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(54) Title: LOW TMF EXTRUDED SOAP BARS HAVING REDUCED CRACKING

(57) Abstract: A low TFM extruded personal washing bar comprising a continuous phase, said continuous phase being substantially free of water soluble detergent builder and comprising: a. 20% to 50% fatty acid soap, wherein the fatty acid soap comprises less than 39% of an unsaturated fatty acid soap by weight of the fatty acid soap; b. a structuring system comprising: i. from 10% to 45% by weight of the continuous phase of a polysaccharide structurant selected from the group consisting of starch, cellulose and a combination thereof, ii. from 6% to 30% by weight of continuous phase of a polyol selected from the group consisting of glycerol, sorbitol and their mixtures, and iii. 0 to 15% by weight of continuous phase of a water insoluble particulate material; c. 0.5% to less than 3% of an anticracking agent selected from the group consisting of carboxymethylcellulose, polyacrylate polymers and mixtures thereof; d. from 10% to 20% water; wherein the bar has a Cracking Index of 1 or less; and wherein the continuous phase is an extrudable mass having a penetrometer hardness of 3 to 8 Kg and a yield stress of 350 to 2000 kPa measured at a temperature of 40°C.

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LOW TMF EXTRUDED SOAP BARS HAVING REDUCED CRACKING

The invention relates to low TFM personal washing bars that are made by high speed extrusion and stamping and are suitable for the mass market. The personal washing bars exhibit a reduced tendency to crack during use.

Personal washing bars such as soap bars have played an important role in hygiene and skin care. Their routine use has been critical in reducing the spread of communicable diseases. Manufacturers have continuously sought ways to improve the in-use properties and skin compatibility of personal washing bars and to increase their affordability to consumers around the world.

Fatty acid soaps derived from triglycerides are a predominant surfactant used in personal wash bars. With the growing demands for triglycerides as foods and recently as alternative fuels, their cost is increasing. Consequently, manufacturers have sought ways to use fatty acid soaps more efficiently in soap bars. When the predominant surfactant in the personal washing bar is fatty acid soap, a reduction in surfactant is commonly expressed as a reduction in "Total Fatty Matter" or TFM. The term TFM is used to denote the percentage by weight of fatty acid and triglyceride residues present in soaps without taking into account the accompanying cations. The measurement of TFM is well known in the art.

One strategy that has been used to reduce the soap content in bars is to replace part of the fatty acid soap by inorganic fillers and/or higher levels of water. However, the use of high levels of inorganic fillers and/or high water levels leads to several negative properties which includes a significant shrinkage of the bar during storage by evaporation of water and to a smaller bar volume because of the higher amount of inorganic materials.

Another approach that has been used to lower the surfactant content of bars is the use of coagels formed in a melt-cast process. Here a molten surfactant solution is poured into a mold and cooled. The surfactant solution in combination with gelling and/or structuring agents forms a highly extended three-dimensional network. Although melt-cast technology yields bars that have a lower surfactant content, their production is less efficient than extruded soaps. Furthermore, melt-cast bars have substantial levels of water and solvents, are subject to drying out, and their rate of wear is much higher than milled toilet soaps. Consequently, melt-cast bars are less economical in use than milled soaps.

Examples of approaches based on the above concepts include: WO 01/42418 to Chokappa et al disclosing detergent bar with amorphous alumina; WO 2006/094586 disclosing low TFM detergent bar including inorganic particulates and alumino-silicate; US 6,440,908 disclosing high moisture bars including borates; WO98/18896 disclosing high moisture laundry bars including structured soap and starch; US 2007/0021314 and US 2007/0155639 disclosing cleansing bars including a component selected from the group consisting of carbohydrate structurant, humectants, free fatty acid, synthetic surfactants, and mixtures thereof; WO 2007/146027 disclosing cleansing bar compositions including carbohydrate stucturants and cationic polymers; U.S. 6838420B2 disclosing translucent or transparent composition including humectants, structurants, and gallants; US 4,808,322 disclosing non-foaming skin cleansing-conditioning bars consisting essentially of specific anionic surfactant materials; specific water-insoluble emollients, optional starch-derived filler and water; WO 99/14307 disclosing high moisture compositions including a soap phase structured with a cellulose material.

Previous work described in Patent Applications GB 806340.6 and GB901953.0 identified lower TFM extrudable soap bar compositions that included starch, specific polyols and optionally water insoluble particles that did not require high

levels of water and inorganic fillers. Although these lower TFM compositions have generally good lather and in-use properties, they have been found to be more susceptible to cracking and fissuring during use than traditional soap bars, which typically contain 75-85% soap, especially when the bars are allowed to repeatedly become water logged followed by drying out.

Cracking of toilet soaps has been considered in EP0311343A2, and GB2114145A1.

Further extensive experimentation has revealed that the cracking tendency of low TFM bars can be dramatically improved by incorporating certain anti-cracking agents in limited amounts into the continuous phase of the bar, provided that the bars are substantially free of water-soluble detergent builders especially those based on water soluble salts of tri- and multivalent anions of strong acids such as sodium polyphosphate and tetrasodium pyrophosphate. This technology is the subject of the present invention.

The personal washing bars of the invention are extruded and preferably stamped bars suitable for mass market applications. One embodiment of the invention is a personal washing bar which includes a continuous phase which is substantially free of a water soluble detergent builder, the continuous phase comprising:

a. 20% to 50%, preferably 25% to 40%, more preferably 30% to 37% fatty acid soap, wherein the fatty acid soap comprises less than 39% of an unsaturated fatty acid soap by weight of the fatty acid soap;
b. a structuring system comprising:
    i. from 10% to 45%, preferably 20% to 40% by weight of the continuous phase of a polysaccharide structurant selected from the group consisting of starch, cellulose and a combination thereof,

ii from 6.0% to 30%, preferably 10% to 20%, by weight of continuous phase of a polyol selected from the group consisting of glycerol, sorbitol and their mixtures, and

iii. 0 to 15%, preferably 4% to 10% by weight of continuous phase of a water insoluble particulate material;

c. 0.5% to less than 3%, preferably 0.5% to less than 2.5%, of an anticracking agent selected from the group consisting of carboxymethyl cellulose, polyacrylates and mixtures thereof;

d. from 10% to 20%, preferably 15% to 18% water;

wherein the continuous phase is an extrudable mass having a penetrometer hardness of 3 to 8 Kg and a yield stress of 350 to 2000 kPa measured at a temperature of 40°C. Conveniently the bar has a Cracking Index of 1 or less.

In another embodiment, the continuous phase composition includes a synthetic surfactant at a level of up to about 10% by weight of the bar, preferably between 2% and 8% by weight.

In yet another embodiment, the continuous phase includes a slip modifier which greatly improves the feel of the wet bar when it is rubbed on the skin, especially when the polysaccharide and/or insoluble particles are present in the bar at levels approaching the upper limits of their useful concentration ranges.

These and other embodiments are described more fully below in the following written description and various embodiments that are illustrated in the examples.

As used herein % or wt % refers to percent by weight of an ingredient as compared to the total weight of the composition or component that is being discussed (generally the composition of the continuous phase or the composition of the fatty acid soap).

Except in the operating and comparative examples, or where otherwise explicitly indicated, all numbers in this description indicating amounts of material or conditions of reaction, physical properties of materials and/or use are to be understood as modified by the word "about." Unless otherwise specified the term composition will refer to the composition of the continuous phase of the bar.

For the avoidance of doubt the word "comprising" is intended to mean "including" but not necessarily "consisting of" or "composed of." In other words, the listed steps, options, or alternatives need not be exhaustive.

The present invention relates to extruded personal washing bars in which the continuous phase of the bar comprises specific levels of fatty acid soaps, hereinafter designated simply as "soap", from about 30% to about 60% of a structuring system; specific anticracking agent which have been found to reduce cracking and various optional ingredients. The compositions used to prepare the continuous phase of bars of the invention and the methods used to manufacture and evaluate the compositions and bars made from the compositions are described below.

The term "continuous phase of the bar" is used herein in a macroscopic sense to describe the predominant soft solid phase into which various macroscopic solid domains, the "dispersed phase" may be optionally dispersed or distributed. The continuous phase is generally not a single phase in the microscopic sense but rather is a substantially uniform mixture (i.e., dispersion) of microscopic soap crystals and liquid crystalline or gel and liquid materials, components of the structuring system (e.g., starch, polyol, insoluble particulate material), and various optional ingredients. The continuous phase generally comprises from 65% to 100% by weight of the personal washing bar. In the majority of applications, the continuous phase comprises 90% to 100% of the personal washing bar.

The optional dispersed phase can be in the form of stripes or variegations, bits (e.g., pieces or chunks of solid), plate-like inclusions, veins and the like and mixtures thereof. The dispersed phase generally has a different overall composition from the continuous phase, but may only differ in the level or type of colorant.

Examples illustrating multiphase personal washing bars having a dispersed macroscopic phase are cited below in the section on Dispersed Phases.

The bar compositions of the invention are capable of being manufactured at high production rates by processes that generally involve the extrusion forming of ingots or billets, and stamping or molding of these billets into individual tablets, cakes, or bars.

By the term “capable of high manufacturing rates” is meant that the mass formed from the continuous phase composition and any dispersed phase is capable of being extruded at a rate in excess of 9 kg per minute, preferably at or exceeding 27 kg per minute and ideally at or exceeding 36 kg per minute.

Personal washing bars produced from compositions according to the invention in addition to being capable of being processed at high production rates also possess a range of desirable physical properties that make them highly suitable for every day use by mass market consumers.

Test methods to assess various physical properties of the composition that provide objective criteria for bars manufacture and in-use properties are described below in the TEST METHODOLOGY section.

The continuous phase compositions according to the present invention are extrudable masses which are substantially free of water-soluble detergent builders

and include fatty acid soap, a structuring system, an anti-cracking agent, water and various optional ingredients.

By the term “substantially free” is meant a level of water-soluble detergent builder that is less than 0.8%, preferably less than 0.5%, still more preferably less than 0.3% and in some cases less than 0.1% based on the total weight of the continuous phase.

For the purposes of the present invention detergent builders (or simply “builders”) are defined as water-soluble salts having di-, tri- and multi-valent anions having a degree of polymerization generally less than 50, generally less than 25, which interact very strongly with di- and multi-valent cations such as calcium and magnesium. Detergent builders are generally salts of strong acids.

Detergent builders have surprisingly been found to negate the effects of the specific agents which we have found to reduce cracking in low TFM extruded bars.

Particularly deleterious builders are the water-soluble alkali-metal salts of phosphate, especially pyrophosphates, orthophosphates, tripolyphosphates, higher polyphosphates, and mixtures thereof. Examples include water-soluble alkali-metal salt of tripolyphosphate, and a mixture of tripolyphosphate and pyrophosphate, e.g., sodium tripolyphosphates (STPP), tetra sodium pyrophosphates (TSPP).

Examples of non-phosphate, inorganic detergent builders include water-soluble inorganic carbonates such as sodium and potassium carbonates and silicates.

Another type of builder is a strong chelating salts which are able to sequester and chelate magnesium and calcium ions and heavy metal cations such as iron,

manganese, zinc and aluminum. These include phosphonate chelants such as diethylenetriamine penta(methylene phosphonic acid), ethylene diamine tetra(methylene phosphonic acid), salts and complexes thereof, and acetate chelants such as diethylenetriamine penta(acetic acid), ethylene diamine tetra(acetic acid) (EDTA) and salts thereof. Low levels of certain chelant salts such as EDTA are widely used as a preservative in soap bars at very levels. This is acceptable provided the level remains below about 0.3%.

The discontinuous phase of the bar may contain some water soluble detergent builder so long as the builder salt does not migrate to the continuous phase, i.e., so long as the continuous phase remains substantially free of detergent builder. However, we have found it preferable that the entire bar be substantially free of builder, especially phosphate builder with the meaning of “substantially” as defined above. That is the level of water soluble detergent builder in the entire personal washing bar (i.e. continuous and discontinuous phase) is less than 0.8%, preferably less than 0.5%, still more preferably less than 0.3% and in some cases less than 0.1% based on the total weight of the bar.

The fatty acid soaps, optional surfactants and in fact all the components of the bars should be suitable for routine contact with human skin, and preferably yield bars that are high lathering.

The preferred type of surfactant is fatty acid soap. The term "soap" is used herein to mean an alkali metal or alkanol ammonium salts of aliphatic, alkane-, or alkene monocarboxylic acids usually derived from natural triglycerides. Sodium, potassium, magnesium, mono-, di- and tri-ethanol ammonium cations, or combinations thereof, are the most suitable for purposes of this invention. In general, sodium soaps are used in the compositions of the invention, but from about 1% to about 25% of the soap may be potassium, magnesium or triethanolamine soaps. The soaps useful herein are the well known alkali metal

salts of natural or synthetic aliphatic (alkanoic or alkenoic) acids having about 8 to about 22 carbon atoms, preferably about 10 to about 18 carbon atoms. They may be described as alkali metal carboxylates of saturated or unsaturated hydrocarbons having about 8 to about 22 carbon atoms.

Soaps having the fatty acid distribution of coconut oil and palm kernel oil may provide the lower end of the broad molecular weight range. Those soaps having the fatty acid distribution of peanut or rapeseed oil, or their hydrogenated derivatives, may provide the upper end of the broad molecular weight range.

It is preferred to use soaps having the fatty acid distribution of coconut oil or tallow, or mixtures thereof, since these are among the more readily available triglyceride fats. The proportion of fatty acids having at least 12 carbon atoms in coconut oil soap is about 85%. This proportion will be greater when mixtures of coconut oil and fats such as tallow, palm oil, or non-tropical nut oils or fats are used, wherein the principle chain lengths are C_{16} and higher. Preferred soap for use in the compositions of this invention has at least about 85% fatty acids having about 12 to 18 carbon atoms.

The preferred soaps for use in the present invention should include at least about 30% saturated soaps, i.e., soaps derived from saturated fatty acids, preferably at least about 40%, more preferably about 50%, saturated soaps by weight of the fatty acid soap.

Soaps can be classified into three broad categories which differ in the chainlength of the hydrocarbon chain, i.e., the chainlength of the fatty acid, and whether the fatty acid is saturated or unsaturated. For purposes of the present invention these classifications are:

"Laurics" soaps which encompass soaps which are derived predominantly from C_{12} to C_{14} saturated fatty acid, i.e. lauric and myristic acid, but can contain minor amounts of soaps derived from shorter chain fatty acids, e.g., C_{10}. Laurics soaps are generally derived in practice from the hydrolysis of nut oils such as coconut oil and palm kernel oil

"Stearics" soaps which encompass soaps which are derived predominantly from C_{16} to C_{18} saturated fatty acid, i.e. palmitic and stearic acid but can contain minor level of saturated soaps derived from longer chain fatty acids, e.g., C_{20}. Stearics soaps are generally derived in practice from triglyceride oils such as tallow, palm oil and palm stearin.

"Oleics" soaps which encompass soaps which are derived from unsaturated fatty acids including predominantly oleic acid ($C_{18:1}$), linoeleic acid(($C_{18:2}$), myristoleic acid ($C_{14:1}$) and palmitoleic acid ($C_{16:1}$) as well as minor amounts of longer and shorter chain unsaturated and polyunsaturated fatty acids. Oleics soaps are generally derived in practice from the hydrolysis of various triglyceride oils and fats such as tallow, palm oil, sunflower seed oil and soybean oil.

Coconut oil employed for the soap may be substituted in whole or in part by other "high-laurics" or "laurics rich" oils, that is, oils or fats wherein at least 45% of the total fatty acids are composed of lauric acid, myristic acid and mixtures thereof. These oils are generally exemplified by the tropical nut oils of the coconut oil class. For instance, they include: palm kernel oil, babassu oil, ouricuri oil, tucum oil, cohune nut oil, murumuru oil, jaboty kernel oil, khakan kernel oil, dika nut oil, and ucuhuba butter.

When a solid mass which includes a mixture of laurics, stearics and oleics soaps is heated, the laurics and oleics soaps which are more water soluble and have lower melting points than stearics soaps, combine with water and other

components present in the composition to form a more or less fluid liquid crystal phase depending on water content and temperature. This transformation of laurics and oleics soaps from a solid to a liquid crystal phase provides plasticity to the mass which allows it to be mixed and worked under shear, i.e. the mass is thermoplastic.

It has been found critical when using a structuring system comprising a mixture of starch and/or cellulose, polyol and optional insoluble particulate material, to control the total level of unsaturated soaps in the continuous phase to achieve bars that can be extruded and stamped at high production rates.

The level of unsaturated soaps in the continuous phase expressed as the weight % of the total soap content of the continuous phase should be less than 40%, preferably less than 39% and preferably between 32% and 37% of the continuous phase.

A preferred soap is a mixture of about 10% to about 40% derived from coconut oil, palm kernel oil or other laurics rich oils and about 90% to about 60% tallow, palm oil, palm stearin or other stearics rich oils or a combination thereof.

Soaps may be made by the classic kettle boiling process or modern continuous soap manufacturing processes wherein natural fats and oils such as tallow, palm oil or coconut oil or their equivalents are saponified with an alkali metal hydroxide using procedures well known to those skilled in the art. Two broad processes are of particular commercial importance. The SAGE process where triglycerides are saponified with a base, e.g., sodium hydroxide, and the reaction products extensively treated and the glycerin component extracted and recovered. The second process is the SWING process, where the saponification product is directly used with less exhaustive treatment and the glycerin from the triglyceride is not separated but rather included in the finished soap noodles and/or bars.

Alternatively, the soaps may be made by neutralizing fatty acids (e.g., distilled fatty acids), such as lauric (C_{12}), myristic (C_{14}), palmitic (C_{16}), stearic (C_{18}) and oleic acid ($C_{18:1}$) acids and their mixtures with an alkali metal hydroxide or carbonate (provided the level of residual sodium carbonate is sufficiently low – see above).

The level of fatty acid soap in the continuous phase of the bar (generally a mixture of different chainlengths and/or isomers) can range from about 20% to less than about 52%, generally less than 50%, preferably 20% to about 45%, preferably 25% to 40%, preferably about 25% to 37% and preferably 30 to 37% based on the total weight of the continuous phase composition.

Surfactants other than soap (commonly known as “synthetic surfactants” or “syndets”) can optionally be included in the bar at levels generally up to and including about 10%, preferably at levels between about 2% to about 7% by weight of the bar. Examples of suitable syndets are described below.

The structuring system includes one or more polysaccharide structurants selected from the group consisting of starch, cellulose and their mixtures; one or more polyols; and optionally, a water insoluble particulate material.

The total level of the structuring system used in the composition is generally about 30% to 70% by weight of continuous phase, preferably 30% to 60% and most preferably about 35% to about 55% based on the total weight the continuous phase. By total level of the structuring system is meant the sum of the weights of the starch/cellulose, polyol, and optional insoluble particulate material in the continuous phase.

Suitable starch materials include natural starch (from corn, wheat, rice, potato, tapioca and the like), pregelatinzed starch, physically and chemically modified

starch and mixtures thereof. By the term natural starch, also known as raw or native starch, is meant starch which has not been subjected to further chemical or physical modification apart from steps associated with separation and milling.

A preferred starch is natural or native starch (commonly also known as raw starch) from maize (corn), cassava, wheat, potato, rice and other natural sources. Raw starch with different ratio of amylose and amylopectin include: maize (25% amylose); waxy maize (0%); high amylose maize (70%); potato (23%); rice (16%); sago (27%); cassava (18%); wheat (30%) and others. The raw starch can be used directly or modified during the process of making the bar composition such that the starch becomes either partially or fully gelatinized.

Another suitable starch is pre-gelatinized which is starch that has been gelatinized before it is added as an ingredient in the present bar compositions. Various forms are available that will gel at different temperatures, e.g., cold water dispersible starch. One suitable commercial pre-gelatinized starch is supplied by National Starch Co. (Brazil) under the trade name FARMAL CS 3400 but other commercially available materials having similar characteristics are suitable.

Suitable cellulose materials include microcrystalline cellulose, hydroxyalkyl alkylcellulose ether and mixture thereof.

A preferred cellulose material is microcrystalline cellulose (a highly crystalline particulate cellulose made primarily of crystalline aggregates) which is obtained by removing amorphous fibrous cellulose regions of a purified cellulose source material by hydrolytic degradation. This is typically done with a strong mineral acid (e.g., hydrogen chloride). The acid hydrolysis process produces microcrystalline cellulose of predominantly coarse particulate aggregates, typically of mean size range 10 to 40 microns. One suitable commercial microcrystalline cellulose is supplied by FMC Biopolymer (Brazil) under the trade name AVICEL

GP 1030 but other commercially available materials having similar characteristics are suitable.

A preferred polysaccharide structurant is starch, most preferably a natural starch (raw starch), a pre-gelatinized starch, a chemically modified starch or mixtures thereof. Raw starch is preferred.

The amount of the polysaccharide (e.g., starch and/or cellulose) component in the continuous phase can range from about 10% to about 40%, preferably 20% to 40%, preferably 25% to 40% and preferably 30% to 40% by weight of the composition.

A second critical component of the structuring system is a polyol or mixture of polyols. Polyol is a term used herein to designate a compound having multiple hydroxyl groups (at least two, preferably at least three) which is highly water soluble, preferably freely soluble, in water.

Many types of polyols are available including: relatively low molecular weight short chain polyhydroxy compounds such as glycerol and propylene glycol; sugars such as sorbitol, manitol, sucrose and glucose; modified carbohydrates such as hydrolyzed starch, dextrin and maltodextrin, and polymeric synthetic polyols such as polyalkylene glycols, for example polyoxyethylene glycol (PEG) and polyoxypropylene glycol (PPG).

Preferred polyols are relatively low molecular weight compound which are either liquid or readily form stable highly concentrated aqueous solutions, e.g., greater that 50% and preferably 70% or greater by weight in water. These include low molecular weight polyols and sugars.

Especially preferred polyols are glycerol, sorbitol and their mixtures.

The level of polyol is critical in forming a thermoplastic mass whose material properties are suitable for both high speed manufacture (27 – 36 Kg/min) and for use as a personal washing bar. It has been found that when the polyol level is too low, the mass is not sufficiently plastic at the extrusion temperature, typically 40° C to 45° C. Conversely, when the polyol level is too high, the mass becomes too soft to be efficiently formed into bars by extrusion at normal process temperatures.

The level of polyol should be between 6% and 30%, preferably 8 to 20% and preferably about 8% to about 15% by weight of composition.

The weight ratio of starch and/or cellulose to polyol in the continuous phase is generally in the range of 2:1 to 6:1, preferably 3:1 to 5:1, e.g., about 4:1.

The structuring system may optionally include one or a combination of insoluble particulate materials. By insoluble particulate material is meant materials that are present in the continuous phase as finely divided solid particles and are suitable for personal washing applications. The particulate material can be inorganic or organic or a combination as long as it is insoluble in water. The insoluble particulate material should not be perceived as scratchy or granular, and thus should have a particle size less than 300 microns, more preferably less than 100 microns and most preferably less than 50 microns.

Preferred inorganic particulate material include talc and calcium carbonate. Talc is a magnesium silicate mineral material, with a sheet silicate structure represented by the chemical formula $Mg_3Si_4(O)_{10}(OH)_2$, and may be available in the hydrated form. Talc has a plate-like morphology, and is substantially oleophilic/ hydrophobic.

Calcium carbonate or chalk exists in three crystal forms: calcite, aragonite and vaterite. The natural morphology of calcite is rhombohedral or cuboidal, acicular or dendritic for aragonite and spheroidal for vaterite.

Commercially, calcium carbonate or chalk (precipitated calcium carbonate) is produced by a carbonation method in which carbon dioxide gas is bubbled through an aqueous suspension of calcium hydroxide. In this process the crystal type of calcium carbonate is calcite or a mixture of calcite and aragonite.

Examples of other optional insoluble inorganic particulate materials include alumino silicates, aluminates, silicates, phosphates, insoluble sulfates, borates and clays (e.g., kaolin, china clay) and their combinations.

Organic particulate materials include: insoluble polysaccharides such as highly cross-linked or insolubilized starch (e.g., by reaction with a hydrophobe such as octyl succinate); synthetic or natural polymers such as various polymer lattices and suspension polymers and mixtures thereof.

The structuring system can comprise up to and including about 15% insoluble particulate material, preferably 4% to about 10%, based on the total weight of the composition.

It has been found that extruded personal washing bars having a continuous phase containing 50% or less of a fatty acid soap are much more susceptible to the formation of cracks and fissures during use than conventional soap bars.

“Cracking” (or “fissuring”) of soap bars is well known in the extruded soap art. The process is thought to arise from delamination or separation of the soap mass especially at the ends of the bar (“end cracking”) and along compression lines formed at the junction of extrusion flow elements known as soap candles. This

type of cracking is perceived by consumer as a distinct negative often associated with low quality soaps. The degree of cracking can be severe especially when the bar is allowed to become water logged by sitting in a poorly drained soap dish and then dried out for long periods of time. A test to assess the potential of an extruded to crack in use in described below in the TEST METHODOLOGY section.

We have found that certain water soluble polymers when incorporated into the continuous phase of a substantially builder free low TFM extruded soap bar are quite effective in reducing or eliminating cracking and fissuring. The polymers which are the most effective anti-cracking agents are carboxymethylcellulose, acrylate polymers and their mixtures.

The sodium carboxymethylcellulose gums employed in this invention are water soluble, anionic, long chain polymers, derived from cellulose generally by the reaction of cellulose with alkali and chloroacetic acid. Properties vary with the average number of carboxymethyl groups that are substituted per anhydroglucose unit in each cellulose molecule. This property is generally referred to as "the degree of substitution". The maximum degree of substitution, DS, possible designated as "3.0" since there are just three hydroxy groups capable of reaction in each anhydroglucose unit. For the practice of this invention, it has been found that carboxymethylcellulose gums having a degree of substitution of from 0.4 to 1.3 are suitable. The viscosity of 1 percent solution of the gum, measured at 25^{o}C, should be in the range of from about 15 to 10,000 preferably 1,000 to 5,000 centipoises.

As examples of commercially available sodium carboxymethylcellulose gums suitable for use in this invention there may be mentioned those sold by Danisco, as types CG BEV350, CG BEV 050, BEV 150, CG MAS 120, SCMC 9H. Type 9H is preferred for use in this invention.

Among the acrylate polymers suitable for the purposes of the present invention include Acrylate crosspolymers (copolymers of acrylic acid, methacrylic acid or one of its simple esters, crosslinked with glycol dimethacrylate), acrylate copolymers (copolymers of two or more monomers consisting of acrylic acid, methacrylic acid or one of their simple esters), carbomers and acrylates/C_{x-y} Alkyl acrylate crosspolymers (copolymers of C_{x-y} alkyl acrylate and one or more monomers of acrylic acid, methacrylic acid or one of their simple esters crosslinked with an alkyl ether of sucrose or an allyl ether of pentaerythritol), where x and y vary from 2 to 30, more preferably from 10 to 30 having viscosity (25°C, 1% solution) from 4,000 to 100,000 cP. Examples include Carbopol ETD 2020, Carbopol 940, Carbopol 934, Carbopol Ultrez 20, Carbopol Ultrez 10, Carbopol 934, Carbopol 941, Carbopol 981.

Neutralizing agents suitable for use in neutralizing the carboxylic groups of the polymer agents discussed above include sodium hydroxide, potassium hydroxide, ammonium hydroxide, monoethanolamine, diethanolamine and triethanolamine.

A preferred anti-cracking agent is sodium carboxymethylcellulose having viscosity (25°C, 1%) of 15 to 10,000 cP and a degree of substitution of 0.5 to 1.3.

The anti-cracking agent is present in the continuous phase at a level of at least 0.3%, preferably at least 0.5% by weight of continuous phase but should be less than about 3%. When present at a level below 0.3%, the agents are not sufficiently effective to reduce cracking of the low TFM bars. However, when the agents are incorporated in the continuous phase at levels of 3% or higher, the polymeric anti-cracking agents have been found to have a negative impact on the volume of lather generated and can also contribute to excessive swelling of the bar when it is contacted with water and/or make the bar slimy during use.

Generally, the optimum level required to achieve a cracking score on the Accelerated Cracking Test (see TEST METHODOLGY section) of 1 or less is between about 0.5 wt% to about 2.5 wt% based on the total weight of the continuous phase.

The optimum level of anti-cracking agent required to substantially reduce or eliminate cracking has been surprisingly found to be inversely related to the total fatty acid soap content of the continuous phase and to depend on the ratio of the unsaturated fatty acid soaps relative to the water content in the bar. This optimal level is approximately given by the following equation:

$$\% \text{ Anti-cracking agent} = (4.3\ e\text{-}1.68R_{us/w}) \pm 15\% \qquad (1)$$

where $R_{us/w}$ is the ratio of the percent total unsaturated soaps in the continuous phase to the percent water in the continuous phase.

For example, using a fatty acid soap having about 36% unsaturation, the optimum level of anti-cracking agent in continuous phases having different levels of soap and containing about 18% water would be:

Wt% Soap in Continuous Phase	Rus/w	Approximate Optimal Level of Anti-Cracking Agent
20	0.4	2.19
30	0.7	1.32
45	0.9	0.94

Surprising it has been found that soap bars which have a fatty acid soap content greater than about 51-52%, e.g., 54-55% and above are far less susceptible to cracking and the influence of the anti-cracking agents identified herein are much less pronounced or negligible than for the low TFM bars which are the subject of the current invention.

The bar compositions of the invention do not comprise an especially high level of water compared to typical extruded and stamped soap bars which typically can range from about 13 to about 18% water when freshly made, i.e., after extrusion and stamping. In fact, it is preferable that the water content of the continuous phase in the freshly made bar should be 10% to about 20% by weight, preferably between 14% and 18% and more preferably 15% to 18% based on the total weight of the continuous phase. Thus, in preferred embodiments, the water level of the freshly made bars of the invention is much lower than the water content of freshly made melt and pour or melt-cast bars, i.e., the nominal water content based on the formulation as made in a factory, which typically exceeds 25% by weight in melt-cast compositions.

It is stressed that the preferred water content levels quoted above refers to freshly made bars, which is designated as the “initial water level” or “initial water content” is also known as the “nominal water content” or “nominal water level” of the composition. As is well known, soap bars are subject to drying out during storage, i.e., water evaporates from the bar when the relative humidity is lower than the partial vapor pressure of water in equilibrium with the bar composition and depends on the rate of diffusion of water from the bar. Hence, depending upon how the bar is stored (type of wrapper, temperature, humidity, air circulation, etc) the actual water content of the bar at the moment of sampling can differ from the nominal water content of the bar immediately after manufacture.

In terms of possible optional ingredients, various additional electrolytes (in addition to the fatty acid soap and other charged surfactants which are electrolyte), especially those having alkali metal cations can be present in the bar. These electrolytes are present either as a result of saponification and neutralization of the fatty acids, e.g., NaCl generated from saponification with sodium hydroxide and neutralization with hydrochloric acid, or as added salts such as sodium or potassium sulfate which may be used to control hardness. Various

electrolytes can be used in modest amounts as long as they are not strong detergent builders or otherwise interfere with the efficacy of the anti-cracking agents.

The level of electrolytes should be less than 2.0%, preferably less than 1.5%, preferably up to about 1.0%, preferably up to and including 0.8%. In some circumstances a level of salt from about 0.3% to about 0.8% is useful. However, in other cases, especially as the total soap content of the continuous phase drops below about 35%, it is may be preferably to limit the total electrolyte to less than 0.8%, e.g., 0.3% to 0.7% or 0.1% to 0.5%.

It should be noted that salts are not used in the present invention to lower water activity so as to accommodate very high levels of water in the bar which is characteristic of some low TFM bars described in the prior art, i.e. the use of electrolytes to prevent or slow the drying out of the bar. In fact, the bars of the current invention have water levels that are not especially high (up to about 20%) compared with normal commercial soap bars which can range from about 13 to about 15-18% nominal water content. In fact, levels of salts in the range of 2.5 to 8% typical of the high water content bars of the prior art, would be detrimental to the bars described herein.

A useful optional ingredient is free fatty acid and/or triglycerides which is useful for improving lather, as well as modifying the rheology at low levels incorporated in composition to increase plasticity.

Potentially suitable fatty acids are C_8-C_{22} fatty acids. Preferred fatty acids are C_{12}-C_{18}, preferably predominantly saturated, straight-chain fatty acids. However, some unsaturated fatty acids can also be employed. Of course the free fatty acids can be mixtures of shorter chainlength (e.g., C_{10}-C_{14}) and longer chainlength (e.g.,

C_{16}-C_{18}) chain fatty acids. For example, one useful fatty acid is fatty acid derived from high-laurics triglycerides such as coconut oil, palm kernel oil, and babasu oil.

The fatty acid can be incorporated directly or they can be generated in-situ by the addition of a protic acid to the soap during processing. Examples of suitable protic acids include: mineral acids such as hydrochloric acid and sulfuric acid, adipic acid, citric acid, glycolic acid, acetic acid, formic acid, fumaric acid, lactic acid, malic acid, maleic acid, succinic acid, tartaric acid and polyacrylic acid. However, care should be taken that the residual electrolyte present in the bar does not substantially reduce the effectiveness of the anticracking agent.

The level of fatty acid having a chainlength of 14 carbon atoms and below should generally not exceed 5.0%, preferably not exceed about 1% and most preferably be 0.8% or less based on the total weight of the continuous phase.

The bar compositions can optionally include non-soap synthetic type surfactants (detergents) – so called “syndets”. Syndets can include anionic surfactants, nonionic surfactants, amphoteric or zwitterionic surfactants and cationic surfactants.

The level of synthetic surfactant present in the bar is generally not greater than about 10% in the continuous phase although inclusion of higher levels in the bar may be advantageous for some applications. Some embodiment of the invention include syndets at a level of about 2% to 10%, preferably about 4% to about 10%.

Suitable syndets include anionic surfactants (non-soap), amphoteric surfactants and nonionic surfactants.

The toilet bar compositions of the present invention may contain one or more non-soap anionic syndet surfactants (simply designated “anionic syndets”) at a level

up to about 20%, preferably 0 to 10% and more preferably 2% to 5% based on the total weight of the continuous phase. Suitable anionic syndets may be, for example, an aliphatic sulfonate, such as a primary alkane (e.g., C_8-C_{22}) sulfonate, primary alkane (e.g., C_8-C_{22}) disulfonate, C_8-C_{22} alkene sulfonate, C_8-C_{22} hydroxyalkane sulfonate or alkyl glyceryl ether sulfonate (AGS); or an aromatic sulfonate such as alkyl benzene sulfonate. Alpha olefin sulfonates are another suitable anionic surfactant.

The anionic syndet may also be an alkyl sulfate (e.g., C_{12}-C_{18} alkyl sulfate), especially a primary alcohol sulfate or an alkyl ether sulfate (including alkyl glyceryl ether sulfates).

The anionic syndet can also be a sulfonated fatty acid such as alpha sulfonated tallow fatty acid, a sulfonated fatty acid ester such as alpha sulfonated methyl tallowate or mixtures thereof.

The anionic syndet may also be alkyl sulfosuccinates (including mono- and dialkyl, e.g., C_6-C_{22} sulfosuccinates); alkyl and acyl taurates, alkyl and acyl sarcosinates, sulfoacetates, C_8-C_{22} alkyl phosphates and phosphates, alkyl phosphate esters and alkoxyl alkyl phosphate esters, acyl lactates or lactylates, C_8-C_{22} monoalkyl succinates and maleates, sulphoacetates, and acyl isethionates.

Another class of anionic syndets is C_8 to C_{20} alkyl ethoxy (1-20 EO) carboxylates.

Another suitable anionic syndet is C_8-C_{18} acyl isethionates. These esters are prepared by reaction between alkali metal isethionate with mixed aliphatic fatty acids having from 6 to 18 carbon atoms and an iodine value of less than 20. At least 75% of the mixed fatty acids have from 12 to 18 carbon atoms and up to 25% have from 6 to 10 carbon atoms. The acyl isethionate may also be alkoxylated isethionates

Frequently, the anionic syndet will comprise a majority of the synthetic surfactants used in the composition.

Amphoteric surfactants which may be used in this invention include at least one acid group. This may be a carboxylic or a sulphonic acid group. They include quaternary nitrogen and therefore are quaternary amido acids. They should generally include an alkyl or alkenyl group of 7 to 18 carbon atoms. Suitable amphoteric surfactants include amphoacetates, alkyl and alkyl amido betaines, and alkyl and alkyl amido sulphobetaines.

Amphoacetates and diamphoacetates are also intended to be covered in possible zwitterionic and/or amphoteric compounds which may be used.

Suitable nonionic surfactants include the reaction products of compounds having a hydrophobic group and a reactive hydrogen atom, for example aliphatic alcohols or fatty acids, with alkylene oxides, especially ethylene oxide either alone or with propylene oxide. Examples include the condensation products of aliphatic (C_8-C_{18}) primary or secondary linear or branched alcohols with ethylene oxide, and products made by condensation of ethylene oxide with the reaction products of propylene oxide and ethylenediamine. Other so-called nonionic detergent compounds include long chain tertiary amine oxides, long chain tertiary phosphine oxides and dialkyl sulphoxides.

The nonionic may also be a carbohydrate or sugar based, ethers, esters or amides, such as alkyl (poly)saccharides and alkyl (poly)saccharide amides.

Examples of cationic detergents are the quaternary ammonium compounds such as alkyldimethylammonium halides.

Other surfactants which may be used are described in U.S. Pat. No. 3, 723,325 to Parran Jr. and "Surface Active Agents and Detergents" (Vol. I & II) by Schwartz, Perry & Berch, both of which is also incorporated into the subject application by reference.

The term “slip modifier” is used herein to designate materials that when present at relatively low levels (generally less than 1.5% based on the total weight of the bar composition) will significantly reduce the perceived friction between the wet bar and the skin. The most suitable slip modifiers are useful at a level of 1% or less, preferably from 0.05 to 1% and more preferably from 0.05% to 0.5%.

Slip modifiers are particularly useful in bar compositions which contain starch/ cellulose and/or insoluble particles whose levels approach the higher end of the useful concentration range for these materials, e.g., 30-40% for starch with 5-10% insoluble particulate material. It has been found that the incorporation of higher levels of starch and/or insoluble particles increases the wet skin friction of the bar and the bars are perceived as “draggy” (have a high perceived level of frictional “drag” on the skin). Although some consumers do not mind this sensory quality, while others dislike the sensation. In general, consumers prefer bars that are perceived to glide easily over their skin and are perceived as being slippery.

It has been found that certain hydrophobic materials incorporated at low levels can dramatically reduce the wet skin frictional drag of bars containing higher levels of starch and/or insoluble particles to improve consumer acceptability.

Suitable slip modifier include petrolatum, waxes, lanolines, poly-alkane, -alkene, -polyalkalyene oxides, high molecular weight polyethylene oxide resins, silicones, poly ethylene glycols and mixtures thereof.

Particularly suitable slip modifiers are high molecular weight polyethylene oxide homopolymer resins having molecular weights of from about 100,000 to about 7,000,000. The polymers have a degree of polymerization from about 2,000 to about 100,000. These polymers are available as white powders.

Preferably the molecular weight of the polyethylene oxide resin is greater than 80,000, more preferably at least 100,000 Daltons and most preferably at least 400,000 Daltons. Examples of suitable high molecular weight polyethylene oxide resins are water soluble resins supplied by Dow Chemical Company under the trade name POLYOX. An example is WSR N-301 (molecular weight 4,000,000 Daltons).

Adjuvants are ingredients that improve the aesthetic qualities of the bar especially the visual, tactile and olefactory properties either directly (perfume) or indirectly (preservatives). A wide variety of optional ingredients can be incorporated in bars of the current invention. Examples of adjuvants include but are not limited to: perfumes; opacifying agents such as fatty alcohols, ethoxylated fatty acids, solid esters, and TiO_2; dyes and pigments; pearlizing agent such as TiO_2 coated micas and other interference pigments; plate like mirror particles such as organic glitters; sensates such as menthol and ginger; preservatives such as dimethyloldimethylhydantoin (Glydant XL1000), parabens, sorbic acid and the like; anti-oxidants such as, for example, butylated hydroxytoluene (BHT); chelating agents such as salts of ethylene diamine tetra acetic acid (EDTA) and trisodium etridronate (provided it is present at less than about 0.3%); emulsion stabilizers; auxiliary thickeners; buffering agents; and mixtures thereof.

The level of pearlizing agent should be between about 0.1% to about 3%, preferably between 0.1% and 0.5% and most preferably between about 0.2 to about 0.4% based on the total weight of the composition.

Adjuvants are commonly collectively designated as “minors” in the soap making art and frequently include at a minimum, colorant (dyes and pigments), perfume, preservatives and residual salts and oils from the soap making process, and various emotive ingredients such as witch-hazel. Minors generally constitute 4 to 10% by weight of the total continuous phase composition, preferably 4 to 8%, and often about 5-7% of the continuous phase.

A particular class of optional ingredients highlighted here is skin benefit agents included to promote skin and hair health and condition. Potential benefit agents include but are not limited to: lipids such as cholesterol, ceramides, and pseudoceramides; antimicrobial agents such as TRICLOSAN; sunscreens such as cinnamates; exfoliant particles such as polyethylene beads, walnut shells, apricot seeds, flower petals and seeds, and inorganics such as silica, and pumice; additional emollients (skin softening agents) such as long chain alcohols and waxes like lanolin; additional moisturizers; skin-toning agents; skin nutrients such as vitamins like Vitamin C, D and E and essential oils like bergamot, citrus unshiu, calamus, and the like; water soluble or insoluble extracts of avocado, grape, grape seed, myrrh, cucumber, watercress, calendula, elder flower, geranium, linden blossom, amaranth, seaweed, gingko, ginseng, carrot; impatiens balsamina, camu camu, alpina leaf and other plant extracts such as witch-hazel, and mixtures thereof.

The composition can also include a variety of other active ingredients that provide additional skin (including scalp) benefits. Examples include anti-acne agents such as salicylic and resorcinol; sulfur-containing D and L amino acids and their derivatives and salts, particularly their N-acetyl derivatives; anti-wrinkle, anti-skin atrophy and skin-repair actives such as vitamins (e.g., A,E and K), vitamin alkyl esters, minerals, magnesium, calcium, copper, zinc and other metallic components; retinoic acid and esters and derivatives such as retinal and retinol, vitamin B3 compounds, alpha hydroxy acids, beta hydroxy acids, e.g. salicylic

acid and derivatives thereof; skin soothing agents such as aloe vera, jojoba oil, propionic and acetic acid derivatives, fenamic acid derivatives; artificial tanning agents such as dihydroxyacetone; tyrosine; tyrosine esters such as ethyl tyrosinate and glucose tyrosinate; skin lightening agents such as aloe extract and niacinamide, alpha-glyceryl-L-ascorbic acid, aminotyroxine, ammonium lactate, glycolic acid, hydroquinone, 4 hydroxyanisole, sebum stimulation agents such as bryonolic acid, dehydroepiandrosterone (DHEA) and orizano; sebum inhibitors such as aluminum hydroxy chloride, corticosteroids, dehydroacetic acid and its salts, dichlorophenyl imidazoldioxolan (available from Elubiol); anti-oxidant effects, protease inhibition; skin tightening agents such as terpolymers of vinylpyrrolidone, (meth)acrylic acid and a hydrophobic monomer comprised of long chain alkyl (meth)acrylates; anti-itch agents such as hydrocortisone, methdilizine and trimeprazine hair growth inhibition; 5-alpha reductase inhibitors; agents that enhance desquamation; anti-glycation agents; anti-dandruff agents such as zinc pyridinethione; hair growth promoters such as finasteride, minoxidil, vitamin D analogues and retinoic acid and mixtures thereof.

Regardless of the optional agent or agents employed, their level should be chosen such that the composition is an extrudable mass (penetrometer hardness of 3 to 8 Kg and a yield stress of 350 to 2000 kPa measured at a temperature of 40°C) and the bars derived from the composition conveniently have a Cracking Index of 1 or less.

OPTIONAL DOMAINS DISPERSED IN THE CONTINUOUS PHASE

The extruded bars according to the invention can include various types of optional macroscopic domains generally having a different composition from the continuous phase which are dispersed or distributed either uniformly or non-uniformly in the continuous phase of the bar. The composition of the domains can

differ from the composition of the continuous phase in for example colorant, surfactant level and type, benefit agents, structurants and/or matrix.

Optional dispersed domains can include one or a combination of stripes or variegations such as described in U.S. Patents 4,634,564, 3,673,294, 3,884,605 and 6,383,999, chucks or bits as described in U.S. Patent 6,730,642, veins as described in U.S. Patent Application no. 2008/214430, plate like inclusions, and surface inclusions such as is described in U.S. Patent 7,538,077.

The domains can be included during extrusion by injection, coextrusion, dispersion and post-extrusion by surface impaction.

MATERIAL PROPERTIES OF AN EXTRUDED MASS

The personal washing bars described herein are extruded masses. By the term “extruded masses” is meant that the bars are made by a process which involves both the intensive mixing or working of the soap mass while it is in a semi-solid plastic state and forming into a cohesive mass by the process of extrusion.

The intensive mixing can be accomplished by one or more unit operations known in the art which can include roller milling, refining, and single or multistage extrusion. Such processes work (shear) the composition at a temperature between about 30°C and about 50°C to form a homogeneous network of insoluble solid materials dispersed in a viscous liquid and/or liquid crystalline phase containing the lower melting, more soluble surfactants (e.g., laurics and oleics soaps and other water soluble/dispersible materials).

An extruded mass must be thermoplastic within the process temperature of extrusion which is generally between about 30°C and about 45°C, preferably at a temperature between about 33°C to about 42°C. Thus, the material must soften within this process temperature window but remain highly viscous, i.e., the

material should not soften excessively to form a sticky mass. The material must regain its structure and harden quickly as the temperature is lowered below its softening point. This means that the internal structure must reform quickly generally by re-solidification of structure forming units, e.g., crystals.

Furthermore, the softened mass although pliable must be sufficiently viscous so that it does not stick to the surfaces of the extruder in order to be capable of conveyance by the extruder screws but not bend excessively when exiting the extruder as a billet. However, if the mass is too viscous it will not be capable of extrusion at reasonable rates. Thus, the hardness of the mass should fall within limits within the process temperature window to be capable of high rates of production as defined above.

Personal washing bars formed by extrusion (also commonly known as milled soaps) have physical-chemical properties and an internal structure which are quite different from soaps that are made by a melt-cast process wherein the bar composition is first melted at high temperature (e.g., 70°C) to form a liquid phase which is then poured into molds to solidify by quiescent cooling.

These differences in internal structure, composition and physical-chemical characteristics provide extruded personal washing bars with overall in-use properties which are better suited for the mass market than cast soaps. These properties include: much lower wear rates, more resistance to scuffing and denting, and a richer, more creamy opaque lather.

The one or more key properties that serve as characteristic “finger-prints” of an extruded mass are: (i) structural anisotropy (on micro and macro scale); (ii) the level of high melting point materials such as stearics soaps; (iii) high melting point and thermal reversibility; and (iv) rapid recovery of hardness after heating and shear. These characteristics are summarized in the Table 1.

Table 1 – Properties of Compositions Suitable for Extrusion

CHARACTERISTIC PROPERTY	EXTRUDABLE MASS	CAST SOAP
Structural anisotropy	Aligned crystals. Distinct flow lines/candle structure evident as small cracks formed after alcohol emersion.	Generally random crystal orientation. Absence of candle structure. No prominent and systematic lines or cracks evident after alcohol emersion.
Levels of stearic-rich soaps (C_{16}/C_{18} soaps)	20% to about 55% based on the total weight of soap.	Generally less than 15% or absent.
Melting point/Thermal characteristics	Melting point above 80°C, typically above 90°C and usually above 100°C. Relatively high degree of thermal reversibility (DSC).	Melting point 70°C to about 80°C. Relatively low degree of thermal reversibility.
Recovery of hardness after heating and shear	Recovers at least about 75%, preferably at least about 85% and more preferably at least about 95% of its initial hardness before shearing. Forms cohesive mass after extensional shear (Orifice Extruder).	After melting and casting ether low recovery of hardness after shear and/or lack of formation of cohesive mass after shear (excessive fracture or softening).

TEST METHODOLOGY

Rheological Properties - Hardness and Yield Stress

For a soap composition to be capable of being extruded and stamped at high speeds its rheological properties should meet certain criteria. Specifically the hardness of the mass and its yield stress should fall within certain limits.

A variety of methods are known in the art to measure the hardness and yield stress of soft solids such as toilet soaps. A technique used in the current work is the Penetration Test which measures the penetration of a needle or tapered rod under load. The distance travelled (penetration of the needle into the test mass) under a constant load or the load required to produce a given distance of penetration can be measured. In the test method used in the present work, the latter measurement approach is employed, i.e., variable load to achieve fixed penetration depth.

Although the invention is described by parameters that are measured by the Penetration Test, various other rheological methods can be used and inter-correlated with the methods used herein.

Hardness penetration measurements were made using finished toilet soap bars using the TA-XT Plus Texture Analyzer supplier by Stable Micro Systems.

The rheological parameters of the finished bars were determined by measuring the weight necessary for the test probe to penetrate the sample to a distance of 15 mm (see table below). The 30° conical test probe is made from X2 stainless steel and the dimensions are: Length, 60.4 mm; Diameter 30 mm.

The instrument parameters used in hardness analyses with TA-XT analyzer are given in the table below.

Table 2

Parameters	Value
Load cell capacity (kg)	10
Pre-speed (mm/s)	2
Return speed (mm/s)	10
Conical Probe Angle (°)	30
Trigger Force (g)	5
Test speed (mm/s)	1
Distance of penetration (mm)	15

The TA-XT Plus Texture Analyser allows for various preset probe speeds. In the examples described herein, the rheological parameters other than hardness were carried out at various speeds (minimum 10), ranging from 0.01 to 10 mm/sec and the forces measured accordingly. Shear stresses and shear rates were calculated and rheological charts were built. The rheological parameters were finally calculated by least-squares utilizing the Herschel-Bulkley equation:

$$\sigma = \sigma_0 + \kappa\dot{\gamma}^n$$

where σ is the shear stress, σ_0 is the yield stress, k stands for the consistency index, n is the flow index and $\dot{\gamma}$ is the shear rate.

Soap bar compositions suitable for the present invention should have a yield stress and penetrometer hardness (or simply a “hardness”) which falls within the following ranges:

Yield stress: from 350 to 2000 kPa when measured at a temperature of 40°C, preferably from 500 to 1000 kPa.

Hardness: from 3 to 8 Kg when measured at a temperature of 40°C, preferably from 4.5 Kg to 6.5 Kg.

The rheological properties of a thermoplastic soap composition not only depend upon the soap composition, structuring system and water content but also upon optional ingredients included in the bar composition. For example, inclusion of excessive amounts of low melting point emollients, e.g., mineral oil and/or liquid nonionic surfactants can lead to excessive softening of the bar composition. Conversely, the inclusion of excessive levels of electrolyte(s) or particulate material can produce a mass which is highly brittle and non-cohesive.

Measurements of yield stress and hardness provide a means for determining whether particular optional ingredients at the levels contemplated can be included in the bar composition without excessively compromising the extrusion and stamping rates of the composition. Thus, optional ingredients can be incorporated in the continuous phase composition provided that the mass has a penetrometer hardness of 3 to 8 Kg and a yield stress of 350 to 2000 kPa measured at a temperature of between 35 and 45, preferably around 40°C.

Wear Rate Test

The wear rate of the bar was measured by the following procedure.

Four weighed samples of each test bar are placed on soap trays. Two types of soap trays are employed: those that have drainers or raised grids so the water left on the bar after rinsing is drained away; and no drainers so that water can be added to the tray to allow the bars to become “water-logged”. The trays are

coded as follows:

With drainers?	Wash temperature (ºC)
Yes	25
Yes	40
No	25
No	40

10 ml of distilled water (ambient temperature) are poured into the undrained tray (25° and 40°C).

Each tablet of soap is treated as follows:

- A washing bowl is filled with about 5 litres of water, at the desired temperature (20°C or 40°C).
- The test tablets (bars of fixed mass and dimensions) are marked to identify top face (e.g. by making a small hole with a needle).
- Wearing waterproof gloves, the tablets are immersed one at a time in the water, and twisted 15 times (180° each time) in the hands above water.
- The tablet is immersed again in the water and twisted for more 15 times (180° each time) in the hands above water.
- The treated tablet is briefly immersed in the water to remove lather.
- The tablet is placed back on its soap tray, ensuring that the opposite face is uppermost (e.g., the unmarked face).

The above procedure is carried out 6 times per day for 4 consecutive days, at evenly spaced intervals during each day. Alternate face of each bar is placed in the downward position (facing the bottom of the tray) after each washdown. Between washdowns the soap trays should be left on an open bench or draining board, in ambient conditions. After each washdown cycle, the position of each soap tray/tablet is changed to minimize variability in drying conditions.

At the end of each day, each soap tray with drainer is rinsed and dried. Soap trays without drainers are refilled with 10 ml distilled water (ambient temperature). After the last washdown (4^{th} day), all soap trays are rinsed and dried. Each washed bar is placed in its tray and allowed to dry for up to a period of 9 days. On afternoon of the 5^{th} day, the samples are turned so that both sides of the tablet is allowed to dry. On the 8^{th} day, each tablet is weighed.

The rate of wear is defined as the percent weight loss as follows:

$$\%Wear = \frac{(\text{initial weight} - \text{final weight}) * 100}{\text{initial weight}}$$

Bar Mush – Mush Immersion Test

Mush is a paste or gel of soap and water, formed when soap is left in contact with water as in a soap-dish. Soluble components of the soap move into solution, and water is absorbed into the remaining solid soap causing swelling, and for most soaps, also re-crystallization. The nature of the mush depends on the balance of these solution and absorption actions. The presence of a high level of mush is undesirable not only because it imparts an unpleasant feel and appearance to the soap, but also especially because the mush may separate from the bar and leaves a mess on the wash basin. Residual mush or soap residue is a known consumer negative.

The Mush Immersion Test described herein gives a numerical value for the amount of mush formed on a bar. The test is carried out as follows:

A rectangular billet from the soap tablet is cut to the required dimensions using a plane, knife or cutting jig. The width and depth of the cut billet are accurately measured (+/- 0.1 cm). A line is drawn across the billet 5 cm from the bottom of the billet. This line represents the immersion depth.

The billet is attached to a sample holder and suspend in an empty beaker.

Demineralised (or distilled) water at 20°C is added to the beaker until the water level reaches the 5 cm mark on the billet. The beaker is placed in a water bath at 20°C (+/- 0.5°C) and left for exactly 2 hours.

The soap-holder + billet is removed, the water emptied from the beaker, and the soap-holder + billet is replaced on the beaker for 1 minute so that excess water can drain off. Extraneous water is shaken off, the billet is removed from the soap-holder, and the weight of the billet standing it on its dry end is recorded (W_M).

All the mush from all 5 faces of the billet is carefully scraped off, and any remaining traces of mush are removed by wiping gently with a tissue. The weight of the billet within 5 minutes of scraping is recorded (W_R).

The quantitative amount of mush is calculated as follows:

$$Mush(g/50cm^2) = \frac{W_M - W_r}{A} \times 50$$

where A is the surface area.

The amount of absorbed water is also calculated as follows:

$$\text{Absorbed water (g/50 cm}_2\text{)} = \frac{(W_M - W_O)}{A} \times 50$$

where W_O is the initial weight.

Accelerated Bar Cracking Test

The potential of bars to crack in use is assessed by washing the bars in a controlled manner 6 times per day for 4 days, storing the bars between washes under different conditions to simulate different consumer habits and then allowing the bars to dry for different periods of time to induce cracking. The procedure is as follows:

- Four weighed samples (bars) of each test composition are placed on soap trays in an identical arrangement as described above in the Wear Rate Test.
- 10 ml of distilled water (ambient temperature) are poured into the undrained tray (25° and 40°C).
- Each tablet of soap then treated in an identical manner as described above for the Wear Rate Test.
- After the last washdown (4^{th} day), all soap trays are rinsed and dried and each washed bar is placed in its tray and allows to dry for up to a period of 9 days.
- A subjective assessment of the degree of cracking is carried out on each bar. Some cracking may occur during the first 5 days of the test, but for maximum sensitivity and realism it is best to assess the cracking after drying out (i.e. on the 8th or 9th day).

A trained assessor examines the appearance of the cracking lines in the tablets and scores them as a function of the amount of cracks and the cracks depth in the whole bar as follows:

The degree of cracking is graded using the following 0-5 scale:

0 – No cracking

1 – Small and shallow cracking: Several short shallow cracks

2 – Small and medium deep cracking: Numerous short shallow cracks, or one or two long fairly deep cracking.

3 – Medium and deep cracking: One long deep wide crack or very numerous minor cracks

4 – Big and deep cracking: Two or more deep wide long cracks.

5 – Very big and very deep cracking: Numerous deep, wide long cracks

It is advantageous to use photographic standards representing each of these grades, produced from typical local soap samples. This gives greater consistency of assessment between technicians.

Swelling of the Bar

The swelling of the bar occur when the soap is left in contact with water in a poorly drained soap-dish. The water absorbed changes the bar size and its appearance which can be assessed visually by comparing with a bar that was kept under dry conditions.

The visual assessment is performed after the wear rate and cracking tests, the soap tablets are assessed visually for swelling. A trained assessor examines the tablets and records the degree of swelling.

The degree of swelling is graded using the following scale:

1 - Slightly swollen

3 - Moderated swollen

5 – Highly swollen

The amount of water absorbed can also be obtained by the following equation already described in the Mush Immersion Test protocol.

$$\text{Absorbed water (g/50 cm}_2\text{)} = \frac{(W_M - W_O) \times 50}{A}$$

EXAMPLES

The following non-limiting examples illustrate various aspects of the invention and preferred embodiments. Examples of the invention are designated with the prefix "Ex" while comparative examples are designated with the prefix "C".

Examples 1-2

These examples illustrates exemplary bar compositions according to the invention. The compositions used to prepare the continuous phase of the personal washing bars of Examples Ex1 and Ex2 are set forth in Table 3A and contain about 30% and 50% soap respectively (about 28% and 47% TFM). The structuring system is based on starch (native corn starch) as the polysaccharide, glycerin as the polyol and talc as the insoluble particulate material. Both compositions contain 1% of the anticracking agent sodium carboxymethyl cellulose (SCMC 9H from Danisco).

The continuous phase compositions and bars were prepared at pilot plant scale using conventional equipment used in the manufacture of extruded soap. The bars are 100% continuous phase, i.e., they do not contain a dispersed phase in the sense defined above. In summary, the compositions were prepared by combining soap noodles with the remaining ingredients in Table 3A in a Z-blade mixer and passing the mixture through a 3-roll mill and a refiner. The compositions of the soap noodles are given in the second row of the table and were composed of a mixture of fatty acids derived from palm kernel oil (PKO - laurics soap) and a mixture derived from of palm oil (PO) and palm stearin (POS), sources of stearics and oleics soaps, using the weight ratios set forth in the table.

The mixture was added to the hopper of a two stage extruder and extruded at a temperature of 35°C to 45°C at an extrusion rate between about 1.2 – 4.0 Kg/min through an eyeplate having a 3.5 X 3.5 cm cross section to form billets that were cut to about 12 cm in lengths. The billets were then transferred to a manual soap stamper and stamped to form the finished personal washing bar utilizing a die set defining a mold having a volume of approximately 79 to 80 cm^3.

The physical and overall user properties are included in the bottom part of the Table 3B. The second column from the left under physical properties provides the "ideal range" for these properties for bars that combines excellent manufacturability (hardness, yield stress, and extrusion throughput at pilot scale) and highly acceptable in-use properties (high lather volume, low wear rate, no excessive swelling when sits in water, and very low cracking potential). The results in Table 3B indicate that the exemplary compositions exhibit a hardness and yield stress that will allow high throughput manufacture (e.g., 36 Kg per minute) and excellent in-use properties with little cracking in the accelerated cracking test.

Table 3: Composition and Physical Properties of Ex1 and Ex2

		Ex 1	Ex 2
A. Continuous Phase Composition			Wt%
Fatty Acid soap (40/40/20 – PO/POS/PKO)		30.5	51.0
Polyol (glycerin)		10.0	6.0
Polysaccharide (Native Corn Starch)		30.0	14.0
Inorganic fillers (Talc)		5.0	10.0
Anticracking agent (SCMC 9H)		1.0	1.0
Minors (colorants, pigments, preservatives)		5.5	0.6
Sunflower Oil		-	-
Sodium Chloride		-	0.7
Water		17.0	16.0
Water Soluble Builder (STPP)		-	-
Fragrance		1.0	0.7
	TFM	**28**	**47**
B. Physical Properties	**Ideal values**		
Hardness	3 - 8	7	3.5
Lather volume (ml)	≥ 200	204	200
Yield Stress (YS)	> 350	600	540
Extrusion throughput	High	High	High
Rate of Wear (%)	< 48	32	42.5
Bar swelling	1	1	1
Cracking	(0 - 1)	1	1

Comparative Examples C1-C4

The compositions used to prepare the personal washing bars of Comparative Examples C1-C4 are shown in Table 4A. Comparative example C1, C3 contains no anticracking agent, while comparative C2, C4 contains anticracking agent and 0.3% of the water soluble builder sodium tripolyphosphate (STPP). The bars were prepared according to the methods described for Examples Ex1 and Ex2. The

organization and form of the Table 4B and the meanings of the parameters are as described above in Ex1 and Ex2.

All the comparative bars can be extruded at high speed. However, in contrast to Examples Ex 1 and Ex 2, the comparative composition C1 and C3 which do not contain the anticracking agent exhibit much higher cracking potential than the corresponding compositions falling within the scope of the present invention Ex1 and Ex2 respectively.

Surprisingly, as little as 0.3% of the water soluble builder salt, STPP, had a very significant impact on cracking potential and completely negated the effect of the anticracking agent (compare Ex1 with C2 and Ex2 with C4).

Table 4: Comparative Examples 1-4

		C1	C2	C3	C4
A. Continuous Phase Composition		Wt%	Wt%	Wt%	Wt%
Fatty Acid soap (40/40/20 – PO/POS/PKO)		30.5	29.2	51.0	50.7
Polyol (glycerin)		10.0	10.0	6.0	6.0
Polysaccharide (Native Corn Starch)		30.0	30.0	14.0	14.0
Inorganic fillers (Talc)		5.0	5.0	10.0	10.0
Anticracking agent (SCMC 9H)		-	1.0	-	1.0
Minors (colorants, pigments, preservatives)		5.5	5.5	0.6	0.6
Sunflower Oil		-	-	-	-
Sodium Chloride		-	-	0.7	0.7
Water		18.0	18.0	17.0	16.0
Water Soluble Builder (Sodium tripolyphosphate)		-	0.30	-	0.3
Fragrance		1.0	1.0	0.7	0.7
	TFM	**28**	**27**	**47**	**47**
B. Physical Properties	**Ideal values**				
Hardness	3 - 8	6	5	3.4	3.9
Lather volume (ml)	>= 200	217	193	213	188
Yield Stress (YS)	> 350	700	600	394	574
Extrusion throughput	High	High	High	High	High
Rate of Wear (%)	<48	44	40	44.1	38.3
Bar swelling	1	1	1	1	1
Cracking	(0 - 1)	5	3	3	3

Comparative Examples 5-6

The compositions of comparative examples C5 and C6 are set forth in Table 5A. and correspond to bars having 40 and 48% soap respectively and contain 3.5% sodium carboxymethyl cellulose. The bars were prepared according to the methods of Examples Ex1 and Ex2 and were 100% continuous phase bars. The organization and form of the Table 5B is the same as described above.

Table 5: Comparative Examples C5 and C6

		C5	C6
A. Continuous Phase Composition			Wt%
Fatty Acid soap (40/40/20 – PO/POS/PKO)		40.5	48.5
Polyol (glycerin)		8.5	6.0
Polysaccharide (Native Corn Starch)		21.00	14.0
Inorganic fillers (Talc)		8.0	10.0
Anticracking agent (SCMC 9H)		3.5	3.5
Minors (colorants, pigments, preservatives)		0.5	0.6
Sunflower Oil		-	-
Sodium Chloride		0.7	0.7
POLYOX© (N60K from Dow Chemicals)		0.1	
Water		16.5	16.0
Water Soluble Builder (STPP)		-	-
Fragrance		0.7	0.7
	TFM	**37**	**45**
B. Physical Properties	**Ideal values**		
Hardness	3 - 8	4.2	4.4
Lather volume (ml)	≥ 200	127	133
Yield Stress (YS)	> 350	574	631
Extrusion throughput	High	High	High
Rate of Wear (%)	<48	30.7	31.2
Bar's swelling	1	5	5
Cracking	(0 - 1)	1	1

The comparative examples C5 and C6 containing 3.5% anticracking agent (SCMC) have a low cracking potential. However, relative to Examples E 1 and E2 there is a dramatic reduction in lather volume and the bars exhibit excessive swelling when they are allowed to contact water for a prolonged period as would occur in a wet soap dish. The level of anticracking agent should be kept below 3% and preferably below about 2.5% based on the weight of the continuous phase.

Example Ex3 and Comparative Examples C7-C9 (40% TFM)

The compositions of example Ex 3 and comparative examples C7-C9 are set forth in Table 6A (about 43% fatty acid soap). The bars were prepared according to the methods of Ex1 and Ex2. The bars have no dispersed phase.

Table 6: Example Ex3 and Comparative Examples C7-C9

		Ex3	C7	C8	C9
A. Continuous Phase Composition			Wt%		
Fatty Acid soap (40/40/20 – PO/POS/PKO)		43.1	**42.1**	**44.0**	**43.3**
Polyol (glycerin)		8.5	8.5	8.5	8.5
Polysaccharide (Native Corn Starch)		21.0	21.0	21.0	21.0
Inorganic fillers (Talc)		8.0	8.0	8.0	8.0
Anticracking agent (SCMC 9H)		0.8	0.8	0.8	0.8
Minors (colorants, pigments, preservatives)		0.6	0.6	0.6	0.6
Polyox (N60K from Dow Chemical)		0.1	0.1	0.1	0.1
Sodium Chloride		0.7	-	-	0.7
Water		16.5	17.9	16.0	16.0
Water Soluble Builder – STP		-	0.3	-	-
Water Soluble Builder – TKPP		-	-	0.3	-
Water Soluble Builder – EDTA		-	-	-	0.3
Fragrance		0.7	0.7	0.7	0.7
	TFM	**40**	**40**	**40**	**40**
B. Physical Properties	**Ideal values**				
Hardness	3 - 8	3.56	3.47	5.0	5.5
Lather volume (ml)	≥ 200	208	186	175	182
Yield Stress (YS)	> 350	455	483	520	620
Extrusion throughput	High	High	High	High	High
Rate of Wear (%)	<48	38.8	39.2	38.2	37.5
Bar's swelling	1	1	1	1	1
Cracking	(0 - 1)	1	3	4	3

The exemplary composition Ex 3 differs from the comparative examples only in that 0.3% of the fatty acid soap has been replaced by 0.3% of the soluble builder salts sodium tripolyphosphate (STP), tetrasodium pyrophosphate (TKPP) or the sodium ethylenediamine tetraacetate (EDTA). It is clear from the results that the inclusion of as little as 0.3% of these water soluble builder salts essentially negates the beneficial effects of the anticracking agent on cracking potential.

Example 4 and 5

The compositions of Examples Ex 4 and Ex5 are set forth in Table 7A. The bars were prepared according to the methods described previously. The bars do not contain a dispersed phase. Examples Ex4 and Ex5 employ Carbopol© 980 from Noveon as the anticracking agent. The bars have excellent properties and exhibit a low cracking potential (1 or less) in contrast to control compositions in which the polymers are replaced by soap (3-5) – not shown.

Table 7: Examples Ex4 and Ex5

		Ex 4	Ex 5
A. Continuous Phase Composition		Wt%	Wt%
Fatty Acid soap (40/40/20 – PO/POS/PKO)		28.7	50.7
Polyol (glycerin)		10	6
Polysaccharide (Native Corn Starch)		30	14
Inorganic fillers (Talc)		5	10
Anticracking agent (Carbopol 940)		1.2	1.0
Minors (colorants, pigments, preservatives)		5.5	0.6
Sunflower Oil		-	-
Sodium Chloride		0.75	0.7
Water		17.85	16
Water Soluble Builder (STPP)		-	-
Fragrance		1.0	1.0
	TFM	**27**	**47**
B. Physical Properties	**Ideal values**		
Hardness	3 - 8	6	4
Lather volume (ml)	≥ 200	200	215
Yield Stress (YS)	> 350	650	520
Extrusion throughput	High	High	High
Rate of Wear (%)	<48	33	40
Bar swelling	1	1	1
Cracking	(0 - 1)	0	1

Examples 6-9

Table 8 further illustrates continuous phase compositions according to the present invention.

Table 8: Compositions of Examples Ex6-Ex9

	Ex6	**Ex7**	**Ex8**	**Ex9**	**Ex10**	**Ex11**
A. Continuous Phase Composition		Wt%				
Fatty Acid Soap #2 (40/40/20 – PO/POS/PKO)	22.6	-	36.2	47.0	43.3	51.5
Fatty Acid Soap #3 (75/5/20 – Tallow/SBO/PKO)	-	28.9		-	-	
Polyol (glycerin)	10	10	10	8.5	8.5	6.0
Polysaccharide (Native Starch)	30	30	25	21	10	10
Polysaccharide (Microcrystalline cellulose)	10	-	-	-	11	-
Inorganic fillers (Talc)	--	5.0	5	-	5	-
Inorganic fillers ($CaCO_3$)		-	-	5.0	-	10
Anticracking agent (SCMC 9H)	1.5	-	-	0.8	1.0	0.5
Anticracking agent (Carbopol 940)	-	1.35	1.0	-	-	-
Auxiliary surfactant 1	5.0	-	5.0	-		2.0
Auxiliary surfactant 2	-	5.0	-	-	3.0	-
Sodium Chloride	0.75	-	-	0.7	0.7	-
Sodium sulfate	-	1.0	1.0	-	-	-
Polyox© N60K from Dow Chemicals	0.1	-	0.1	0.1	-	-
Emotive agent 1	0.5	-		-	-	-
Emotive agent 2	-	0.3		-	-	-
Emotive agent 3	-	-	0.2	-	-	0.5
Minors (colorants, pigments, preservatives)	0.5	0.5	0.5	0.5	0.5	0.5
Fragrance	1.0	1.0	1.0	0.8	1.0	1.0
Water	18.05	16.95	15	15.6	16.0	18.0

Example 10 and Comparative Example 10-12

The compositions of example Ex 10 and comparative examples C11-C13 are set forth in Table 9A. The bars were prepared according to the methods described previously. The bars do not contain a dispersed phase.

Table 9: Examples Ex10 and Comparative examples C11-C12

		Ex 10	C11	C12	C13
A. Continuous Phase Composition			Wt%		
Fatty Acid soap (75/5/20 – Tallow/Soya Bean Oil/PKO)		51.0	51.0	55	55
Polyol (glycerin)		6.0	6.0	6.0	6.0
Polysaccharide (Native Corn Starch)		14.0	14.0	14	19
Inorganic fillers (Talc)		10.0	10.0	5	-
Anticracking agent (Carbopol© 940)		1.0	-	-	-
Minors (colorants, pigments, preservatives)		0.6	0.6	0.6	0.6
Sunflower Oil			-	-	-
Coconut Fatty Acid		-	1.0	1.0	1.0
Sodium Chloride		0.7	0.7	0.7	0.7
Water		16.0	16.0	16.7	16.7
Water Soluble Builder (STPP)		-	-	-	-
Fragrance		0.7	0.7	1.0	1.0
	TFM	**47**	**47**	51	51
B. Physical Properties	**Ideal values**				
Hardness	3 - 8	3.9	3.4	4.2	5.0
Lather volume (ml)	≥ 200	217	213	210	200
Yield Stress (YS)	> 350	517	394	435	510
Extrusion throughput	High	High	High	High	High
Rate of Wear (%)	<48	39.9	44.1	39	42
Bar swelling	1	1	1	1	1
Cracking	(0 - 1)	1	3	1	1

Example Ex11 and Comparative Examples C14-C16

These Examples illustrate optimal levels of anti-cracking agent computed from Equation 1 above. The compositions of Example Ex 11 and Comparative Examples C14-C16 are set forth in Table 10A. The bars were prepared according to the methods of Ex1 and Ex2. The bars have no dispersed phase.

The exemplary composition Ex 11 differs from the comparative examples principally in having a level of anti-cracking agent (SCMC) in the optimal range computed according to Equation 1 and is free of detergent builder salts. This bar is essentially crack-free. In contrast bars having a continuous phase which either contains a lower level of anticracking agent which is outside the required optimal range (C14 and C15) or which contains as little as 0.3% builder salt exhibit substantial cracking.

Table 10: Example Ex 11 and Comparative Examples C14-16

		Ex11	C14	C15	C15
A. Continuous Phase Composition		Wt%			
Fatty Acid soap (40/40/20 – PO/POS/PKO)		20.54	20.54	19.54	20.24
Polyol (glycerin)		10.0	10.0	10.0	10.0
Polysaccharide (Native Corn Starch)		40.0	40.0	40.0	40.0
Inorganic fillers (Talc)		5.0	5.0	5.0	5.0
Anticracking agent (SCMC 9H)		2.0	1.0	-	2.0
Minors (colorants, pigments, preservatives)		5.46	5.46	5.46	5.46
Sodium Chloride			-	1.0	
Water		16.0	17.0	18.0	16.0
Water Soluble Builder – STP		-	-	-	0.3
Fragrance		1.0	1.0	1.0	1.0
%Unsaturated Soaps/Water Ratio		0.41	0.41	0.39	0.40
Required level anticracking agent according to Equation 1		1.9 to 2.9			
	TFM	19	19	18	19
B. Physical Properties	**Ideal values**				
Hardness	3 – 8	7	6	7	8
Lather volume (ml)	≥ 200	207	199	201	195
Yield Stress (YS)	> 350	730	720	700	700
Extrusion throughput	High	High	High	High	High
Rate of Wear (%)	<48	46	47	48	45
Bar swelling	1	1	1	1	1
Cracking	(0 – 1)	0	3	5	3

CLAIMS

1. A low TFM extruded personal washing bar comprising a continuous phase, said continuous phase being substantially free of water soluble detergent builder and comprising:
   a. 20% to 50% fatty acid soap, wherein the fatty acid soap comprises less than 39% of an unsaturated fatty acid soap by weight of the fatty acid soap;
   b. a structuring system comprising:
      i. from 10% to 45% by weight of the continuous phase of a polysaccharide structurant selected from the group consisting of starch, cellulose and a combination thereof,
      ii. from 6% to 30%, by weight of continuous phase of a polyol selected from the group consisting of glycerol, sorbitol and their mixtures, and
      iii. 0 to 15% by weight of continuous phase of a water insoluble particulate material;
   c. 0.5% to less than 3% of an anticracking agent selected from the group consisting of carboxymethyl cellulose, polyacrylates and mixtures thereof;
   d. from 10% to 20% water;

   wherein the bar has a Cracking Index of 1 or less; and wherein the continuous phase is an extrudable mass having a penetrometer hardness of 3 to 8 Kg and a yield stress of 350 to 2000 kPa measured at a temperature of 40° C.

2. The composition according to claim 1 wherein the fatty acid soap is derived from triglycerides selected form the group consisting of tallow, coconut oil,

palm oil, palm kernel oil, palm stearin, babasu oil, soybean oil, sunflower oil, crude soybean oil and mixtures thereof.

3. The composition according to claim 1 or claim 2 wherein the fatty acid soap comprise 25% to 40% of the continuous phase by weight.

4. The composition according to any one of the preceding claims wherein the fatty acid soap component comprises 32% to 37% unsaturated fatty acid soap by weight of the fatty acid soap.

5. The composition according to any one of the preceding claims wherein the polysaccharide structurant comprises 20% to 40% and the polyol comprises 10% to 20% by weight of the continuous phase.

6. The composition according to any one of the preceding claims wherein the starch is a natural starch, a pre-gelatinized starch, a chemically modified starch or mixtures thereof; and wherein the cellulose is microcrystalline cellulose, hydroxyalkyl alkylcellulose ether or mixtures thereof.

7. The composition according to any one of the preceding claims wherein the water insoluble particulate material is an inorganic particulate material selected from the group consisting of carbonates, sulfates, silicates, clays, aluminates, phosphates, talc, and mixtures thereof or an organic particulate material selected from synthetic polymers.

8. The composition according to any one of the preceding claims wherein the water insoluble particulate material comprise from 4% to 10% of the continuous phase by weight.

9. The composition according to any one of the preceding claims wherein the anti-cracking agent comprises 0.5% to less than 2.5% of the continuous phase.

10. The composition according to any one of the preceding claims wherein the weight % of anti-cracking agent in the continuous phase falls within a range which given by the following relationship (1):

$$\% \text{ Anti-cracking agent} = (4.3\ e\text{-}1.68R_{us/w}) \pm 15\% \qquad (1)$$

where $R_{us/w}$ is the ratio of the percent total unsaturated soaps in the continuous phase to the percent water in the continuous phase.

11. The composition according to any one of the preceding claims wherein the continuous phase further comprises less than 2% of a soluble inorganic electrolyte containing sodium or potassium cations in combination with a monovalent or divalent anion.

12. The composition according to claim 11 wherein the total concentration of soluble inorganic electrolyte is no more than 0.85 % of the continuous phase.

13. The composition according to any one of the preceding claims further comprising a synthetic surfactant present at a level of 10% or less based on the total weight of the bar composition wherein the synthetic surfactant is selected from the group consisting of non-soap anionic surfactants, nonionic surfactants, amphoteric surfactants and mixtures thereof.

14. The composition according to any one of the preceding claims further comprising from 0.05 to 1% of a slip modifier selected from the group consisting of petrolatum, waxes, lanolines, polyalkanes, polyalkenes, high

molecular weight polyethylene oxide resins, silicones, poly ethylene glycols and mixtures thereof.

15. The composition according to any one of the preceding claims wherein the composition immediately after processing has a nominal water content of between 15% and 18% based on the total weight of composition.

16. The composition according to any one of the preceding claims wherein the continuous phase has a level of water soluble detergent builder which is less than 0.5%, preferably less than 0.3% by weight based on the weight of the continuous phase.

17. The composition according to any one of the preceding claims wherein the personal washing bar has a level of water soluble detergent builder which is less than 0.5%, preferably less than 0.3% by weight based on the weight of the bar.

18. The composition according to any one of the preceding claims wherein the anti-cracking agent is carboxymethyl cellulose,

INTERNATIONAL SEARCH REPORT

International application No
PCT/EP2010/069926

A. CLASSIFICATION OF SUBJECT MATTER
INV. C11D9/00 C11D9/18 C11D9/22 C11D9/26
ADD.

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
C11D

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)
EPO-Internal, WPI Data

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	GB 2 459 093 A (UNILEVER PLC [GB]; UNILEVER PLC [GB]) 14 October 2009 (2009-10-14) cited in the application page 12; claims; examples	1-18
A	EP 0 186 148 A2 (GOODRICH CO B F [US]) 2 July 1986 (1986-07-02) claims; examples	1-18
A	US 2007/155639 A1 (SALVADOR CHARLIE R [CN] ET AL) 5 July 2007 (2007-07-05) paragraph [0021]; claims	1

☐ Further documents are listed in the continuation of Box C.

☒ See patent family annex.

* Special categories of cited documents :

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Date of the actual completion of the international search	Date of mailing of the international search report
8 April 2011	20/04/2011

Name and mailing address of the ISA/	Authorized officer
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INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No
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