United States Patent

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[54] **HYDROGENATED OLEFIN SULFONATE DETERGENT BARS**
11 Claims, No Drawings

[52] **U.S. Cl.** **252/554, 252/555**
[51] **Int. Cl.** **C11d 1/12**
[50] **Field of Search** 252/161; 260/513

[56] **References Cited**

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ABSTRACT: Nonsoap detergent toilet bars are prepared from a complex mixture of hydrogenated olefin sulfonates containing from 10 to 24 carbon atoms and a plasticizing amount of water.

HYDROGENATED OLEFIN SULFONATE DETERGENT BARS

BACKGROUND OF INVENTION

The present invention if concerned with the field of synthetic nonsoap detergent bars and more particularly with the preparation of bars or cakes for toilet or bath use from complex mixtures of hydrogenated olefin sulfonates.

Although synthetic detergents have largely replaced soaps for most household laundering and dishwashing uses, they have found little acceptance in the household toilet bar area. Although the detergent literature is replete with examples of synthetic detergent bars and synthetic detergent-soap combination bars, the toilet bar market continues to be dominated by soap bars. The combination bars have had appreciable acceptance but they exhibit the high pH characteristic of soap bars. At the present time it appears that less than 1 percent of the bar market is satisfied by all-synthetic detergent bars.

Two factors seem to account for the small impact of all-synthetic detergent bars on the bar market, physical property deficiencies and high cost.

The essential physical properties requires in a synthetic detergent bar use are relatively low solubility and a generally amorphous rather than crystalline character. High solubility results in high wear rate, short life in use and sloughing when the bar is laid aside wet or allowed to stand on a wet surface. High crystallinity makes it extremely difficult to press the detergent into a bar which has good physical strength and is resistant to cracking or crumbling. The alkyl benzene sulfonates are representative of the synthetic detergents which have failed in bar use because of too high solubility and while many suggestions have been made as to additives which would control the high solubility deficiency, no bar based on a highly soluble detergent has succeeded commercially. The alkyl sulfates are representative of synthetic detergents which have failed in bar use because of too crystalline a character. Various additives have been proposed for use with the alkyl sulfates in forming bars but no satisfactory combination appears to have been found. It is common experience that an additive which does tend to modify the undesirable properties of a synthetic detergent introduces new problems which arise out of the character of the additive itself, for example, the development of rancidity on storage.

DESCRIPTION OF INVENTION

It has now been discovered that superior nonsoap synthetic detergent bars can be formed employing hydrogenated linear olefin sulfonates as the major active detergent component. In particular, mixtures of straight-chain hydrogenated olefin sulfonates containing from 10 to 24 carbons constitute a superior detergent component for nonsoap detergent toilet bars.

Not only do nonsoap detergent bars based on hydrogenated olefin sulfonate as the major active detergent material have excellent physical properties, they also have a pH in the range 6.5 to 7, the pH being measured in a 1 percent solution of the active material in water. In this respect bars based on hydrogenated olefin sulfonate are markedly superior to soap and to combination soap-synthetic detergent bars. The importance of the pH property is clearly shown by Dr. Irwin Kantor's paper entitled, "Value of a Neutral Detergent Bar Instead of Alkaline Soap in the Routine Care of Aging Skin," Journal of the American Geriatric Society, Mar. 1962, pp. 242–246. In addition to the superior physical and chemical properties of nonsoap synthetic detergent bars based on hydrogenated olefin sulfonate, it should be noted that these properties are found not in an esoteric chemical compound but in a material that can be easily and cheaply produced in large volume from readily available and inexpensive starting materials.

The term "olefin sulfonates" as used in the present invention defines the complex mixture obtained by the SO_3 sulfonation of straight-chain olefins containing 10 to 24 carbon atoms and subsequent neutralization and hydrolysis of the sulfonation reaction product. This complex mixture contains hydroxyalkane sulfonates and alkene sulfonates as its major components and a lesser proportion of disulfonated product.

While the general nature of the major components of the complex mixture is known, the specific identity and the relative proportions of the various hydroxy sulfonate and disulfonate radicals and double bond locations are unknown. Accordingly, a determination of the entire chemical makeup is exceedingly difficult and has not heretofore been successfully accomplished. The mixture is best defined by the process used for producing it.

Optimum detergent bar properties are exhibited by an olefin sulfonate product which contains from about 25 to 75 percent by weight alkene sulfonates, from about 25 to 65 percent by weight hydroxyalkane sulfonates and not more than 20 weight percent disulfonates. These optimum compositions are obtained by SO_3-air sulfonation of C_{10-24} straight-chain olefins with an SO_3:air volume ratio of about 1 to 5–100 and an SO_3:olefin mole ratio of 0.95 to 1.15, and neutralization and hydrolysis of the sulfonation reaction product at temperatures of 145° to 200° C. using one equivalent of base per mole of SO_3 consumed in the sulfonation step.

The following examples describe the preparation of hydrogenated olefin sulfonates and of detergent bars using hydrogenated olefin sulfonates as the major active detergent component.

Example 1—Preparation of Olefin Sulfonates

The reactor used for this sulfonation consisted of a continuous falling film-type unit in the form of a vertical water-jacketed tube. Both the olefin and the SO_3-air mixture were introduced at the top of the reactor and flowed concurrently down the reactor. At the bottom the sulfonated product was separated from the air stream.

The feed was a straight-chain 1-olefin blend produced by cracking highly paraffinic wax and having the following composition by weight: 1 percent tetradecene, 27 percent pentadecene, 29 percent hexadecene, 28 percent heptadecene, 14 percent octadecene and 1 percent nonadecene. This material was charged to the top of the above described reactor at a rate of 306 pounds/hour. At the same time 124.2 pounds/hour of SO_3 diluted with air to 3 percent by volume concentration of SO_3 was introduced into the top of the reactor. The reactor was cooled with water to maintain the temperature of the effluent product within the range of 43°–46° C. The average residence time of the reactants in the reactor was less than two minutes.

After passing out of the reactor the sulfonated product was mixed with 612 pounds/hour of 11.2 percent aqueous caustic and heated to 145°–150° C. in a tubular reactor at an average residence time of 30 minutes. This step neutralized the sulfonic acids contained in the sulfonation reaction product, hydrolyzed the sultones to hydroxy sulfonic acids and neutralized the hydroxy sulfonic acids. Olefin sulfonates were produced at the rate of 463 pounds per hour as an aqueous solution having a 45 percent by weight solids content and a pH of 10.8.

A portion of this product was analyzed and shown to be made up of the sodium salts of alkene sulfonic acids, hydroxy alkane sulfonic acids, and disulfonic acids. These three major components were present in a weight ratio of about 50/35/15.

Example 2—Preparation of Olefin Sulfonates

A straight-chain 1-olefin mixture produced by cracking a highly paraffinic wax and containing 1 percent tetradecene, 18 percent pentadecene, 17 percent hexadecene, 16 percent heptadecene, 16 percent octadecene, 14 percent nonadecene, 13 percent eicosene, and 5 percent heneicosene was processed following the procedure in example 1.

In addition to the straight-chain alpha-olefins from wax cracking suitable olefin starting materials include straight-chain alpha-olefins produced by Ziegler polymerization of ethylene, or internal straight-chain olefins prepared by catalytic dehydrogenation of normal paraffins or by chlorination-

dehydrochlorination of normal paraffins. The olefins may contain from 10 to 24 carbon atoms, usually 13 to 22 carbon atoms, and preferably 15 to 18 carbon atoms per molecule. Olefin mixtures should have an average molecular weight of at least about 200.

The amount of SO_3 utilized in the sulfonation reaction may be varied but is usually within the range of 0.95 to 1.25 moles of SO_3 per mole of olefin and preferably in the range 1:1 to 1:1.15. Greater formation of disulfonated products is observed at higher SO_3:olefin ratios. Disulfonation may be reduced by carrying the sulfonation reaction only to partial conversion of the olefin, for example by using SO_3:olefin ratios of less than 1 and removing the unreacted olefins by a deoiling process. The unreacted olefins may be removed by extracting the reaction product with a hydrocarbon such as pentane.

In order to obtain a product of good color, the SO_3 employed in the sulfonation reaction is generally mixed with an inert diluent or with a modifying agent. Inert diluents which are satisfactory for this purpose include air, nitrogen, SO_2, dichloromethane, etc. The volume ratio of SO_3 to diluent is usually within the range of 1:100 to 1:1.

The reaction product from the sulfonation step may be neutralized with aqueous basic solutions containing compounds such as hydroxides, carbonates and oxides of the alkali metals, alkaline earth metals and ammonium. In the preferred method sufficient neutralizing solution may be added to provide for neutralization of the hydroxy alkane sulfonic acids formed by sultone hydrolysis. Generally, one equivalent of base for each mole of SO_3 consumed in the sulfonation reaction is added to the sulfonation reaction product.

The proportion of hydroxyalkane sulfonates to alkene sulfonates in the hydrolyzed neutralized product may be varied somewhat by the manner in which neutralization and hydrolysis are carried out. Thus reduced amounts of hydroxyalkane sulfonates are obtained by carrying out the neutralization and hydrolysis at temperatures in the range of 145°–200° C. while higher yields of hydroxy sulfonate are favored by carrying out the neutralization and hydrolyses at temperatures below 100° C. Suitable hydrolysis temperatures range from about 100° to 200° C.

Example 3—Preparation of Hydrogenated Olefin Sulfonates

The apparatus for this hydrogenation consisted of a 1-liter Magne-Drive autoclave equipped with an accumulator, a constant pressure regulator, and a temperature recording means. The product of example 1 was diluted with water to a 26 percent solids concentration and was filtered to remove a trace amount of insoluble material. The pH was adjusted to a value of 6.5–7.5 by neutralizing the slight excess of NaOH used in the neutralization and hydrolysis step with H_2SO_4 and 100 parts of 30 percent hydrogen peroxide was added to 3,850 parts of the filtered 26 percent solution in an open glass vessel. This mixture was heated to 80° C. and stirred for 1 hour at this temperature, after which time no hydrogen peroxide remained. After cooling this solution to room temperature, 650 g. of it was charged to the previously described autoclave along with 8.5 g. of Raney nickel, The system was purged with nitrogen and then with hydrogen. It was them pressured with hydrogen to 50 p.s.i.g. The autoclave was warmed to 100° C. at which temperature hydrogen was again introduced to bring the pressure up to 100 p.s.i.g. The hydrogen pressure was maintained constant at 100 p.s.i.g. throughout the run. After 1½ hours of stirring at this temperature and pressure, and at which time there was no additional hydrogen uptake, the solution was cooled to about 70° C. filtered, and then allowed to cool.

Example 4—Preparation of Hydrogenated Olefin Sulfonates

The product of example 2 was reduced as in example 3 to give a substantially 100 percent reduction of double bonds in the olefin sulfonate.

The hydrogen peroxide treating step prior to hydrogenation increases hydrogenation efficiency. The olefin sulfonate prior to such treatment contains unidentified compounds which poison hydrogenation catalysts. Without the hydrogen peroxide pretreat catalyst consumption is much higher. Other oxidizing agents may be used instead of hydrogen peroxide in the pretreating step, preferably oxidizing agents which leave no solid residues in the product such as elemental oxygen or air.

In addition to the Raney nickel exemplified, a wide variety of known hydrogenation catalysts may be used in the hydrogenation step. These include the noble metals and various forms of nickel other than Raney nickel such as nickel on kieselguhr, and other supported nickel catalysts. Palladium on carbon and ruthenium on alumina are effective noble metal catalysts, although Raney nickel and palladium on carbon are preferred catalysts.

The amount of catalyst employed in the hydrogenation of olefin sulfonates may vary in a range from about 0.05 to 30 percent by weight based on the olefin sulfonate present. Increasing the amount of catalyst will usually result in a shortening of the time necessary for complete hydrogenation.

The hydrogenation reaction is usually carried out at temperatures of from about 20° to 200° C. and preferably 70° C. to 120° C. At temperatures appreciably above 200° C. unnecessary hydrogenation of hydroxyalkane sulfonates and hydrogenative degradation of the product tend to occur.

Hydrogen pressure during the reaction is not a critical variable. Reduction may be carried out at pressures varying from less than atmospheric to 5,000 p.s.i.g., but preferably from 30 to 200 p.s.i.g.

In examples 3 and 4 above hydrogenation of the alkene sulfonate component of the neutralized sulfonation reaction mixture to alkane sulfonate was essentially complete. Partial hydrogenation of the olefin sulfonate to the extent that at least 50 percent of the alkene sulfonate is converted to alkane sulfonate yields a hydrogenated olefin sulfonate suitable for use in producing high quality nonsoap detergent bars. Partial hydrogenation may be accomplished by proceeding as in example 3 but discontinuing the hydrogenation reaction before hydrogen takeup ceases. Partial hydrogenation can also be carried out by subjecting the olefin sulfonate to hydrogenation after neutralization but prior to hydrolysis.

Example 5—Preparation of Partially Hydrogenated Olefin Sulfonate

The procedure of example 3 was followed except that reduction was allowed to continue for only 30 minutes. The product was worked up as before. Analysis of a small aliquot by bromine number titration showed that 55 percent of the double bonds originally contained had been saturated.

Example 6—Hydrogenation of an Unhydrolyzed Olefin Sulfonate

1-Hexadecene was sulfonated in a continuous falling-film reactor with SO_3/olefin mole ratio of about 1.2. The product from this reaction, 184 g. was dissolved in 198 g. of dioxane to give a 48 percent solution.

Fifty grams of this 48 percent solution was heated with stirring to 60° C. Then 2.1 g. of 34 percent hydrogen peroxide was added. Stirring was continued at this temperature for one hour. The solution was allowed to cool to room temperature.

Of the hydrogen peroxide treated material, 17.3 g. was diluted to 50 ml. with dioxane and charged to a 200 ml. Fischer-Porter bottle along with 0.80 g. of 5 percent palladium-on-carbon hydrogenation catalyst. The mixture was heated to 24°–26° C. and pressured to 50.5 p.s.i.g. with hydrogen. After 30 minutes of reaction, the pressure had dropped 18.4 p.s.i.g. No further drop in pressure occurred after this time. This pressure drop corresponds to hydrogenation of 31 percent of the original olefin-SO_3 reaction product.

The hydrogenated material was filtered to remove the catalyst. The dioxane was removed by evaporation at temperatures below 40° C. under reduced pressure. In this way, there was obtained 8.3 g. of a low melting solid. A portion of this solid, 6.4 g. was mixed with 2.0 g. of 50 percent aqueous sodium hydroxide in 35 ml. of water and heated at 150°–155° C. for 2 hours. The water was then removed by evaporation. A bromine number analysis of the final product indicated that about 25 percent of the product was unsaturated. Ac-

cordingly, the calculated weight ratio of hydroxyalkane, alkene and alkane sulfonates would be 44/25/31, respectively.

Example 7—Preparation of a Hydrogenated Alpha-Olefin Sulfonate Bar

Detergent bars are prepared from various hydrogenated olefin sulfonates by the following procedure. In the first step the hydrogenated olefin sulfonate and 2–15 percent water are milled into ribbons to provide a homogeneous composition. It is then formed into a bar by molding in a conventional soap bar mold. The bars formed in this way are about 2⅛ inch by 1⅝ inch by ⅝ inch in size. These bars are aged by exposure to air in a room at ambient temperature and humidity for 1 week. The bars then weigh between 23 and 26 grams.

The cohesiveness of the bars is measured by determinating the impact strength according to the method of ASTM D 256–56 part 27. In this procedure the test specimen was 0.44–0.48 inch (corrected to 0.50 inch) in the direction of impact and 1⅜ inch in width.

Bars of varying hydroxyalkane sulfonate contents were tested for strength and cohesiveness.

TABLE I

Strength of Hydrogenated Olefin Sulfonate Bars

Sample No.	% Hydroxyalkane Sulfonate	Impact Strength in./lb.
1	0[1]	2.07[2]
2	25	2.34
3	45	4.72
4	82	2.47[2]

[1]Primary paraffin sulfonate obtained by the addition of bisulfite to a mixture of 1-olefins containing from 15 to 20 carbon atoms.

[2]Brittle, easily cracked.

Although sample 4 possessed good impact strength, it lacked cohesiveness, was quite brittle and cracked easily as did sample 1.

The data in table I indicates that the better bars have a substantial content of hydroxyalkane sulfonate. Hydroxyalkane sulfonate contents in the range 25 to 65 percent by weight have superior cohesiveness and impact strength.

Bars prepared in accordance with example 7 were also tested for slough loss and wear rate and the results are given in table II. The slough loss test was run by placing the bar in a 3½ inch I.D. Petri dish containing 30 ml. of water having 50 p.p.m. hardness. After 18 hours the bar was removed and any loose gel was rubbed off. Then the bar was allowed to dry for 24 hours and weighed. The loss in weight in grams of the bar is reported as slough loss.

An important property of detergent bars is their solution rate under actual washing conditions. A convenient method of measuring the solubility characteristics of a detergent bar is by determining its loss in weight in grams per handwashing. This value is referred to as the bar wear rate. The values are obtained by averaging the weight loss from a number of hand washings by different individuals using tap water at temperatures of about 110° F.

TABLE II

Wear Rate and Slough Loss of Hydrogenated Olefin Sulfonate Bars

Sample No.	Active Source	Wear Rate, g./Washing	Slough Loss, g.
1	Example 1	0.99	23.5
2	Example 3	0.63	9.0
3	Example 4	0.63	9.2
4	Example 4	0.60	7.2

The importance of slough loss and wear rate is particularly important in terms of the useful life of the detergent bars. As can be seen from table II the hydrogenated olefin sulfonate bars show an excellent low wear rate and slough loss.

The thermal stability of the bars of the present invention was compared to that of other detergent bars. Thermal stability is a measure of the resistance of the bars to discoloration and odorification with aging. Bars of low thermal stability require the addition of large amounts of perfumes and coloring additives to mask undesirable odors and discoloration.

In this test the bars were ground into a fine powder and 4 g. were placed in a stoppered weighing bottle (80 mm. height and 40 mm. I.D.) and exposed to the air in an oven at a temperature of 70° C. Both odor and color were observed periodically. The odor was classified as to increasing intensity as follows: odorless, trace, slight, mild, moderate and strong. The nature of the odor was observed to be rancid in the indicated examples. The results of this test are given in table III.

TABLE III.—THERMAL STABILITY OF DETERGENT BARS

Sample No.	Test material	Description of test sample at time, days: 0 Odor	0 Color	5 Odor	5 Color	12 Odor	12 Color	19 Odor	19 Color
1	Example 4	Slight	White	Slight	White	Slight	White	Mild	White.
2	70/30 mixture of C_{15}–C_{30} PPS[1] and LAS.[2]	do	Very pale yellow.	Mild	Tan	Mild (rancid)	Tan	Strong (rancid).	Pale brown.
3	70/30 mixture of C_{15}–C_{20} PPS and SAS.[3]	Trace	White	Moderate	Pale tan	Moderate	Light tan	Moderate (rancid).	Tan.

[1] See Table I, footnote 1, for definition.
[2] LAS, linear alkylbenzene sulfonate wherein the linear alkyl group has from 11 to 14 carbon atoms in the chain.
[3] SAS, secondary alcohol sulfates prepared from straight-chain secondary alcohols having from 15 to 18 carbon atoms in the chain.

The data in table III indicates that the hydrogenated olefin sulfonate bars possess both excellent odor and color retention characteristics as compared to other detergent bars.

Bars of essentially equivalent properties were prepared as in the above example using hydrogenated olefin sulfonate in which the cation was potassium, magnesium and a 50/50 mixture of sodium and magnesium.

While completely satisfactory bars can be prepared from hydrogenated olefin sulfonate as shown in example 7, the feel and appearance of the bars may be improved by incorporation of conventional emollients, superfatting agents, opacifiers, fillers, perfumes, dyes and the like. These additives may constitute up to about 40 percent by weight of the finished bar. Representative conventional additives are the polyethylene glycols, C_{12}–C_{16} fatty alcohols, stearic acid, mineral oil, fatty acid amides, mixed fatty acid alkanolamine compounds, lau-

ric-isopropanolamide, polyethylene glycol monostearates and glycerol monostearate.

Example 8

Hydrogenated olefin sulfonate prepared pursuant to example 3 and bleached with NaOCl having a small content of NaCl resulting from the bleach, a small content of sodium sulfate produced by neutralization of unreacted SO_3 and a small content of unsulfonated oil was mixed into a bar formulation having the following composition:

HAOS BARS—COMPOSITION

	Wt. %
C_{16}-½ C_{18} HAOS	61.2
Glycerol monostearate	3.8
Carbowax® (1) 4000	3.8
Stearic Acid (triple pressed)	15.1
NaCl (Est.)	3.2
Na_2SO_4 (Est.)	2.5
Oil	2.2
H_2O	3.9
Perfume	0.95
Trichlorocarbanilide	1.4
Hexachlorophene	1.4
Versene	0.3
TiO_2	0.25
Dye	Tr.
Total	100.00

(1) Polyethylene glycol of 3,000–3,700 Avg. Molecular Wt.

The glycerol monostearate, carbowax and stearic acid were added to give the solid a gloss and soaplike feel. The trichlorocarbanilide and hexachlorophene are bactericides. The titanium dioxide provides opacity. The versene was added because the bar mill to be used experienced some corrosion and the chelate prevented discoloration due to iron. The water is the plasticizer. Five hundred and fifteen pounds of this formulation were run through an amalgamator, two two-stage plodders, a cutter, an aging tunnel and a stamper to produce 3,000 bars 68 mm. long, 42 mm. wide and 15 mm. thick.

The bars had excellent texture, a soaplike feel, were thermally stable, had high impact strength and cohesiveness and low slough loss.

In addition it may be desirable to have incorporated within the nonsoap synthetic bar other detergent active materials compatible with the hydrogenated olefin sulfonates in an amount of from 0 to 25 percent by weight of the hydrogenated olefin sulfonates. Such detergent actives would include straight-chain alkylbenzene sulfonates, straight-chain secondary alkyl sulfates, polyoxyethylene alkylphenol sulfates, acylisothionates, alkyl glyceryl ether sulfonates and sulfated fatty acid monoglycerides.

As will be evident to those skilled in the art, various modifications of the present products can be made or followed, in the light of the foregoing disclosure and discussion without departing from the spirit or scope of the disclosure or the scope of the following claims.

We claim:

1. A nonsoap detergent bar consisting essentially of
   A. a mixture of hydrogenated olefin sulfonates obtained by
      1. sulfonating straight-chain olefins containing from 10 to 24 carbon atoms with diluted SO_3 at an SO_3:olefin mole ratio of 0.95 to 1.25,
      2. neutralizing the product of (1) with at least one equivalent of base per mole of SO_3 consumed in (1) and hydrolyzing the product of (1) at a temperature of between 100° and 200° C. and
      3. hydrogenating from 50 to 100 percent of the unsaturated carbon-carbon double bonds in the product of (2), and
   B. from about 2 to 15 percent by weight of water based on the hydrogenated olefin sulfonate content of the bar.

2. A nonsoap detergent bar as in claim 1 wherein the straight-chain olefins are alpha-olefins.

3. A nonsoap detergent bar as in claim 2 wherein the straight-chain olefin contains from 15 to 20 carbon atoms.

4. A nonsoap detergent bar as in claim 3 wherein the sulfonation reaction is conducted with an SO_3:olefin ratio of from 0.95 to 1.15 and the hydrolysis reaction is conducted at temperatures of from 145° to 200° C. so as to produce an olefin sulfonate composition having a content of from about 25 to 65 weight percent hydroxyalkane sulfonates and less than 20 weight percent of disulfonates.

5. A nonsoap detergent bar as in claim 4 wherein from 75 to 100 percent of the unsaturated carbon-carbon double bonds are hydrogenated.

6. A nonsoap detergent bar consisting essentially of a mixture of hydrogenated olefin sulfonate obtained by
   1. sulfonating straight-chain olefins containing from 10 to 24 carbon atoms with diluted SO_3,
   2. neutralizing the product of (1),
   3. hydrogenating from 75 to 100 percent of the unsaturated carbon-carbon double bonds in the product of (2), and
   4. hydrolyzing the product of (3), and a plasticizing amount of water in the range of 2 to 15 percent by weight based on the weight of hydrogenated olefin sulfonate.

7. A nonsoap detergent bar as in claim 6 wherein the straight-chain olefins are alpha-olefins.

8. A nonsoap detergent bar as in claim 7 wherein the straight-chain olefin contains from 15 to 20 carbon atoms.

9. A nonsoap detergent bar as in claim 8 wherein the sulfonation reaction is conducted with an SO_3:olefin ratio of from 0.95 to 1.15 and the hydrolysis reaction is conducted at temperatures of from 145° to 200° C. so as to produce an olefin sulfonate composition containing from 25 to 65 weight percent hydroxy-alkane sulfonates and less than 20 weight percent of disulfonates.

10. A nonsoap synthetic detergent bar containing
   1. at least 60 percent by weight of active detergent material of which at least 80 percent by weight consists of the product obtained by
      a. reacting linear monoolefins containing from 10 to 24 carbon atoms and having an average molecular weight of at least 200 with sulfur trioxide,
      b. reacting the product obtained in (a) with about one equivalent of an inorganic base per mole of combined sulfur trioxide,
   2. from 0 to 25 percent by weight based on the product of (1), (a), (b) and (c) of a synthetic detergent selected from the group consisting of straight-chain alkyl benzene sulfonates and straight-chain alkyl sulfates,
   3. a quantity of water in the range 2 to 15 percent by weight of the active detergent component sufficient to plasticize the active detergent component, and
   4. conventional additives such as superfatty agents, fillers, perfumes, and the like, to constitute the remainder of the bar.

11. A nonsoap detergent bar consisting essentially of a mixture of hydrogenated olefin sulfonates obtained by
   1. sulfonating straight-chain olefins containing from 10 to 24 carbon atoms with diluted SO_3,
   2. hydrogenating from 50 to 100 percent of the unsaturated carbon-carbon double bonds in the product of (1), and
   3. neutralizing and hydrolyzing the product of (2); and from about 2 to 15 percent by weight of water based on the hydrogenated olefin sulfonate content.

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UNITED STATES PATENT OFFICE
CERTIFICATE OF CORRECTION

Patent No. 3,625,910 Dated December 7, 1971

Inventor(s) William Alan Sweeney and Gar Lok Woo

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

Col. 1, line 23, "requires" should read --required--.

Col. 2, line 19, "5-100" should read --50-100--.

Col. 3, line 59, "them" should read --then--.

Table III, Sample No. 2, "C_{15}-C_{30}" should read --C_{15}-C_{20}--.

Claim 10, line 48, after "trioxide," add

-- (c) hydrogenating the product of (b) to reduce its ethylenic saturation by at least 50%, --.

Signed and sealed this 23rd day of May 1972.

(SEAL)
Attest:

EDWARD M.FLETCHER,JR.
Attesting Officer

ROBERT GOTTSCHALK
Commissioner of Patents