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PROCESS OF PRODUCING A DETERGENT COMPOSITION

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This invention relates to new detergent compositions comprising alkyl sulfate salts. It also deals with a method of improving the detergency of water-soluble salts of alkyl sulfuric acid esters having 10 to 25 carbon atoms per molecule by admixing therewith under controlled conditions salts of higher fatty acids.

Alkyl sulfuric acid ester salts such as may be derived from fatty alcohols or olefins having 10 to 25 carbons per molecule are known to have certain advantages over ordinary soaps, particularly with respect to lime-resistance and wetting power. However, the alkyl sulfate salts as a class are more or less inferior to the fatty acid soaps in regard to detergency. An important object of the present invention is to overcome this disadvantage of prior alkyl sulfate salt compositions and to produce a new detergent which will not only have the advantageous lime-resistance and wetting power of the alkyl sulfate salts but also will have improved detergent properties.

It has been found that compositions having the foregoing desirable combination of properties can be obtained from certain properly prepared mixtures of alkyl sulfate salts and fatty acid salts wherein the fatty acid content is at most equal to, and more preferably is considerably lower than, the alkyl sulfate salt content. Simple mixing of the alkyl sulfate salt and fatty acid soap, as by grinding together or otherwise intermingling these two components in preformed conditions, is not a suitable method of preparation of the improved compositions. The fatty acid soap must be formed in an aqueous medium containing the higher alkyl sulfate salt in order to obtain products of most desirable detergent power, lime-resistance and wetting characteristics. Products prepared in this manner, namely, formation of the fatty acid soap in situ in an aqueous alkyl sulfate salt solution, have a different colloid-chemical structure from those obtained by later mixing of the alkyl sulfate salt with a previously prepared fatty acid soap. Whatever the true explanation, the new compositions have been found to possess a combination of properties which is superior to those obtainable with other mixtures comprising alkyl sulfate salts.

The process is preferably carried out by mixing an aqueous solution of, for example, 20% to 25% of the alkyl sulfate salt with the desired quantity of one or more higher fatty acids, i. e. fatty acids containing at least 12 carbon atoms, and preferably about 14 to 20 carbon atoms, to the molecule, and neutralizing the mixture thus obtained by the addition of a base or some other basic substance. Instead of free fatty acids one may also take oils or fats, i. e. fatty acid glycerides, and convert the latter into fatty acid soap

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by means of a saponification agent after mixing them with the alkyl sulfate salt in an aqueous medium.

The fatty acids, or the oils or fats, may be of any vegetable or animal origin. The higher fatty acids prepared synthetically, for example by oxidation of paraffin wax, may also be used. Especially fatty acids or fatty acid mixtures with a comparatively high melting point, such as palmitic acid, stearic acid, and palm oil fatty acids, and also oils or fats rich in these fatty acids, such as palm oil, tallow, bone fat, partially hardened linseed oil or train-oil and the like, have been found highly suitable.

Although the preparation of the products according to the invention, if fatty acids are started from, may take place at room temperature, it is preferably carried out at moderately elevated temperature, for example from about 50° C. to 70° C., as this promotes a ready mixing of the fatty acids with the aqueous solution of the alkyl sulfate salt as well as a smoother progress of the neutralization. If oils or fats are employed, the process should take place at a sufficiently elevated temperature to cause the saponification of the glycerides; in general this makes it necessary to apply temperatures above 70° C., e. g. from about 90° C. to 100° C.

The quantity of fatty acids, oils or fats used in the preparation of the products according to the invention may vary within wide limits. However, it is not allowed to use such large quantities as to make the content of fatty acid exceed that of the alkyl sulfate salt in the final product, because this gives rise to products with unsatisfactory properties, particularly as regards lime-resistance.

It has been found that even with a relatively small weight ratio between fatty acid and alkyl sulfate salt in the final product, for example below 30:100, compositions with substantially improved properties, particularly as to detergent power, may be obtained by the process according to the invention, without an increase of the said ratio involving any further improvement. In general, the said ratio amounts to at least about 5:100, since no appreciable improvement is generally attained below this ratio. The quantity of fatty acids which, starting from a given alkyl sulfate salt, is required for the preparation of products with the highest possible detergent power, while preserving as much as possible the original great lime-resistance and the excellent wetting power, depends to some extent on the nature of the fatty acids and of the alkyl sulfate salt, and also on the consistency of the final product desired. A ratio of fatty acid to alkyl sulfate salt in the final product lying be-

tween about 10:90 and about 35:65 yields products with particularly favorable properties, which generally have the consistency of soft soap. For the preparation of hard products it is usual to apply higher ratios, for example from about 40:60 up to about 50:50. These products possess better detergent properties than the alkyl sulfate salts alone, while their lime-resistance and wetting power not only have not, or substantially not, been impaired compared with the alkyl sulfate salts alone but also, in the case of products with a low fatty acid content, for example 10% or less, the lime-resistance is even increased slightly.

The higher secondary sulfuric acid alkyl ester salts of 10 to 18 carbon atoms per molecule such, for example, as may be produced by sulfating olefins and neutralizing the resulting esters with bases which yield water-soluble alkyl sulfate salts as described in U. S. Patent 2,152,292, for instance, are especially advantageous starting materials for the preparation of the new compositions of the invention. However, primary sulfuric acid alkyl ester salts such as sodium lauryl sulfate, ammonium myristyl sulfate, etc. which may be produced as described in U. S. Patent 1,968,794, for example, or tertiary sulfuric acid alkyl ester salts such as are disclosed in U. S. Patent 2,139,394, for instance, may also be used in the process of the invention.

When using the preferred secondary sulfuric acid alkyl ester salts, the process is preferably carried out with such a quantity of fatty acid or fatty acid glyceride that the ratio of fatty acid to alkyl sulfate salt is about 15:85 to 20:80. In this manner it is possible to obtain products resembling soft soap, which are characterized by particularly good properties.

The fatty acids or fatty acid glycerides used in the process according to the invention as a rule are completely converted into fatty acid soaps. If desired, however, it is also possible to apply only a partial neutralization or saponification, in which case the final product will contain part of the fatty acids or glycerides in unconverted condition.

The neutralization of the fatty acids or saponification of the fatty acid glycerides may be carried out with any suitable inorganic or organic base or other basic substance, such as sodium or potassium hydroxide or carbonate, or triethanol amine or other alkylol amines. The fatty acid soap may thus be derived from the same base as the alkyl sulfate salt or from a different base. The amount of the basic compound will be an amount of from the equivalent of about 5% by weight of the fatty acid soap based upon the total amount of the alkyl sulfate salt and fatty acid soap to the substantially stoichiometric equivalent of the fatty acid or glyceride.

The products obtained according to the invention may be employed without undergoing any further treatment. If desired, they may also be subjected to an additional treatment; thus, it is, for example, possible to remove a portion of the water, e. g. by evaporation, in order to give them the desired consistency, or the products may be worked up to dry powders according to the roller-drying process or by atomization-drying.

It was further found that the properties of the capillary-active products prepared in the above manner, in particular their detergent power, may be enhanced even further by combining them with water-soluble alkaline-reacting inorganic salts, such as alkali silicates, phosphates, borates

and carbonates. Especially, the addition of silicates, in particular sodium metasilicate and waterglass or mixtures thereof, was found to exert a particularly strong influence, which not only gave rise to improved detergent power, but remarkably enough also to increased lime-resistance. The mixtures thus produced, the lime-resistance of which may even considerably exceed that of the preferred alkyl sulfate salt, which in itself is already highly lime-resistant, are exceptionally suitable to be used for washing textile materials, as in this case the risk of deposition of calcium salts on the fibers, especially during the rinsing process, is greatly reduced.

In general, additions of, for example, 10% of the alkaline salts, calculated on the combined quantity of alkyl sulfate salt and fatty acid soap, already bring about a distinct increase of the lime-resistance and of the detergent power. When the quantities of the salts added are increased, the effect increases up to a maximum, while after continued additions the properties of the products deteriorate again. In view of this, additions of more than 50% of the said salts are generally not to be used.

When silicates were added, the maximum effect, both as regards increased lime-resistance and with respect to the detergent effect, was usually found to be attained by the use of about 20% to 30% silicate.

The addition of sodium carbonate or other carbonates, or of phosphates or borates, as alkaline salts, has a similar effect to that of silicates, though less pronounced. It is often less suitable to add large quantities of sodium carbonate, because this may readily cause crystallization of the sodium carbonate from the products after some time, which is highly undesirable since it is detrimental to the appearance of the product.

To the products prepared according to the invention may be added, if desired, soluble cellulose derivatives, for example methoxy cellulose or any of the fillers, colorants or scents usually added to detergents. Whether or not such additives are employed, the products are eminently suitable for use in laundries and further for domestic use in general. They may also be employed for other purposes, e. g. in the textile industry.

The process according to the invention is of particular importance for the preparation of unctuous products resembling soft soap, in which case the fatty acid soap formed during the preparation may be sodium soap, such in contrast with the ordinary soft soap, which is known invariably to consist of fatty acid potassium soap. When compared with soft soap, entirely based on fatty acid soap, these products of the invention display not only a substantially greater resistance to calcium ions and acid, and increased wetting power, but also a more favorable lathering effect, especially in hard water. Besides, they are superior to the ordinary soft soap in imparting no unpleasant smell to the clothes washed with them.

However, the process is not restricted to the preparation of products with the consistency of soft soap, but may also be applied to the preparation of products which are hard at ordinary temperatures.

As a rule it is to be recommended to keep the products at room temperature for some time after their preparation, e. g. a few days, so as to obtain mature products with a satisfactory consistency before applying them to their intended use.

The invention is further illustrated with ref-

erence to the following examples of the preparation of improved products, without, however, being restricted thereto.

Example I

Three hundred and twenty parts by weight of a 25% aqueous solution of secondary sodium alkyl sulfates, produced by neutralization of the sulfation products of a C_{10}–C_{18} alkene cracking fraction, are mixed with 20 parts by weight bleached palm oil fatty acids (titer 44° C.) at a temperature of about 60° C. After this, sufficient 25% sodium hydroxide lye is added, while stirring, to neutralize the fatty acids completely, and subsequently 23 parts by weight sodium metasilicate ($Na_2SiO_3 \cdot 9H_2O$) and 27 parts by weight waterglass of 38° Bé. The resulting mixture is then evaporated at 95° C. to about 275 parts by weight and then cooled down to room temperature, after which it is left to stand at this temperature for another 48 hours approximately. The product thus obtained, the consistency of which resembles that of soft soap, has a particularly great lime-resistance, which even exceeds that of the secondary sodium alkyl sulfate solution from which it was derived, and very high wetting power, while the detergent effect also exceeds that of the secondary sodium alkyl sulfate solution. The product constitutes an excellent detergent, particularly suitable for washing at comparatively low temperatures.

Example II

One hundred and sixty parts by weight of the same alkyl sulfate salt solution as used in Example I are mixed at about 60° C. with 17 parts by weight bleached palm oil fatty acids (titer 44° C.), upon which about 10 parts by weight 25% sodium hydroxide lye are added, while stirring, in order to neutralize the fatty acids. After this, 45 parts by weight sodium metasilicate are added, upon which the mixture is cooled and left to stand at room temperature for about 48 hours. About 230 parts by weight of a product resembling soft soap and having excellent properties were thus obtained, which product was eminently suitable to be used for washing at any temperature usually applied.

Sodium lauryl sulfate may be used in place of the secondary alkyl sulfate salt with good results.

Example III

Two hundred parts by weight of the alkyl sulfate salt solution of the preceding examples are mixed, while stirring vigorously, at about 90° C. with 50 parts by weight tallow (animal fat, titer 40.2° C.), upon which the fat is saponified with 31 parts by weight 25% sodium hydroxide lye. After completion of the saponification the mixture is cooled and left to stand at room temperature for about 48 hours. The product thus obtained is sufficiently hard to be cut. The originally sticky surface of the bars hardens after a few hours. A highly neutral hard soap is obtained, which has an excellent detergent effect at any temperature. After the saponification, 50 parts by weight tetra sodium pyrophosphate may, if desired, be added to the mixture obtained.

Example IV

One hundred parts by weight of the 25% alkyl sulfate salt solution employed in the preceding examples are mixed at 60° C. with 25 parts by weight bleached palm oil fatty acids (titer 44° C.) After this, 10 parts by weight triethanol amine, corresponding to about two-thirds of the quantity theoretically required, are added, as a result of which the fatty acids are partially neutralized. After cooling and keeping at room temperature, a comparatively hard product is obtained, which makes an excellent shaving soap, hard enough to be used as so-called cup-soap.

I claim as my invention:

1. A process of producing a detergent of improved lime-resistance and detergent and wetting powers which comprises intimately mixing an aqueous solution of a water-soluble salt of a secondary alkyl sulfuric acid ester having 10 to 25 carbon atoms per molecule with a fatty acid of 12 to 20 carbon atoms per molecule in an amount such that the weight ratio of said acid to said secondary alkyl sulfate salt is 5:100 to 50:50, adding to the mixture sodium hydroxide in an amount of from the equivalent of about 5% by weight of the sodium soap of the fatty acid based upon the total amount of said sodium soap and said secondary alkyl sulfate salt to the substantially stoichiometric equivalent of said fatty acid and maintaining the mixture at saponification temperature to effect saponification of said fatty acid.

2. A process of producing a detergent which comprises intimately mixing an aqueous solution of a water-soluble salt of a secondary alkyl sulfuric acid ester having 10 to 25 carbon atoms per molecule with a higher fatty acid glyceride in an amount equivalent to a fatty acid to secondary alkyl sulfate salt ratio of 10:90 to 35:65, adding to the mixture an alkali metal hydroxide which forms water-soluble soap and heating to a temperature of 70° C. to 100° C. to effect saponification of said glyceride, the alkali metal hydroxide being added in an amount sufficient to form a water-soluble soap.

3. A process of producing a detergent which comprises intimately mixing an aqueous solution of a water-soluble salt of an alkyl sulfuric acid ester having 10 to 25 carbon atoms per molecule with a fatty compound of the group consisting of soap-forming higher fatty acids and glycerides thereof in an amount equivalent to a weight ratio of fatty acid to said alkyl sulfate salt of 5:100 to 50:50, adding to the mixture a basic compound, the soap of which with the fatty acid of said fatty compound is water-soluble, in an amount of from the equivalent of about 5% by weight of said soap based upon the total amount of said soap and said alkyl sulfate salt to the substantially stoichiometric equivalent of the fatty acid and maintaining the mixture at saponification temperature to effect saponification.

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