PROCESS FOR PREPARING AQUEOUS SOAP-SYNTHETIC DETERGENT MIXTURES IN RIBBON FORM

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The present invention relates to a process for preparing aqueous soap-synthetic detergent mixtures in the form of ribbons suitable for drying on belt conveyortype driers. More particularly it relates to a process for preparing ribbons of soap and fatty acid monoglyceride sulfate detergent which are suitable for processing into chip form, and thence into toilet bar form.

It has previously been proposed to prepare soap-fatty acid monoglyceride sulfate detergent bars so as to realize the desirable properties of both soap and the synthetic detergent within a single bar. Various methods have been proposed for preparing such bars, one of such methods consisting of "dry milling" in which soap chips, containing usually about 5 to 10% moisture, are mixed with substantially dry particles of the synthetic detergent, the mixture is passed through milling rolls to grind and homogenize the ingredients, and the mixture is then molded, cut, and stamped in the fashion of soap bars. This "dry milling" procedure is not entirely satisfactory in that such milling seldom effects complete homogenization—some discrete particles usually remain in the final product, which may even have a speckled appearance.

A satisfactory method of producing a more truly homogeneous soap-fatty acid monoglyceride sulfate detergent "combination bar" involves "wet milling." In this process, the synthetic detergent, in the form of an aqueous slurry, is mixed with ordinary kettle soap, the aqueous mixture is thoroughly agitated to achieve uniformity, and the homogeneous mixture is then processed similarly to ordinary toilet soap, i.e., it is fed to a chill roll, taken off as ribbons, dried in a belt conveyor-type drier, and broken into chips which are then milled, plodded and stamped in the manner of soap bars.

The wet milling procedure is highly satisfactory for combination bars containing a maximum of up to about 25 to 30% of the synthetic detergent. Unfortunately, above this range of synthetic detergent content, the wet mixture gradually loses certain of its soap-like characteristics and normally does not coagulate sufficiently on a chill roll to enable the development and removal from the roll of numerous form-retaining ribbons. Rather, an amorphous, soft sticky elongated mass forms where the serrated ribbon-forming removal blade contacts the drum. These masses frequently have the appearance of and are referred to as "cigars." Such "cigars" are highly undesirable as they cannot be properly supported and dried on the woven mesh belts of the belt conveyor-type driers employed in soap processing. Thus, it has not been possible to satisfactorily handle mix containing relatively high proportions of the synthetic detergent in conventional soap-processing equipment.

It has now been discovered that by the proper selection of higher fatty acid soap, it is possible to carry out the "wet milling" process with compositions containing as much as about 65% fatty acid monoglyceride sulfate detergent and still obtain a form-retaining ribbon on chilling the wet mix. In accordance with the present invention, a process for the preparation of homogeneous form-retaining ribbons of an aqueous mixture of synthetic detergent and soap, suitable for drying on belt conveyor-type driers and processing into toilet bars, comprises forming a homogeneous aqueous mixture of fatty acid monoglyceride sulfate detergent and sodium soap of a substantially completely saturated higher fatty acid containing at least about 16 carbon atoms, said soap comprising about 35 to 70% of the solids in said aqueous mixture, chilling said mixture in sheet form to a temperature below its setting-up point, and dividing the resulting chilled sheet into form-retaining ribbons. The thus formed ribbons may be dried in a conventional belt conveyor-type drier, milled, plodded into bar form, cut and stamped.

The crux of the present invention is the discovery that by employing substantially completely saturated soaps of the higher fatty acids, i.e., palmitic and stearic acids or mixtures thereof such as hydrogenated tallow, it is possible to prepare a form-retaining ribbon under conditions which normally lead merely to the formation of soft amorphous masses, i.e., "cigars." It has been found that to satisfactorily carry out the process of the present invention, the saturated higher fatty acid soap employed must constitute a minimum of about 35% and preferably about 40% of the composition to be prepared. Normally, the instant soaps will not form more than about 65% to 70% of the final product, as, at higher soap contents more conventional soap process satisfactorily and are more desirable from the viewpoint of rapid foaming and increased solubility, due to the diminished proportion of the synthetic detergent present. The soaps of the present invention normally have an iodine value (I.V.) of a maximum of about 15, and when the proportion of soap is relatively low, preferably less than about 5. While the soap may be prepared in situ by neutralization of the proper fatty acid with caustic or other base, it is preferably supplied in the form of kettle soap containing about 65 to 70% solids and 35 to 30% water.

The synthetic detergent of the instant invention is a sulfated higher fatty acid monoglyceride as exemplified by hydrogenated coconut oil monoglyceride sulfate, coconut oil monoglyceride sulfate and tallow monoglyceride sulfate, and, more broadly, sulfated monoglycerides of fatty acids containing about 12 to 18 carbon atoms and mixtures thereof. Under certain circumstances this material may be replaced in part by suitable amounts of higher fatty alcohol sulfates containing about 8 to 18 carbon atoms.

The fatty acid monoglyceride sulfate detergent, when intended to be mixed with kettle soap, contains about 60%, and preferably about 70% detergent solids, the remainder being substantially completely water. Thus, the aqueous mixture of soap and synthetic detergent normally contains about 70%, and not less than about 65% solids, or, putting it inversely, the aqueous mixture normally contains no more than about 35% water, as mixes containing a higher proportion of water are generally unsatisfactory in that ribbon formation becomes difficult therewith. Of course, sufficient water must be present so that the mixture is fluid and easily homogenizable (i.e., about 30%). The fatty acid monoglyceride detergent is free of accompanying sodium sulfate. However, it is one modification of the instant process to have present in the aqueous mixture of soap and the synthetic detergent, up to about 4 to 5% of sodium chloride which acts to "shorten" the synthetic detergent mixture and render it more amenable to easy coagulation and ribbon formation on the chilling roll.

The mixing of the soap and the synthetic detergent occurs at an elevated temperature (i.e., normally about 160 to 190° F.) above the "setting" or "setting-up" point.
of the mixture. Operation at such temperatures assures the fluidity necessary to a homogenizable, pumpable mix, which contains sufficient solids to rapidly "set-up" or become non-fluid when chilled below its "setting-up" point, i.e., about 75° to 90°F. The range of temperatures commonly realized on chill rolls (which are normally cooled to about 55°-65°F).

The following examples are given to additionally illustrate the nature of the invention and it will be understood that the invention is not limited thereto. (All parts are by weight unless otherwise specified.)

**EXAMPLE I**

Soap/synthetic detergent = 45:55

In a jacketed Abbé-type mixer are placed 60.35 parts of a 61.89% solids (97.7% alcohol soluble) detergent solution of sodium hydrogenated coconut oil monoglyceride sulfate. 27.42 parts of commercial stearic acid (52-48 palmitic-stearic acids, IV. 3 maximum) and the mixture is agitated until uniform. 9.32 parts of a 44% solution of caustic soda are then incorporated, followed by 2.91 parts of sodium chloride. Agitation is continued, and when homogeneous, the mixture is transferred to the hopper of a conventional chill roll, dropped on the rotating roll (maintained at 60 to 65°F.) and formed into ribbons. The ribbons are dried on a belt conveyor-type drier and processed into chips at 5-6% moisture, and subsequently into milled and pooled bars using conventional toilet soap making equipment. Perfume, pigment, and preservative is used as desired.

**EXAMPLE II**

Soap/synthetic detergent = 57:43

Into a jacketed Abbé-type mixer containing 55.15 parts of detergent solution as in Example I above is added with mixing, 41.94 parts of hydrogenated tallow (5 I.V.) melt soap (30% water) and 2.91% of sodium chloride. When uniformly mixed, the batch is transferred to the hopper of the chill roll of a conventional toilet soap chip drier and processed into chips at 5.7% moisture as in Example I. The chips are mixed with perfume, preservative and titanium dioxide and are then milled, pooled, cut and stamped into finished cakes.

The instant soap-synthetic detergent mixtures are not narrowly limited to soap or synthetic detergent as constituents. Although there are their principal components, there may also be present in the final mixture small proportions or various other substances such as slip improving agents, e.g., carboxymethylcellulose, higher fatty alcohols, and diethylene glycol monostearate, germicides, fillers, builders, coloring matter, pigments, dyes and the like. Furthermore, compositions processed according to the instant invention are not restricted to use in preparing toilet bars but may be used in chip, pulverized or other form for any desired purpose.

While there has been disclosed and described what is at present considered to be the preferred embodiments of the invention it will be understood, of course, that many changes, modifications, and substitutions may be made therein without departing from the true scope of the invention as defined in the appended claims.

What is claimed is:

1. A process for the preparation of homogeneous, form-retaining ribbons of an aqueous mixture of synthetic detergent and soap comprising forming, at a temperature above its setting-up point, a homogeneous aqueous mixture of a sodium soap of a substantially completely saturated higher fatty acid containing from about 16 to 18 carbon atoms and a sodium salt of a higher fatty acid monoglyceride sulfate detergent wherein said fatty acid contains about 12 to 18 carbon atoms, said aqueous mixture containing about 30 to about 50% water and said soap comprising about 35 to 70% of the solids in said mixture, chilling said mixture to a temperature below its setting-up point, and forming the thus set-up mixture into form-retaining ribbons.

2. A process for the preparation of homogeneous, form-retaining ribbons of an aqueous mixture of synthetic detergent and soap as set forth in claim 1 wherein said substantially completely saturated higher fatty acid is derived from hydrogenated tallow having an iodine value of a maximum of about 15.

3. A process for the preparation of homogeneous, form-retaining ribbons of an aqueous mixture of synthetic detergent and soap which comprises forming, at a temperature above its setting-up point, a mixture of an aqueous sodium salt of a higher fatty acid monoglyceride sulfate detergent wherein said fatty acid contains about 12 to 18 carbon atoms and a substantially completely saturated higher fatty acid containing from about 16 to 18 carbon atoms, introducing into said mixture sufficient caustic soda to substantially completely neutralize said saturated fatty acid to form a homogeneous mixture of sodium soap, water, and said sulfite detergent, said homogeneous mixture containing about 30 to about 35% water and said soap comprising about 35 to 70% of the solids in said mixture, chilling said homogeneous mixture to a temperature below its setting-up point, and forming the thus set-up mixture into form-retaining ribbons.

4. A process for the preparation of homogeneous, form-retaining ribbons of an aqueous mixture of synthetic detergent and soap as set forth in claim 3 wherein said substantially completely saturated fatty acid is palmitic acid.

5. A process for the preparation of homogeneous, form-retaining ribbons of an aqueous mixture of synthetic detergent and soap as set forth in claim 3 wherein said substantially completely saturated fatty acid is stearic acid.

6. A process for the preparation of homogeneous, form-retaining ribbons of an aqueous mixture of synthetic detergent and soap comprising forming, at a temperature of about 160° to 190° F., a homogeneous aqueous mixture of a sodium salt of a higher fatty acid monoglyceride sulfate detergent wherein said fatty acid contains from about 12 to 18 carbon atoms and sodium soap of a substantially completely saturated higher fatty acid containing about 16 to 18 carbon atoms, said mixture containing about 30 to about 35% water and said soap comprising about 35 to 70% of the solids in said mixture to a temperature of about 75° to 90°F. to cause it to set-up, and forming the thus set-up mixture into form-retaining ribbons.

7. A process for the preparation of homogeneous, form-retaining ribbons of an aqueous mixture of synthetic detergent and soap comprising forming, at a temperature above its setting-up point, a homogeneous aqueous mixture of a sodium salt of a higher fatty acid monoglyceride sulfate detergent wherein said fatty acid contains from about 12 to 18 carbon atoms, sodium soap of a substantially completely saturated higher fatty acid containing about 16 to 18 carbon atoms, water and up to about 5% of sodium chloride, said aqueous mixture containing about 30 to 35% water and said soap comprising about 35 to 70% of the solids in said mixture, chilling said mixture to a temperature below its setting-up point, and forming the thus set-up mixture into form-retaining ribbons.

**References Cited in the file of this patent**

**UNITED STATES PATENTS**

2,572,077 Preston Oct. 24, 1950

2,649,417 Cramer Aug. 18, 1953

2,704,279 Heald Mar. 15, 1955

**FOREIGN PATENTS**

723,925 Great Britain Feb. 16, 1955