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TREATMENT OF SOAP PARTICLES

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The present invention relates to an improved type of soap particles and methods for preparing same and particularly relates to a process for reducing the balling tendencies of silicate-containing soap particles.

Soap products in particulate form such as spray dried soaps, powdered or ground soaps, granules, etc. that are prepared from fat formulas having little or no fatty matter derived from oils of the coconut oil group, have a tendency, when added to warm water, to form difficultly soluble masses or aggregates of the individual particles before solution of the soap can be effected. This tendency which is sometimes referred to as "balling" is characteristic of many of the products on the market today. While balling is increased in part by variables such as decreased moisture and increased bulk density of the product, it is dependent to a greater degree on the fat formulation prior to saponification. Thus, the use of large amounts of oils of the coconut oil group in the fat formula has generally been the most effective means of controlling the balling tendency. Heretofore, numerous methods of particle treatment have also been proposed for reducing or eliminating balling, including surface application of materials such as glues, starches, water, wetting agents, gums and mineral oils. However, as far as I am aware these procedures have not met with commercial success, primarily because the performance of the product in sudsing and detergency is adversely affected by the large amount of material required to achieve desired improvement in balling.

It is an object of this invention to produce silicate-containing soap products having reduced tendency to ball.

It is a particular object to produce built particulate silicate-containing soap products that are substantially free of balling tendencies.

It is another object to produce built particulate silicate-containing soap products that are substantially free of balling tendencies without adversely affecting the dissolving, cleansing and sudsing performance of the soap.

Other objects of this invention will be apparent in the course of this description.

I have discovered that the above objects can be achieved by applying to the surface of the soap particles a minor amount of fatty acid having from 8 to 14 carbon atoms in the molecule, or mixtures consisting predominantly of such fatty acids, and thereafter effecting reaction between the fatty acid and silicate at the particle surface. I have further discovered that of the fatty acids thus employed the saturated C₈–C₁₀ fatty acids appear to be the most effective and that mixtures of fatty acids of oils of the coconut group as defined below and which contain appreciable amounts of saturated C₈ to C₁₄ fatty acids give excellent results.

In the practice of my invention, the application of the fatty acid to the particle surface can be practiced in numerous ways, as for example, by spraying the fatty acids onto the silicate-containing soap particles while the latter are agitated to insure relatively uniform distribution on the majority of the particles. The application can also

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be effected by passing the soap particles through a mist of the fatty acids. Numerous other methods will become apparent to those skilled in the art during the course of this description.

The unusual effectiveness of this process in reducing balling tendencies appears to be achieved by a mixture of reaction products of the fatty acids with silicates at the surface of the particles, which I shall call soap-silicate complexes, which possess outstanding effectiveness in the reduction of balling tendencies. I have found that my invention is particularly effective in the treatment of soaps which contain 1½% or more of sodium silicate. Under the microscope this complex looks like a thin crust on the soap particle. It has outstanding properties with regard to preventing the formation of gels that cause balling when the particle is contacted with warm water. It appears that the beneficial action of this crust-like complex is in part due to a combination of factors resulting from (1) the removal of Me₂O from the silicate molecules by the fatty acid to form C₈ to C₁₄ soaps and (2) the resulting high SiO₂/Me₂O ratio in the silicate portion of the soap-silicate complex formed thereby, wherein "Me" is used to designate an alkali metal.

My invention is of value in that it lowers the amount of oils of the coconut oil group that would be otherwise required to manufacture soaps of like balling tendencies. Thus I have found in general that in the treatment of silicate-containing soap granules with fatty acids derived from coconut oil, each percent of the fatty acids in the range of 1–5% used according to my invention effects an improvement in balling equivalent to that obtained by increasing the coconut oil content about 7 to 10% in the fat mixture used for preparing the soap. This enables the manufacturer to greatly reduce his overall usage of coconut type oils, and is of particular economic value when the supply of coconut type oils is short as during war time.

By the term "oils of the coconut oil group," I mean vegetable seed oils or fats at least 50% by weight of the combined fatty acids of which are lauric and/or myristic acids. These oils (many examples of which are given in Hilditch's "The Chemical Constitution of Natural Fats," 2nd Edition (1947), pages 198–205) are commonly derived from the seed of members of the botanical families Lauraceae (Tangkallak kernel oil, for example), Myristicaceae (Ucahuba nut oil for example), Vochysiacae (Jaboty kernel oil for example), Salvadoraceae (Khakan kernel oil for example), and more especially the Palmae family. Coconut oil is the outstanding example of an oil derived from seeds of the Palmae family, but other non-limiting examples of such oils are oils of murumuru kernel, tucuma kernel, cohune nut, ouricoury nut, babassu kernel and palm kernel.

It is to be understood that the specific fatty acids used in the process of my invention need not originate in the above mentioned oils; fatty acids from other sources may also be used, such as for example, synthetic fatty acids prepared by oxidation of petroleum or by hydrogenation of carbon monoxide by the Fischer-Tropsch process, which contain appreciable amounts of C₈ to C₁₄ fatty acids.

The following "balling test" has been used in evaluating the improvement effected by employing the process of the present invention:

Balling test

180 cc. of the particulate soap under test were dropped through a funnel having a ¾ inch diameter bottom opening located 9 inches above the surface of ¾ gallon of water in a pan having a diameter of 9¾ inches, the water having a temperature of 120° F., after which the mixture was allowed to stand undisturbed for 10 seconds.

Dilution and agitation of the mixture was then effected by directing a stream of water (temperature—120° F.) through a nozzle (consisting of a Bunsen burner wing tip having an orifice $\frac{1}{32}$ inches wide and $1\frac{1}{8}$ inches long) toward the mixture in the pan. The nozzle formed a sheet of water which hit the soap-water mixture at an angle of about 45° with the vertical and along a line radial from the center of the pan, thereby causing a swirling action in the water. In this manner, 1 gallon of water was added over a period of 40 seconds. The final mixture of soap and water was then poured onto a tared 20 mesh screen. The gummy mass of balled soap granules which was retained on the screen was weighed and the weight recorded in grams as the "balling grade." Such balling grades are determined on badly balling soap products will run from about 100 gms. to about 160 gms., whereas soaps that are considered to be substantially free of balling will have a rating of about 30 gms. and under when evaluated according to this test.

The invention will be more clearly understood from the following examples in which parts and percentages are by weight.

Example 1.—Neat soap was prepared by customary saponification with caustic soda of a glyceride mixture consisting of 27% coconut oil and 73% of a mixture of beef tallow and hog grease. This soap was crutched with builders and spray dried in conventional manner (i. e. dried by spraying the crutched soap in particulate form in a stream of hot air) to give a granular product containing 74% real soap, 11% sodium silicate solids having a $\text{SiO}_2/\text{Na}_2\text{O}$ ratio of about 2.6/1, 6% tetrasodium pyrophosphate and 8% moisture. Its bulk density was about 0.35 gm./cc. 98 parts of these granules were treated by spraying 2 parts of mixed fatty acids, obtained by the hydrolysis of coconut oil, upon the surface of the granules while the granules were agitated by tumbling in a rotating drum. The temperature of the soap granules during the latter step was maintained at approximately 100° F. and the temperature of the fatty acids sprayed thereon was about 120° F. The application of the fatty acids at the start caused the granules to have a soft mealy and somewhat tacky nature and the bulk density decreased. However, this change was strictly temporary and after 20 minutes' storage time without substantial change in temperature, during which period the fatty acids reacted with the silicate at the surface of the particles to form the soap-silicate complex, the density had increased to substantially its former value. The soft mealy feel had also disappeared and the product was actually less tacky and more free flowing than it was before being sprayed with the fatty acids. The table below shows the marked improvement produced in the balling grade of the product, wherein the results of three separate operations, each conducted as described above, are shown.

Samples of the treated and untreated soap granules were subjected to the above described balling test with the following results.

Run No.	Balling Grades of Granules	
	Untreated, grams	Treated with Fatty Acids, grams
1.....	46	17
2.....	43	16
3.....	55	15

The balling grades of the treated soap granules in this instance were as good as those normally obtained with untreated granules produced from similarly built soaps made from oils containing from 40% to about 45% coconut oil.

Example 2.—A neat soap composed of the sodium salts

of the fatty acids derived from a mixture of 16% coconut oil and 84% of tallow and grease was crutched with builders and spray dried in conventional manner to give a granular product containing 62% real soap, 18% sodium silicate solids having a $\text{SiO}_2/\text{Na}_2\text{O}$ ratio of about 2.5/1, 6% tetrasodium pyrophosphate and 13% moisture. The granules had a bulk density of 0.41 gms. per cc. 96 parts of these granules were treated by spraying 4 parts of molten (temperature about 125° F.) mixed fatty acids (obtained by the hydrolysis of coconut oil) upon the surface of the granules while the granules were tumbled in a rotating drum. The temperature of the granules during the latter step was on the order of 100° F. As an immediate result of the fatty acid application, the granules decreased about 0.06 gm./cc. in bulk density and had a soft, tacky feel. However, after 15 minutes' reaction time during which period there was substantially no change in the temperature, the density of the granules was again about 0.41 gms./cc. and the granules were less dusty and more free flowing than prior to the application of the fatty acid.

Samples of the treated and untreated soap granules were subjected to the balling test. The table below shows the results of two runs made in accordance with the example.

Run No.	Balling Grades of Granules	
	Untreated, grams	Treated with Fatty Acids, grams
1.....	105	32
2.....	93	35

Example 3.—A neat soap composed of the sodium salts of the fatty acids derived from a mixture of 3% coconut oil and 97% of tallow and grease was crutched with builders and spray dried in conventional manner to give a granular product containing 62% real soap, 18% silicate solids having a $\text{SiO}_2/\text{Na}_2\text{O}$ ratio of about 2.6/1, 6% tetrasodium pyrophosphate and 13% moisture. The granules had a bulk density of 0.41 gm./cc. 96 parts of these granules were treated by spraying 4 parts of molten (temperature about 100° F.) mixed fatty acids (obtained by the hydrolysis of coconut oil) upon the granules while the granules were tumbled in a rotating drum. The temperature of the granules during the latter step was on the order of 95° F. As an immediate result of the spraying process, the bulk density of the granules decreased about 0.06 gms./cc. and the granules had a soft, mealy appearance. However, after 15 minutes' storage without substantial change in temperature, the density had returned to 0.40 gms./cc. and the granules were less dusty, tougher, and more free flowing than they were before being sprayed with fatty acids.

Samples of the treated and untreated soap granules were subjected to the balling test. The table below shows the results of two runs made in accordance with the example.

Run No.	Balling Grades of Granules	
	Untreated, gms.	Treated with Fatty Acids, gms.
1.....	123	25
2.....	110	42

Essentially the same results can be obtained when the granules of the above examples are sprayed with fatty acids derived from palm kernel oil.

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I have also found that a mixture of lower fatty acids as may be derived by fractionating fatty acids of oils of the coconut oil group, are particularly effective in reducing the balling tendencies. C₁₀ fatty acids alone are also outstandingly effective as is shown in the following example.

Example 4.—Soap granules were prepared in the same manner as described in Example 3 except that 3 parts of molten capric acid (a C₁₀ fatty acid) was used instead of coconut fatty acids for treating 97 parts of the soap granules at room temperature. The following data show the very marked improvement in balling grades, a substantially non-balling product being obtained.

Run No.	Balling Grades of Granules	
	Untreated, gms.	Treated with C ₁₀ Acid, gms.
1	132	17
2	132	17

Attention is called to the fact that by the reaction of the fatty acids with silicate at the surface of the soap particles, and the resultant formation of high SiO₂/Na₂O ratio silicate-soap complexes, a material having detergent and sudsing power is formed, thereby avoiding the detrimental effect characteristic of earlier processes wherein the soaps were merely coated with inert materials or even materials that repressed the sudsing and detergent properties of the soap.

It is the saturated fatty acids containing from 8 to 14 carbon atoms that are particularly useful in my process. Whereas the mixed fatty acids obtained from oils of the coconut oil group contain minor amounts of unsaturated fatty acids, such as oleic and linoleic, the main value of these unsaturated fatty acids lies in their low melting points and their liquifying or solvent effect on the higher melting saturated fatty acids. Highly unsaturated and high molecular weight fatty acid mixtures, such as commercial oleic acid (commonly known as red oil) and the mixed fatty acids derived from cottonseed oil, are practically ineffective as compared to the fatty acid mixtures derived from oils of the coconut oil group for the purposes of my invention.

It is to be understood that I contemplate the use of other builders along with the water-soluble silicates in the practice of my invention. Examples of such other soap builders are the water-soluble pyrophosphates (e. g. the sodium pyrophosphate used in the above examples), carbonates, orthophosphates, tripolyphosphates, borates, etc. Whereas the more alkaline of such builders may also neutralize some of the fatty acids applied in this process the soaps produced therewith have not been found to detract from the benefits obtained with the soap-silicate complex.

In the practice of this invention, I prefer to employ soap compositions containing at least 1½% by weight of silicate solids, which may be derived from a wide variety of water-soluble silicates. Sodium silicates having SiO₂/Na₂O ratios varying from 2.0/1 to 4.0/1 can be used in the preparation of soap granules treated in this invention. In general, however, I prefer to employ silicates having ratios in the range of 2.3/1 to 3.3/1. I may also employ alkaline materials such as caustic soda or soda ash in conjunction with the silicate to modify the ratio of the silicates. Sometimes the builder contents of commercial soap preparations are composed entirely of silicate solids but the silicate solids usually do not exceed about 50% of the weight of the soap product.

I usually prefer to treat the soap particles with amounts of fatty acids in the range of 2–5% of the weight of

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the soap particle. In this range optimum benefits are obtained. However, smaller amounts can be used to advantage, as for example, the use of 1% fatty acids is often desirable, particularly in soap containing small amounts of silicate. Although the reduction in balling obtained per unit of fatty acid is somewhat less when about 7% or more fatty acid is used, larger amounts, as for example 8 to 9%, can be applied successfully in some instances, particularly when the soap is built with large amounts of silicate and when the temperature of the granules is in the neighborhood of or below the melting point of the fatty acids. However, when low temperatures are used the efficiency of the process may be somewhat reduced since the reaction of the fatty acids with the silicate in the soap granule is retarded; however, even under such conditions a marked improvement in balling is observed in the treated granules.

In the practice of this invention, the fatty acids can also be mixed in powdered form with the soap granules. However, in so doing or when molten fatty acids are applied to cold soap particles, e. g. at temperatures below 50° F., the temperature of the soap particles is preferably increased at some point in the process so that the fatty acids will melt and be absorbed at the surface of the particles to insure reaction with the silicate. If the use of low temperatures of application are desirable the fatty acids can be sprayed onto the particles in solution in solvents such as ethyl alcohol, thereby promoting absorption and subsequent reaction. In other words in order to obtain the full benefits of my invention it is highly desirable to have the fatty acids in a fluid or liquid condition during or at least at the start of their reaction with the silicate in the soap; i. e. either at a temperature high enough to liquify the fatty acids with or without the aid of solvents or other liquifying media or compositions. The mixed fatty acids from coconut oil, for example, are in a liquid form at 75° F., even though some of the fatty acids contained therein are normally solid in the pure form at that temperature.

In general to facilitate the operation I prefer to carry out my process in a manner such that the granules to which the fatty acids have been applied will not drop substantially in temperature during the subsequent reaction period. In such cases I prefer to spray the fatty acids in molten form (preferably over 80° F.) onto soap particles having a temperature of at least 50° F., and preferably over 80° F., while tumbling the soap particles in a rotating drum, followed by storing the soap particles in bulk, as for example in large buggies or bins. The period of time consumed in normal operations such as screening, weighing, and transferring the product to and from storage in addition to the storage period is usually many times greater than the few minutes in which substantially complete reaction of the fatty acids and silicate occurs. Operating in this manner, and by keeping the temperature below the point at which the product begins to soften or melt (generally not over 115° F.), agitation need not be resorted to during the reaction-storage period for the purpose of avoiding lumping or caking. However, the fatty acids can be applied to hot particulate products such as freshly spray dried compositions leaving a spray drying tower, which sometimes have temperatures in the neighborhood of 200 to 210° F., under which conditions the fatty acids will react very rapidly, after which it is desirable to agitate and cool the granules to avoid caking and lumping.

The moisture content of commercial granular soap products is usually controlled within well defined ranges that vary with the type of product and according to the amount and types of builders included therein. I have found that within limits the benefits obtained with my process are increased as the product moisture is increased. Therefore, I prefer to maintain the moisture on the high side of the range, but yet low enough so that the product will not cake or lump during processing and packing

operations. Such optimum moistures can be about 4% for products low in builder content, whereas with more highly built products the optimum can be about 20% or even greater. In fact the process reduces balling tendencies in soap compositions containing as much as 30% moisture. If the untreated granules have been dried below the optimum moisture level, the reduction in balling tendencies can be improved by applying water or steam to the granules in addition to the fatty acids.

The wide variations in conditions of temperature, solvent and moisture level can cause the time of reaction to vary considerably. While I prefer to use conditions such that the reaction will be substantially complete within a few minutes so as to facilitate control of the process, it is obvious that under some conditions the reaction will approach completeness very slowly. In such cases some improvement in the product may well occur even after the product has been packed.

Not only is the rate of the reaction step affected by operating conditions but so also is the time at which it starts. Thus when the fatty acids are applied in the solid state, the start of the reaction step can conceivably be delayed for hours or days. However, seldom are such delays commercially desirable, and normally it is preferred to apply the fatty acids in liquid form on soap particles having a temperature above the melting point of the fatty acids. Under such conditions we find that the reaction, which follows the application of the initial part of the fatty acids to any one portion of the soap particles, is already well under way or indeed can be substantially complete by the time that the final part of the fatty acids is applied. Thus the time element of the second step compared to that of the first step can be controlled with regard to both rate and period by varying the operating conditions, particularly with regard to temperature.

The depth to which the fatty acids penetrate into the surface portion of the soap particles is variable. Obviously, it can vary with the amount of fatty acid used, temperature of soap, silicate content and density of the soap particles. Furthermore, the rate at which it is neutralized will depend in part on the amount of silicate in the soap and the temperature employed.

The process of this invention can be used for particulate soap products in general that show a tendency to ball. Thus, for example, very marked improvement is obtained with regard to balling in ground products such as ground soap flakes, chips or threads that have balling tendencies.

It is to be understood that the foregoing more particularly described processes are to be considered as illustrative of preferred methods only; such changes and modifications therein are contemplated as would normally occur to those skilled in the art to which the invention relates.

Having thus described my invention, what I claim and desire to secure by Letters Patent is:

1. In the process of reducing the balling tendencies of particulate compositions consisting essentially of sodium soaps containing insufficient soaps of saturated fatty acids having 8 to 14 carbon atoms to prevent balling of the particles and an amount of sodium silicate solids not less than 1½% and not greater than 50% of said composition and uniformly distributed therein, the steps of applying to the particles from 1% to about 7% of a saturated fatty acid having 8 to 14 carbon atoms and reacting said fatty acid in fluid condition with silicate solids at the surface of said particles.

2. The process of claim 1 wherein the soap particles are treated with from 2 to 5% of the fatty acid.

3. The process of claim 1 wherein the soap particles are treated by spraying the fatty acid on the soap particles, while maintaining the temperature of the particles within a range of from 50–115° F., and maintaining the moisture of the particles within a range of 4 to 20%.

4. The process of claim 1 wherein the fatty acid is a

mixture of fatty acids derived from an oil of the coconut oil group.

5. The process of claim 4 wherein the fatty acids are the mixed fatty acids of coconut oil.

6. The process of claim 4 wherein the fatty acids are the mixed fatty acids of palm kernel oil.

7. The process of claim 1 wherein the applied fatty acid is C₁₀ fatty acid.

8. A particulate soap product consisting of an inner portion of a soap composition consisting essentially of sodium soaps containing insufficient soaps of saturated fatty acids having 8 to 14 carbon atoms to prevent balling and at least 1½% and not more than 50% of sodium silicate uniformly distributed therein, and a crust portion comprising the reaction products of silicate and applied saturated fatty acid having from 8 to 14 carbon atoms in the molecule, the amount of said fatty acid being equal to from 1% to about 7% of the weight of the soap particle.

9. The product of claim 8 wherein said fatty acid is a mixture of fatty acids derived from an oil of the coconut oil group.

10. The product of claim 8 wherein said fatty acid is a mixture of fatty acids derived from coconut oil.

11. The product of claim 8 wherein said fatty acid is a mixture of fatty acids derived from palm kernel oil.

12. The product of claim 8 wherein the amount of said fatty acids is from 2 to 5% of the weight of the soap particle.

13. A particulate soap product consisting of an inner portion of a soap composition consisting essentially of sodium soaps containing insufficient soaps of saturated fatty acids having 8 to 14 carbon atoms to prevent balling and at least 1½% and not more than 50% of sodium silicate uniformly distributed therein, and a crust portion comprising the reaction products of silicate and saturated fatty acids having from 8 to 14 carbon atoms in the molecule, the amount of said fatty acid being equal to from 1% to about 7% of the weight of the soap particle, the SiO₂/Na₂O ratio of the silicate in the crust portion being greater than that of said inner portion.

14. In the process of reducing balling tendencies in soap particles consisting essentially of sodium soaps containing insufficient soaps of saturated fatty acids having 8 to 14 carbon atoms to prevent balling and at least 1½% and not more than about 50% sodium silicate solids uniformly distributed therein, the steps of treating the surface of the soap particles at temperatures of from 50° F. to 115° F. with from 2 to 5% by weight of fatty acids derived from coconut oil, the moisture content of said soap particles being in the range of 4 to 20%; and reacting the fatty acids with the silicate in the surface of said particles.

15. A particulate soap product consisting of an inner portion of a soap composition consisting essentially of sodium soaps containing insufficient soaps of saturated fatty acids having 8 to 14 carbon atoms to prevent balling and at least 1½% and not more than 50% of sodium silicate uniformly distributed therein and a crust portion comprising the reaction product of mixed fatty acids derived from an oil of the coconut oil group and silicate in the surface portion of the soap particle, the amount of fatty acids being equal to from 2 to 5% of the weight of the soap particle.

16. In the process of reducing balling tendencies in soap particles containing substantially 1½% to 50% sodium silicate solids, the steps of treating the surface of the soap particles at temperatures from 50° F. to 115° F. with from 1 to about 7% by weight of saturated mixed fatty acids derived from oils of the coconut oil group, reacting the fatty acids with the silicate in the surface of said particles and continuing the reaction under bulk storage conditions.

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