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PREPARATION OF NONDUSTING ORGANIC
DETERGENT COMPOSITIONS

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1

This invention relates to the preparation of
non-dusting organic detergents, other than soap,
and particularly to the method of preparing non-
dusting organic compounds, having a non-car-
boxylic acid salt group, in granular, bead, flake, 5
chip, powder or like forms.

In recent years numerous new synthetic deter-
gents have been developed as substitutes for fat-
ty acid soaps, which novel materials have a great
many advantages for special purposes over the
present-day soaps. However, many of these novel
materials also have properties which are not
wholly desirable in preparing marketable prod-
ucts in competition with the fatty acid soaps.

One of these properties is the tendency of many
of these products, when it is attempted to pro-
duce them as flakes, grains, hollow beads, ribbons
or chips, to form a fine dust. It has been found
that the formation of such a dust is a serious
disadvantage in the marketing of the newer deter-
gents since the fine dust sifts from its package
with a consequent loss. Furthermore, because of
the small particle size, the dust remains suspended
in the air when the detergent is poured from
the container, thus causing an unpleasant irrita-
tion of the nose and eyes of the housewife or other
user of the product. In the case of the more in-
flammable materials, the possibility of fires or
dust explosions is likewise increased. In addi-
tion, the products are non-uniform in size which
causes difficulty in dispensing and handling in
general. When the detergent products are too
finely divided they tend to cake and form gels
with water and they do not dissolve as readily as
beads of the same products. These undesirable
results have lessened the sales appeal of the dust-
containing product.

Furthermore, if the material is formed into
grains or similar particles, the proportion of fines
is very high and these fines must be screened from
the product and reworked. This entails addi-
tional expense and causes further breakdown and
dusting of the larger particles during the screen-
ing treatment.

Attempts to overcome this dusting have in-
cluded the hydration of the organic detergents,
but the results proved to be unsatisfactory. If
sufficient water is present to appreciably over-
come the dusting of the detergent material, then
the latter will generally be tacky or sticky and
tend to ball or gum up when produced in the forms
having at least one dimension relatively small.

It has now been discovered that normally-dust-
ing, solid organic detergents having an inorganic
acid salt radical in the molecule, which agents 55

2

usually do not contain free hydroxy groups in
their molecule, can be sprayed, rolled or extruded
to form substantially dry, non-dusting products
by incorporating in the organic detergents a hy-
droxy aliphatic compound boiling above 150° C.
and preferably a polyhydroxy compound of this
type.

It has been found that the incorporation of
such substances, preferably between about 1%
and about 10% based on total solids content, with
organic detergents such as salts of long-chain
alcohol sulphates and alkylated aromatic sul-
phonic acids having not more than about 3%
moisture in the final product, produces a com-
position which is especially adapted for econom-
ical sheeting on rolls, spray-drying and the like.
The products are more plastic and can be worked
readily without substantial dusting, crumbling,
and formation of fines. The final products are
not sticky and possess all the advantages of the
original detergent and in some cases improved
surface active properties.

The type of compounds which are preferably
incorporated with the organic detergents, either
alone or in any desired combination, are hydro-
philic, including water-soluble, polyhydric al-
iphatic substances such as glycerol, polyglycerols,
glyceroses, glyceramine, chlorhydrins, glyceric
acid; monoglycerides of carboxylic acids including
the long and short-chain fatty acids, and naph-
thenic acids; glycerol mono-sulphate salts; gly-
cerol monoethers; polyglycerol ethers and esters
having at least two free hydroxy groups; pentan-
triol 1,2,5 and the monoethers and monoesters
(organic and inorganic) thereof; glycols includ-
ing ethylene glycol, pentan-diol 1,2, pentan-diol
1,5, octadecan-diol from hydrogenated ricinoleic
acid, polyethyleneglycols and other polyglycols;
aliphatic polyhydric alcohols having more than
three hydroxy groups in the molecule including
erythritol, arabitol, mannitol, sorbitol; the water-
soluble aldehyde, ketone, halide, ether, acid and
amine derivatives of these alcohols as well as the
water-soluble partial esters and ethers thereof in-
cluding glucose, sorbitol monooctylate, glucose
monoacetate, sucrose, arabinose and fructose;
water-soluble gums and starches as well as their
water-soluble esters or ethers including gum
arabic, tragacanth; water-soluble cellulose ethers
such as methyl and ethyl celluloses; and water-
soluble polyvinyl alcohols and water soluble par-
tial ethers and esters thereof.

The organic detergents may be combined with
other agents which reduce dusting such as min-
eral oils, fatty oils, long chain aliphatic mono-

hydric alcohols, cyclohexanol, tetrahydrofurfuryl alcohol, decalin and like agents, but it is preferable that at least one agent of the class of water-soluble polyhydric aliphatic substances also be incorporated.

The substances, alone or in combination, which can be improved by this present method of preventing dusting include salts of the long-chain alkyl primary and secondary sulphates, phosphates and borates, such as the sodium salt of the dodecyl sulphate; salts of the monosulphate of glycol monoethers and monocarboxylic acid esters, such as the sodium salt of the sulphate of diethylene glycol monolaurate; salts of aromatic sulphonates including the alkylated derivatives thereof such as the sodium salt of hexyl naphthalene sulphonic acid, salts of diphenyl and long-chain alkylated diphenyl mono- and polysulphonic acids, and the neutralized products prepared by the sulphonation of aromatic mineral oil extracts in liquid sulphur dioxide; alkyl sulphonic acid salts including tetradecyl sulphonic acid sodium salt and aliphatic mineral oil sulphonate salts; salts of long-chain alkyl esters and long-chain alkyl amides of sulpho-acetic acid; the salts of monoethers and monocarboxylic acid esters of isethionic acid, and the salts of monocarboxylic acid amides of taurine. The tendency for these materials to dust is amplified if they contain inorganic salts, such as sodium carbonate, borax, sodium silicate, sodium phosphate or sodium sulphate, the last two salts often being formed in the product during the neutralization.

The following examples indicate some of the advantages obtainable by the present invention but they are not intended to limit the scope thereof.

Example I

One thousand parts of an aqueous preparation containing 200 parts by weight of the sodium salt of sulphated fatty alcohols obtained, by hydrogenation, from coconut oil, and 200 parts by weight of sodium sulphate are intimately mixed with twenty-five parts by weight of the monoglyceride of coconut oil fatty acids. The mixture is then spray-dried, by the method described in U. S. Patent 1,652,900, to form a uniformly granular product having only a small percentage of fines and substantially no tendency to dust.

If the same sulphate mixture is similarly treated without the addition of the monoglycerides, a large quantity of fines will be produced which are hard to collect and which must be sieved from the remainder of the product thus reducing the efficiency of the process. In addition the resulting product shows a marked tendency to dust and irritate the nose and eyes of the user.

Example II

An aqueous preparation containing 50 parts by weight of the sodium salts of sulphonated, long-chain alkylated phenols, 50 parts sodium sulphate, 10 parts of borax and 3 parts of glycerol were spray-dried in a tower with hot gases to form a finely divided product having little fines content. The glycerol prevents the formation of excessive quantities of fines and the product resists dusting.

The borax is added to the aromatic sulphonate material to be spray-dried for a particular purpose. It has been found that in the spray-drying of salts of aromatic sulphonic acids including the alkylated and other derivatives thereof, there is

a tendency for the materials to take fire, even when associated with the inorganic sulphates which are usually present in these products as a result of the neutralization of the excess of sulphonating agents. This problem appears to be unique with the sulphonated aromatic compounds since it is possible to spray-dry aliphatic sulphates such as monoglyceride monosulphates without fire hazard. This is most unusual since these sulphonates are considerably more stable than the surface active aliphatic sulphates and if flame is applied they will not burn freely and will not sustain combustion when the flame is removed, whereas the aliphatic sulphates, on the other hand, with or without inorganic sulphates will burn more freely and will, in some instances, sustain combustion even when the flame is removed.

It is a feature of this invention that the presence of alkaline builder materials of the type of borax, trisodium phosphate, and sodium silicate, during the spray-drying of salts of aromatic sulphonic acids as well as alkylated and other derivatives thereof, prevents these materials from taking fire and burning during the process.

It is also possible to add polyhydroxy alkyl and/or polyalkyl amines and/or ammonium bases to the normally dusting organic detergents for the purpose of preventing such dusting, and in the case of salts of aromatic sulphonic acids these substances serve the additional purpose of preventing fires. Triethanolamine, diethanolamine, glycerolamine, diglycerolamine, and triglycerolamine serve very well for this purpose.

Example III

To 600 parts by weight of an aqueous preparation containing 100 parts of a mixture of 37% sodium sulphate and 63% of the sodium salt of the sulphonic acids produced by sulphonating a refined fraction of a liquid sulphur dioxide extract of a Gulf Coast petroleum with fuming sulphuric acid while dissolved in liquid sulphur dioxide, there is added about 2.0 parts of triethanolamine and 2.0 parts glycerol. The detergent composition is dried on heated rolls to a powder with little dusting and substantially no gumming. If the quantities of triethanolamine and glycerol are doubled, practically all dusting is absent.

Example IV

Two hundred and seventy-five parts of an aqueous preparation containing 2 parts by weight of glycerol, 6 parts by weight of triethanolamine, and 100 parts by weight of a mixture of about 40% sodium sulphate and about 60% of the sodium salts of the aromatic sulphonic acids produced by sulphonating a liquid sulphur dioxide extract of a Borneo petroleum with fuming sulphuric acid while dissolved in liquid sulphur dioxide, is spray-dried with heated air in a spray tower without any fire hazard to yield a relatively uniform product with little fines content and substantially no tendency to dust.

Example V

A preparation containing 500 parts by weight of water, 8 parts by weight of the monoglycerides of coconut oil fatty acids, 100 parts by weight of the mixture of about 37% sodium sulphate and 63% of the sodium salts of the sulphonic acids prepared by sulphonating a refined fraction of a liquid sulphur dioxide extract of a Gulf Coast petroleum with fuming sulphuric acid while dis-

5

solved in liquid sulphur dioxide, is dried on heated rolls with substantially no dusting.

Even half the quantity of monoglycerides will likewise reduce the amount of dusting but not to the same extent as the 8% of monoglyceride does in the above example.

Although the above examples are all directed to the treatment of sodium salts of the organic detergents and sulphuric acid, it is also within the scope of this invention to similarly process other salts including those of calcium, magnesium, and other alkaline earth metals; alkaline metals; earth metals; ammonium and substituted ammonium such as ethanolamine, and mono-, di- and tri-amyl amine; and pyridinium and substituted pyridinium.

The fire hazard in spray-drying is particularly troublesome in the spray-drying of salts of sulphonated diphenyl and alkylated (long-chain) derivatives thereof. For example, a mixture of equal parts sodium sulphate and sodium salts of sulphonated alkylated diphenyls, when an aqueous preparation thereof is sprayed in a spray tower, takes fire, causing injury to the apparatus and destruction of the product. However, a mixture of 5 parts of the sodium salts of sulphonated alkylated diphenyls, 3 parts of sodium sulphate and 2 parts alkaline soap builder such as borax will not burn during the spray-drying of preparations thereof.

It should be noted that among the substances suggested as being suitable as dust preventing and fire preventing agents there are found many materials which serve additional highly desirable functions. For example, monoglycerides have super-fatting properties and appear to improve materially the functions of the organic detergents.

The alkylol amine compounds are not only effective in the prevention of fires and dusting, but they also serve as wetting and emulsifying agents. Many of the other materials likewise serve these and/or other functions including flavoring, plasticizing, solubilizing, preserving, anti-spattering, lubricating, water-softening, thickening, humidifying, and the like.

Other agents may be added before, during or after producing the final product, among which are soaps; coloring agents; inert fillers; alkaline soap builders such as sodium carbonate; ammonia and ammonium salts; organic liquids; perfumes; fats; oils; waxes; gums; resins; germicides and deodorants; water-softening pyro- and hexameta-phosphoric acid compounds; or any of the common water-soluble alkaline metal salts. The type of addition agent which is to be incorporated will depend on the purposes for which the composition is to be used. The resulting compositions can be used wherever agents of this type have been previously employed or are likely to be employed such as in the household, as well as in the textile, cosmetic, paper, plastic and leather industries.

This application is a continuation of our prior application Serial No. 256,720, filed February 16, 1939 (now abandoned).

While the process herein described is well adapted for carrying out the objects of the present invention, it is understood that various modifications may be made thereof. The invention includes all such modifications and changes as come within the scope of the appended claims.

We claim:

1. The process of preparing improved detergents which comprises spray-drying an aqueous

6

alkaline preparation of a sodium salt of an alkylated aryl sulphonic acid, glycerine, and a monoglycerine of a fatty acid having at least six carbon atoms.

2. The process of preparing improved detergent compositions which comprises spray-drying an aqueous alkaline preparation comprising essentially water, a sodium salt of an alkylated aryl sulphonic acid and a monoglyceride of a fatty acid having at least six carbon atoms.

3. The process of preparing improved detergent compositions which comprises spray-drying an aqueous preparation comprising essentially water, a water-soluble salt of alkyl aromatic sulphonic acids and a small proportion of monoglycerides of fatty acids, each fatty acid having at least six carbon atoms to the molecule said proportion being within the range of about 1 to 10% based on total solids content.

4. The process of preparing improved detergent compositions which comprises spray-drying an aqueous preparation comprising essentially water, a normally-dusting water-soluble salt of an alkyl aryl sulphonic acid and a monoglyceride of a fatty acid which has at least six carbon atoms to the molecule.

5. The process of preparing improved detergent compositions which comprises preparing an aqueous mixture comprising essentially water, a normally-dusting water-soluble salt of an aryl sulphonic acid and a monoglyceride of a fatty acid having at least six carbon atoms to the molecule, and converting said mixture to particle form with substantial drying.

6. The process of preparing improved detergent compositions which comprises spray-drying an aqueous preparation comprising essentially water, a solid normally-dusting water-soluble salt of an alkylated aryl sulphonic acid and a small proportion of an aliphatic polyhydroxy compound which contains only carbon, hydrogen and oxygen, said proportion being within the range of about 1 to 10% based on total solids content.

7. The process of preparing improved detergent compositions which comprises preparing an aqueous mixture comprising essentially water, a normally-dusting, solid synthetic organic detergent having a non-carboxylic acid group in the molecule, and an aliphatic polyhydroxy compound which contains only carbon, hydrogen and oxygen, and converting said mixture to particle form with substantial drying to produce a product which is substantially dry and which would form a fine dust in the absence of the aliphatic polyhydroxy compound.

8. The process of claim 7 in which product is formed by spray-drying.

9. The process which comprises spray-drying an aqueous preparation comprising essentially water, a normally-dusting water-soluble salt of a sulphated fatty alcohol and a monoglyceride of a fatty acid containing at least six carbon atoms to the molecule.

10. The process of preparing improved detergents which comprises spray-drying an aqueous preparation comprising essentially water, the mono-glycerides of coconut oil fatty acids and the sodium salts of the mixed sulphates of fatty alcohols, derived by hydrogenation from coconut oil.

11. The process of preparing improved detergents which comprises preparing an aqueous mixture comprising essentially water, the monoglycerides of coconut oil fatty acids and the sodium salts of the mixed sulphates of fatty al-

cohols, derived by hydrogenation from coconut oil, and then converting said mixture to particle form with substantial drying.

12. The process of preparing improved detergents and like agents which comprises spray-drying an aqueous alkaline preparation comprising essentially water, a water-soluble salt of an alkylated aryl sulphonic acid and a polyhydroxy aliphatic compound which contains only carbon, hydrogen and oxygen.

13. The process of preparing improved detergents which comprises preparing an aqueous mixture comprising essentially water, the sodium salts of the mixed sulphates of fatty alcohols, derived by hydrogenation from the fatty oil, and a monoglyceride of a fatty acid having at least six carbon atoms, and then converting the mixture to particle form with substantial drying.

14. The process of preparing improved detergents which comprises spray-drying an aqueous preparation comprising essentially water, a normally-dusting, solid synthetic organic detergent having a non-carboxylic acid group in the molecule, glycerine and a monoglyceride of a fatty acid having at least six carbon atoms.

15. A composition of matter in the form of spray-dried particles comprising a normally-dusting, solid synthetic organic detergent having a non-carboxylic acid group in the molecule, glycerine and a monoglyceride of a fatty acid having at least six carbon atoms, the sum of the glycerine and monoglyceride amounting to about 1 to 10% of the total solids content.

16. A composition of matter in particle form having at least one short dimension comprising a normally-dusting, solid synthetic organic detergent having a non-carboxylic acid group in

the molecule, and an aliphatic polyhydroxy compound which contains only carbon, hydrogen and oxygen.

17. A composition of matter in particle form having at least one short dimension comprising a normally-dusting water-soluble salt of an alkyl aromatic sulphonic acid and a small proportion of a monoglyceride of a fatty acid having at least six carbon atoms, said proportion being within the range of about 1 to 10% based on total solids content.

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