MILLED DETERGENT BAR

Richard Henry Okenfuss, Cincinnati, Ohio, assignor to The Procter & Gamble Company, Cincinnati, Ohio, a corporation of Ohio

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This invention relates to detergent compositions and processes for preparing said compositions. Specifically, this invention relates to a detergent laundry bar which has acceptable sudsing characteristics in warm and cool, hard and soft water; which has an acceptable wear rate; which has an acceptable feel, and which has a pleasing appearance. This invention also relates to a process for neutralizing a detergent material by a dry-mix method, the neutralized detergent material being useful, for example, in the preparation of said detergent laundry bar.

For many years, people have tried to formulate a synthetic detergent laundry bar which would have the acceptable features hereinbefore enumerated. The usual approach has been to combine the synthetic detergent with some essentially inert organic material such as starch or insoluble soap to enable one physically to form a bar. This results in an expensive bar which is normally at a cost disadvantage with respect to the usual laundry soap bars and which may be satisfactory for toilet purposes, but not for heavy-duty laundry operations.

An object of this invention is to provide a synthetic detergent laundry bar which will be inexpensive and have the acceptable features hereinbefore enumerated, and a process for preparing said bar.

A further object of this invention is to provide a process for dry neutralization of detergent materials.

THE PRODUCT

The first object of this invention can be achieved by formulating a homogeneous mechanically worked synthetic detergent bar consisting essentially of: (A) from about 12% to about 30% by weight of detergent surfactant selected from the group consisting of alkali and alkaline earth metal alkyl aryl sulfonates, alkyl sulfonates, alkyl ethylene oxide ether sulfates, acyl N-methyl taurides, alkyl sulfates, and acyl monoglyceride sulfates; (B) from about 10% to about 25% by weight of sodium tripolyphosphate; (C) from about 25% to about 55% by weight of sodium bicarbonate; (D) from about 2% to about 15% by weight of trisodium orthophosphate; (E) from 0% to about 5% by weight of amide selected from the group consisting of ammonia amides, monoethanol amides, and diethanol amides of fatty acids having an acyl chain of from about 8 to about 18 carbon atoms; (F) from 0% to about 5% by weight of silicate solids with an Na₂O:SiO₂ ratio of from about 1.0:0.90 to about 1.0:3.25; (G) from about 5% to about 25% by weight of water; and (H) the balance minor ingredients, the total of (A), (B), (C), and (D) being at least 75% of the bar, all weight percents except those in (G) being on an anhydrous basis: preferably, the sodium tripolyphosphate is hydrated.

Ingredients (A) through (D) and (G) in the above formulation, are essential to the preparation of an effective and acceptable laundry detergent bar. The detergent surfactant (active) is essential to provide cleaning and sudsing and to act as a plasticizer to hold the formulation in the shape of a bar. When less than about 12% by weight of the bar is detergent surfactant, the bar will not give acceptable cleaning and sudsing. Also, the bar will not have satisfactory strength since it will be difficult to weld the bar together during extrusion. There is no real advantage obtained by formulating a bar containing more than about 30%, by weight of the bar, of detergent surfactant, and if too large an excess of the detergent surfactant is used, the product becomes sticky and much more difficult to handle in processing. The preferred level is about 24% by weight of the bar and the preferred active is sodium tetrapropylene benzene sulfonate.

Examples of suitable detergent surfactants are:

1. Alkali metal and alkaline earth metal alkyl aryl sulfonates containing alkyl groups of from about 9 to about 15 carbon atoms, especially those of the types described in United States Letters Patent Numbers 2,220,099 and 2,477,383. The alkyl group can be derived from alcohol derivatives, e.g., halides, prepared from naturally occurring glycerides such as coconut oil, palm oil, etc., or from petroleum or olefins such as the propylketone trimers, tetramer, and pentamer. The aryl group can be either benzene or naphthalene. Examples of these detergent surfactants include sodium tetrapropylene benzene sulfonate and sodium n-dodecyl benzene sulfonate.

2. Alkali metal and alkaline earth metal (a) alkyl sulfates, (b) alkyl sulfonates, and (c) alkyl ethylene oxide ether sulfates wherein said alkyl groups contain from about 8 to about 20 carbon atoms and wherein said alkyl ethylene oxide ether sulfates contain from one to about nine moles of ethylene oxide per molecule. Said alkyl groups can be derived from alcohol derivatives such as nitriles or from fatty alcohols which are normally derived from naturally occurring glycerides (e.g., tallow, coconut oil, palm oil, soybean oil, lard, etc.), but which can also be derived synthetically (e.g., from fatty acids prepared by the oxidation of petroleum, or by hydrogenation of carbon monoxide by the Fischer-Tropsch process). The alkyl groups can also be derived from other sources (e.g., olefins). Examples of these detergent surfactants include sodium tallow alkyl sulfate, sodium coconut alkyl sulfate, sodium lauryl sulfate, sodium dodecyl sulfate, and the sodium salt of the sulfated condensation product between one mole of coconut alcohol and three moles of ethylene oxide.

3. Alkali metal and alkaline earth metal acyl N-methyl taurides and acyl monoglyceride sulfates wherein said acyl groups contain from about 8 to about 20 carbon atoms. Said acyl groups are normally derived from naturally occurring glycerides (e.g., tallow, coconut oil, palm oil, soybean oil, lard, etc.), but can also be derived synthetically (e.g., by oxidation of petroleum, or by hydrogenation of carbon monoxide by the Fischer-Tropsch process). Examples of these detergent surfactants include sodium N-methyl N—coconut oil acid—taurate and sodium coconut monoglyceride sulfate.

The alkyl metals preferred herein are sodium and potassium. The preferred alkaline earth metals are calcium and magnesium.

The sodium tripolyphosphate is added to the bar formulation as a cleaning aid (detergency builder), a pH buffer, and to bind water. If less than about 10% by weight of the bar is sodium tripolyphosphate (on an anhydrous basis), the result is an unacceptable loss in cleaning ability of the bar, and if more than about 25% by weight of the
bar is used, there is no improvement in cleaning advantage over 25%. About 18% by weight of the bar is preferred. If desired, sodium tripolyphosphate can be hydrated (preferably before the bar is formed) to prevent “frosting” of the bar wherein crystals of sodium tripolyphosphate hexahydrate are formed on the surface of the bar in the presence of moisture. Sodium tripolyphosphate is selected rather than pyro- or meta-phosphates because of better cleaning performance. “Frosting” does not affect the efficacy of the bar, but merely presents an unusual appearance.

The sodium bicarbonate serves several functions. It is a mild alkaline, which keeps the pH of the bar within an acceptable range for contact with the skin, i.e., from about 8 to about 11, and provides bulk for the bar without adversely affecting other properties. For example, it does not cause “frosting” of the bar and the solubility characteristics are suitable. It is also inexpensive and has cleansing properties of its own. From about 25% to about 55% and preferably about 32% by weight of the bar gives acceptable bulk to the bar without adversely diluting the effects of the other ingredients.

The trisodium orthophosphate aids in forming an acceptable bar. In the first place, the trisodium orthophosphate is a pH buffer and is a cleaning aid (detergency builder) in much the same way that sodium tripolyphosphate is. However, the trisodium orthophosphate is primarily added for the purpose of modifying the processing characteristics of the formula and the “feel” of the bar. It has been found surprisingly that trisodium orthophosphate modifies the normally sticky nature of the active, especially sodium alkyl benzene sulfonate, to permit working the formulation. From about 2% to about 15% and preferably about 5% by weight gives proper processing characteristics. With less the product is too sticky to process easily and with more it is too dry to process easily. This modification of the sticky feel is also seen on the bar itself. Without the trisodium orthophosphate the bar has a rougher surface during use whereas with the trisodium orthophosphate, the bar has a smoother, more uniform feel. A bar can be made without trisodium orthophosphate, but is more difficult to do so and some quality characteristics are sacrificed.

The total of the active, the sodium tripolyphosphate, the sodium bicarbonate, and the trisodium orthophosphate should be at least about 75% by weight of the bar, preferably from about 85% to about 92% by weight.

The amide is added optionally to the bar as a sudsy builder. It is inert and functions to about 2% by weight of the bar, but from 0% to about 5% by weight of the bar can be used. Sudsing is poorer with smaller amounts of amide and when too much amide is used, the product becomes too sticky.

The amide is also very useful in that it acts in the above proportions as a processing aid in forming the bar. The amide softens the composition during processing which aids the mechanical working necessary to form the bar. The amide also helps to weld the bar together. Although a bar can be prepared without amide, it is somewhat more difficult to do so and Sudsing qualities are considerably improved by the addition of amide.

The amides which can be used in this invention are the ammonia, monoethanol, and diethanol amides of fatty acids having an acyl chain of from about 8 to about 18 carbon atoms. These acyl chains are normally derived from naturally occurring glycerides (e.g., coconut oil, palm kernel oil, and tallow), but can be derived synthetically (e.g., by the oxidation of petroleum, or by hydrogenation of carbon monoxide by the Fischer-Tropsch process).

The silicate solids are added optionally as a detergency builder, as an aid in processing the bar when sodium alkyl benzene sulfonate is the raw material, and as a corrosion inhibitor. From about 0% to about 5%, and preferably 3%, by weight of silicate solids having an Na₂O:SiO₂ ratio of from about 1.0:0.90 to about 1.0:3.25 is used when silicate is a part of the formulation. As will hereinafter be discussed more fully, whether the silicate solids are added as liquid (aqueous) silicate or as powdered silicate depends upon the processing used in forming the bar. The silicate is added as liquid silicate to aid in processing formulations from sodium alkyl benzene sulfonate flakes. Without the water which is part of the liquid silicate, such formulations are difficult to process. The silicate is normally added to the bar ingredients in its powdered form when a preparatory dry-neutralization process is used as hereinafter described. Regardless of the processing used, the use of too little silicate can result in difficulties in processing equipment and in use of the bar. It is preferred to have silicate present in the formulation, however, in many cases the silicate will be left out since the presence of silicate in the bar tends to promote the formation of a surface with a rough feel.

Water is present in the bar in an amount from about 5% to about 25% by weight of the bar. This water in the finished bar is primarily combined water (water of hydration). Much of the water which is present is added to the formulation as a plasticizer during the processing step. This plasticization is necessary to allow the formulation containing less than 5% water. Although it is possible to substitute other plasticizers for water, it is not normally practical in view of the excellent plasticizing characteristics of water.

Many other materials can be added to the bar formulation, if desired, with excellent results, but they are not essential. For example, soil anti-redeposition agents such as sodium carboxymethylcellulose can be included in the formula for their usual purpose in amounts up to about 3% by weight of the bar. More than 3% appears to give no additional advantage over 3%.

Desirable additives are materials which are high molecular weight polymers such as the ones sold under the trade name “Polyox WSR 301.” (These “Polyox” resins are ultra high molecular weight condensates of ethylene oxide. The molecular weight of ethylene oxide. The molecular weight of ethylene oxide. The molecular weight of ethylene oxide. The molecular weight of ethylene oxide. The molecular weight of ethylene oxide.) These resins can be used in amounts up to about 1%, preferably 0.1% to 0.3%, by weight of the bar to give the bar a smoother feel. Another type of slip agent which can be used in the material sold under the name “Carbopol” and which is a carboxyvinyl polymer of extremely high molecular weight.

Other additives which can be used include absorbing material, optical brighteners, benzotriazole, etc., can be added to the formulations of this invention.

**Processes for Forming a Bar**

The bars of this invention are formed by (A) forming a homogeneous mixture of the ingredients hereinafter described and thereafter (B) extruding said mixture at a temperature of from about 100°F to about 170°F. In order to form a homogeneous bar the starting materials must be thoroughly mixed together. There are three major ways of forming the mixture.

1. The dry active can be mixed with the inorganic components and sufficient plasticizing agent such as water or sodium silicate solution is added to plasticize the mixture so that when it is mechanically worked it is formed into a homogeneous composition (for example, the mixture can be milled into homogeneous flakes on conventional mixing equipment).

2. The active in aqueous paste form can be mixed with the inorganic compounds to form a slurry which can then be dried by conventional methods (e.g., drying on a drum to flakes or spray drying to a granule).

3. The unneutralized acid form of the active can be spayed used, as a raw material, and mixed with a mixture of alkaline inorganic salts as will hereinafter be described in detail and the resulting partially neutralized mixture is mechanically worked to-
fect homogeneity and complete neutralization of the mixture.

The resulting homogeneous mixture, however formed, is then extruded on conventional equipment into bar form at a temperature in the range of from about 100° F. to about 170° F., preferably from about 120° F. to about 150° F. A temperature in these ranges can be achieved either by mechanically working the composition and thereby changing mechanical energy into thermal energy, or by a direct application of heat to the mixture. The temperature must be at least about 100° F. to effect a welding action, but it must not be too hot or there is a possibility the bar will become too soft and difficult to process.

The bar can be cut into appropriate lengths and stamped, if desired, into various shapes or stamped with various indicia.

A preferred method of forming a homogeneous mixture and consequently forming a bar of the type described, above or other types is by "dry neutralization," (3) when the active is produced in an acid form which will permit convenient processing. ("Dry neutralization," as used herein, refers to neutralization which occurs under conditions such that the amount of fluid present is insufficient to destroy the particulate nature of solid particulate alkaline material used to effect said neutralization.) Such a method is the following one for preparing a synthetic detergent (preferably alkyl aryl sulfonate) bar. The method consists essentially of: (A) spraying from about 12 parts to about 40 parts of the liquid forms of alkyl aryl sulfonic acids, alkyl sulfonic acids, alkyl sulfonic acids, and ethylene oxide ether sulfuric acids as hereinafter more fully described (preferably an alkyl aryl sulfonic acid phase which is prepared by sulfonating an alkyl aryl compound having an average molecular weight of from about 150 to about 280 with a sulfonating agent such as SO$_3$O, oleum or sulfuric acid, said alkyl aryl sulfonic acid phase having a sulfonic acid:sulfuric acid ratio in the range of from about 50:1 to about 51:1 on a weight basis and containing up to about 25% water), onto about 45 parts to about 93 parts of an agitated mixture wherein (1) at least a stoichiometric amount, sufficient to neutralize said acids, of said mixture is selected from the group consisting of solid particulate alkali metal and alkaline earth metal carbonates and bicarbonates (or sesquico-arbonates) and (2) the remainder of said mixture is a builder and/or filler selected from the group consisting of: solid particulate alkali and alkaline earth metal borates, chlorides, phosphates, metaphosphates, pyrophosphates, tripolyphosphates, and sulfates, giving a neutral to alkaline reaction (in water), the amount of water present being insufficient to destroy the particulate nature of said mixture to effect a partial neutralization of said acids; (B) optionally adding up to about 5 parts silicate solids having an Na$_2$O:SiO$_2$ ratio of from about 1.0:0.90 to about 1.0:3.25, amide, sodium carboxymethylcellulose, and other minor ingredients; (C) mechanically working the resulting partially neutralized mixture until complete neutralization is effected; (D) extruding the mechanically worked neutralized mixture at a temperature of from about 100° F. to about 170° F.; and (E) forming the extruded material into bars.

The amount of inorganic salts and especially carbonates and bicarbonates present in the solid particulate mixture should preferably be sufficient to formulate a detergent bar of the composition hereinbefore described. Since the carbonates and bicarbonates react to neutralize the acid form of the detergent surfactant and form CO$_2$ and water, there must be an excess of carbonate or bicarbonate over that amount needed to neutralize said acid form in order to have sufficient bicarbonate in the finished bar.

Preferably in the above process

(A) the alkyl aryl compound is alkyl benzene with a molecular weight of from about 208 to about 276;

(B) the agitated mixture comprises salts selected from the group consisting of sodium carbonate, sodium bicarbonate, sodium sesquisulfate, sodium sulfate, sodium tripolyphosphate, trisodium orthophosphate, disodium orthophosphate, tetrasodium pyrophosphate, disodium acid pyrophosphate, sodium trimetaphosphate, sodium carbonate, potassium carbonate, potassium sulfate, potassium tripolyphosphate, potassium chloride, calcium carbonate, calcium sulfate, calcium pyrophosphate, and calcium chlorides;

(C) the mechanical working is done by milling.

Most preferably, the agitated mixture contains from 0% to about 15% sodium carbonate and other inorganic salts in amounts such that the finished bar contains: (1) from about 10% to about 25% sodium tripolyphosphate, (2) from about 2% to about 15% trisodium phosphate, and (3) from about 25% to about 55% sodium bicarbonate.

**DRY NEUTRALIZATION**

It was discovered that the dry neutralization method of preparing the bars of this invention has wide applicability and scope. Broadly speaking, the process is one in which any liquid acid form of an anionic detergent surfactant which is capable of reacting with a basic carbonate to achieve neutralization is brought into contact with a solid particulate alkaline material capable of reacting with said acid form to produce neutralized anionic detergent surfactant and a by-product selected from the group consisting of water, a gas, and combinations of water and gas to effect partial neutralization and thereafter effecting homogeneity of the mixture by mechanically working said mixture to effect complete neutralization, the amount of fluid, including water, present being insufficient to destroy the particulate nature of said solid particulate alkaline material (before the mixture is mechanically worked).

The types of actives, the acid forms of which are adaptable to dry neutralization, include:

(1) Alkyl aryl sulfonic acids having an average molecular weight of from about 200 to about 380. The liquid acid form of these materials will normally be found as a mixture of alkyl aryl sulfonic acid with water and sulfuric acid.

(2) Alkyl sulfonic acids having from about 8 to about 20 carbon atoms, as, for example, dodecyl sulfonic acid, tallow alkyl sulfonic acid, and coconut alkyl sulfonic acid.

These acids will normally be associated with water and sulfuric acid to form a liquid.

(3) Alkyl sulfonic acid and alkyl ethylene oxide ether sulfate salts containing alkyl chains having from about 8 to about 20 carbon atoms and containing from 0 to about 9 ethylene oxide group, as, for example, tallow alkyl sulfonic acid, coconut alkyl sulfonic acid, and coconut alkyl ethylene oxide ether sulfate acid, wherein each molecule contains an average of three ethylene oxide groups.

(4) Fatty acids and acyl monoglyceride sulfuric acids, the acyl containing an average of from about 8 to about 20 carbon atoms, as, for example, coconut oil fatty acids, oleic acid, etc., and the corresponding acyl monoglyceride sulfuric acids.

(5) Combinations of the above.

The inorganic alkaline material is selected from the group consisting of solid particulate alkali and alkaline earth metal (e.g., sodium, potassium, lithium, calcium, barium and magnesium) carbonates and bicarbonates.

This material is present in at least a sufficient amount to neutralize all the acid added, including inorganic acids. Other inorganic builder and/or filler salts having a neutral to alkaline reaction action can be mixed in, if desired, with the carbonates and bicarbonates including
alkali and alkaline earth metal salts of halides, phosphates (ortho-, meta-, pyro-, tripoly-, etc.), borates, and sulfates. These materials are all in solid particulate form.

"Mechanical working" as used hereinbefore and hereinafter in relation to both product and processes refers to mechanical work done on a material by direct shearing action, as defined more fully in U.S. Patent 2,941,949, column 2, line 32 et seq.

The type of mechanical equipment which is used to effect homogeneity is any mixer, or combination of mixers capable of imparting mechanical work by a direct shearing action. Examples of such mixers include: Roll mills (soap or paint mills—this is the preferred type of mixer); kneaders; extruders; plodders, attrition mills; edge running mills; etc.

The amount of mechanical working needed to complete neutralization and effect homogeneity will depend upon the extent of contact originally present between the liquid acid form of the surfactant and the particulate inorganic salt. For example, with dodecyl benzene sulfonic acid, from 90 to 95% completeness of the neutralization can be achieved by having a finely-divided, agitated bed of the inorganic alkaline material and spraying onto said bed a finely divided spray of said dodecyl benzene sulfonic acid. The additional 5% to 10% of neutralization can be achieved with a minimum amount (e.g., two passes through a 3-roll mill) of mechanical working.

The following examples illustrate but do not limit the practice of this invention.

**Example I**

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<th>Composition</th>
<th>1</th>
<th>2</th>
<th>3</th>
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<tbody>
<tr>
<td>Sodium tetrapropylene benzene sulfonate flakes, containing NaOH, water and tetrapropylene benzene:</td>
<td>31.0</td>
<td>31.0</td>
<td>31.0</td>
</tr>
<tr>
<td>(a) 100% active</td>
<td>27.0</td>
<td>27.0</td>
<td>27.0</td>
</tr>
<tr>
<td>(b) 90% active</td>
<td>23.1</td>
<td>23.1</td>
<td>23.1</td>
</tr>
<tr>
<td>Sodium tripolyphosphate hexahydrate</td>
<td>4.7</td>
<td>4.7</td>
<td>4.7</td>
</tr>
<tr>
<td>Sodium dibenzoate</td>
<td>6.2</td>
<td>6.2</td>
<td>6.2</td>
</tr>
<tr>
<td>Sodium sesquicarbonate</td>
<td>1.7</td>
<td>1.7</td>
<td>1.7</td>
</tr>
<tr>
<td>1.000% sodium silicate aqueous solution (60% solids)</td>
<td>7.3</td>
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<tr>
<td>Sodium carbonate</td>
<td>2.0</td>
<td>2.0</td>
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<tr>
<td>Water</td>
<td>2.0</td>
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The above compositions were formulated into bars by mixing the detergent surfactant and inorganic materials together and then adding the other ingredients, particularly the silicate solution. The resulting mixture was then milled on heated rolls to form homogeneous flakes of the composition and these resulting flakes were fed to a plodder and extruded in the shape of a continuous bar at a temperature of about 135° F. The continuous bar was cut into small bars. These bars were outstandingly useful as laundry detergent bars, particularly as regards cleaning, Sudsing, wear rate and feel.

Composition 3, which does not contain trisodium orthophosphate, was more difficult to process and the bar formed therefrom had a rougher surface than bars 1 and 2, which also contain sodium tetrapropylene benzene sulfonate.

When sodium coconut acyl monoglyceride sulfite is substituted for the coconut methyl taurine, comparable results are obtained.

**Example II**

**DRIY NEUTRALIZATION**

4.8 lbs. of sodium tripolyphosphate hexahydrate was mixed with 1.0 lbs. of trisodium orthophosphate, 7.3 lbs. of sodium bicarbonate, and 1.2 lbs. of sodium carbonate and agitated. Onto this finely divided, agitated mixture was sprayed 5.8 lbs. of a fine spray of a slightly diluted dodecyl benzene sulfonic acid phase containing water, sulfuric acid, unsulfonated dodecyl benzene, and about 77% dodecyl benzene sulfonic acid. The weight ratio of sulfonic acid to sulfuric acid was 13:1. 0.4 lb. of coconut monoethanol amide and 0.2 lb. of 65% active sodium carboxymethylcellulose were added to the sulfonate, and the complete mixture was milled on a three roll soap mill to a flake thickness of about 0.007 inch with the mixture making two passes through the mill. The sulfonic acid neutralization was about 90-95% complete before milling and was complete after milling. When the resulting flakes were plodded (extruded) at a temperature of approximately 125° F. an excellent detergent bar was formed.

When dodecyl naphthalene sulfonic acid, coconut alkyl sulfonic acid, coconut alkyl sulferic acid, coconut alkyl ethylene oxide ether sulfonic acid, and a mixture of three ethylene oxide groups, or coconut fatty acids are neutralized in a manner similar to that used for the dodecyl benzene sulfonic acid in this example, comparable results are obtained. When sodium bicarbonate, potassium bicarbonate, potassium carbonate, magnesium carbonate, and calcium carbonate are substituted for the sodium carbonate in the above formulas on an equivalent basis in an amount sufficient to neutralize the acid mix, comparable results are obtained in that the acid mix is neutralized.

What is claimed is:

1. A homogeneous synthetic, mechanically worked, milker's detergent bar consisting essentially of:
   (a) from about 12% to about 30% by weight of sodium alkyl benzene sulfonate wherein the alkyl group contains from about 9 to about 15 carbon atoms; (b) from about 10% to about 25% by weight of sodium tripolyphosphate; (c) from about 25% to about 55% by weight of sodium bicarbonate; (d) from about 2% to about 15% by weight of trisodium orthophosphate; (e) from 0% to about 5% by weight of amide selected from the group consisting of ammonia amides, monoethanol amides, and diethanol amides of fatty acids having a molecular chain of from about 8 to about 18 carbon atoms; (f) from 0% to about 5% by weight of silicate solids with an Na₂O:SiO₂ ratio of from about 1.0:0.90 to about 1.0:3.25; and (g) from about 5% to about 25% by weight of water; the total of (a), (b), (c), and (d) being from about 85% to about 92% by weight, the weight percents in (a), (b), (c), (d), (e), and (f) being on an anhydrous basis.

2. The process for preparing a homogeneous detergent bar containing sodium alkyl benzene sulfonate comprising:
   (a) Spraying from about 12 parts to about 40 parts of the liquid form of an alkyl benzene sulfonate prepared by sulfonating an alkyl benzene having a molecular weight of from about 208 to about 276 with a sulfonating agent selected from the group consisting of SO₃, oleum, and sulfuric acid, said alkyl benzene sulfonic acid phase having a sulfuric acid ratio in the range of from about 50:1 to about 5:1 on a weight basis and containing up to about 25% water, onto about 45 parts to about 93 parts of an agitated mixture containing (1) at least a stoichiometric amount sufficient to neutralize said sulfonic acid, of sodium carbonate and (2) other inorganic salts in amounts such that the finished bar contains: (A) from about 10% to about 25% sodium tripolyphosphate, (B) from about 2% to about 15% trisodium orthophosphate, and (C) from about 25% to about 55% sodium bicarbonate, the amount of water present being insufficient to destroy the particulate nature of said mixture; to effect a partial neutralization of said alkyl benzene sulfonic acid phase;
   (b) Mechanically working by milling the resulting partially neutralized mixture to effect homogeneity and until complete neutralization is effected.
   (c) Extruding the mechanically worked neutralized
mixture at a temperature of from about 120° F. to about 135° F.; and

(d) Forming the extruded material into bars.

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<table>
<thead>
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<th>Patent Number</th>
<th>Date</th>
<th>Inventor(s)</th>
<th>Class</th>
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<td>6/40</td>
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JULIUS GREENWALD, Primary Examiner.

ALBERT T. MEYERS, Examiner.