chain-length distributions of the acyl portions of the acyl isethionates resulting from the procedure are shown below in Table III, and further illustrate the variations contemplated within the instant invention.

The composition distributions are expressed in percent fatty acids, based on the total of the fatty acids combined as acyl isethionates.

TABLE III

Composition No.	5	6	7	8	9	10	11	12	13
	PERCENT								
Decanoyl	28	29	30	32	32	33	34	35	37
Dodecanoyl	23	23	22	21	23	24	20	23	18
Tetradecanoyl	19	20	19	21	18	18	20	17	16
Hexadecanoyl	20	11	14	14	16	11	12	9	19
Octadecanoyl	10	17	15	12	11	14	14	16	10
	100	100	100	100	100	100	100	100	100

EXAMPLE 3

The mixed acyl isethionates prepared as in Example 2 are formulated into toilet detergent bars containing 45% of the acyl isethionate, and 26.73% free fatty acid, the balance of the compositions being the same as in bars 2 and 3 of Table I.

EXAMPLE 4

Acyl isethionate mixtures having the acyl distribution of composition 10 in Table III, Example 2, are formulated into toilet detergent bars at levels of 25 and 60%, in conjunction with three supplemental detergents, namely, sodium lauryl sulfate, sodium alkyl benzenesulfonate wherein the alkyl group has about 13 carbon atoms, and the fatty acid amides of N-methyl taurine, wherein the fatty acid portion is derived from coconut oil. The compositions are shown in Table IV.

TABLE IV

The following are illustrative of the toilet bar compositions within the present invention.

Composition No.	1	2	3	4	5	6
	PERCENT					
Acyl isethionate[a]	25	25	25	60	60	60
Na tallow soap	25	20	18	5	5	3
Stearic acid	40	38	36	15	15	10
Na lauryl sulfate	5	—	—	2	—	—
Na dodecylbenzene-sulfonate	—	10	—	—	2	—
Na coco amide of N-methyl taurine	—	—	10	—	—	7
Na stearate	3	3	3	3	3	3
Na isethionate	4	4	4	4	4	4
Misc. extraneous matter associated with the components	2	2	2	3	3	3
Water	3	5	9	4	6	8
Pigment + Perfume	2	2	2	2	2	2
	100	100	100	100	100	100

[a]The acyl portion is a mixture having the chain-length distribution of the mixture set forth in composition No. 10, Table III, above.

Having described the invention, many modifications will occur to those skilled in the art and the invention is to be limited only by the scope of the appended claims.

What is claimed is:

1. A mixture of fatty acids comprising about 28 to about 37% decanoic acid, about 18 to about 24% dodecanoic acid, about 16 to about 21% tetradecanoic acid, about 9 to about 20% hexadecanoic acid, and about 10 to about 17% octadecanoic acid, said percentages being on the total fatty acid basis, and said mixture being adaptable for use in the preparation of an acyl isethionate mixture having superior lathering properties.

2. A sodium acyl isethionate mixture having mixed acyl groups of 10, 12, 14, 16, and 18 carbon atoms, said acyl groups having the molecular weight distribution of the fatty acid composition of claim 1.

3. A detergent bar comprising from about 25 to about 60% of a water-soluble alkali-metal detergent salt of an ester of isethionic acid with mixed aliphatic fatty acids having from 10 to 18 carbon atoms and an iodine value of 0 to about 1, said mixture of fatty acids comprising about 28 to 37% decanoic, about 18 to 24% dodecanoic, about 16 to 21% tetradecanoic, about 9 to 20% hexadecanoic, and about 10 to 17% octadecanoic acids, total esterified acid basis, from 0 to about 10% of at least one water-soluble suds-boosting detergent salt selected from the group consisting of alkali metal and organic amine higher aliphatic fatty alcohol sulfates, alkyl aryl sulfonates, and higher aliphatic fatty acid taurides, from about 1 to about 9% water, from about 2.5 to about 25% of water-soluble higher fatty acid soap, and from 10 to 40% of at least one higher fatty acid having from about 12 to about 25 carbon atoms as a binder and plasticizer, said bar having a pH within the range from 6 to 8, measured as a 10% aqueous solution of the bar composition at 35°C.

4. A mixture of saturated fatty acids wherein decanoic acid is present in the proportion of about 50%, dodecanoic acid is present in the proportion of about 25%, and tetradecanoic acid is present in the proportion of about 25%, basis by weight of said mixture.

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UNITED STATES PATENT OFFICE

CERTIFICATE OF CORRECTION

PATENT NO. : 3,879,309

DATED : April 22, 1975

INVENTOR(S) : Louis Gatti and Raymond George Matthaei

It is certified that error appears in the above-identified patent and that said Letters Patent are hereby corrected as shown below:

Under the heading of the Title Page, an Assignee should be included as follows:

Assignee: Lever Brothers Company, New York, N.Y.

Signed and Sealed this

fifth Day of *August 1975*

[SEAL]

Attest:

RUTH C. MASON
Attesting Officer

C. MARSHALL DANN
Commissioner of Patents and Trademarks

UNITED STATES PATENT OFFICE

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[SEAL]

Attest:

RUTH C. MASON
Attesting Officer

C. MARSHALL DANN
Commissioner of Patents and Trademarks