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WATER-DISPERSIBLE METAL SOAP COMPOSITIONS

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This invention relates to metal soap compositions in a dry powdered form which may readily and easily be dispersed in water to produce stable aqueous dispersions of metal soap.

It has long been known that the metal soaps are hydrophobic, and, consequently, for many years the metal soaps have been employed for a great variety of waterproofing applications. In utilizing the metal soaps as waterproofing agents and for various other purposes, the most common practice has been to employ organic solvent solutions of the metal soaps. In a great many instances it is not possible to dissolve any appreciable amount of a metal soap in an organic solvent and still have a fluid material since the addition of only a very small amount of a metal soap causes many organic solvents to form firm gels. A number of materials have been suggested as peptizing agents to prevent such gelation from taking place and in some cases these peptizing agents have given very good results allowing solvent solutions containing relatively large amounts of metal soaps to be prepared. In many instances and for many purposes, however, an organic solvent solution of a metal soap is not a particularly desirable form in which to use a metal soap.

For many applications it would be highly desirable to have available aqueous dispersions of metal soaps, and in recent years a number of proposals have been advanced relative to the production of aqueous dispersions of metal soaps. However, because of the highly hydrophobic characteristics of the metal soaps, such dispersions are often difficult to prepare. In many cases the preparation of such dispersions requires the use of expensive dispersing or homogenizing equipment such as colloid mills, etc. In other cases the metal soap is first dissolved in an organic solvent therefor, often with the aid of a solubilizing agent since, as has been pointed out above, metal soaps tend to form gels in many organic solvents whenever present in any appreciable concentration, and the solvent solution then emulsified with water employing various emulsifying agents and vigorous methods of agitation, e. g. colloid mill, etc.

Ammonia and various alkylamines have been found to be fairly effective in dispersing metal soaps in aqueous mediums. When these aqueous dispersions which have been so prepared are used as coating agents, the water and the ammonia or alkylamine which has been employed as the dispersing agent will evaporate leaving a film of metal soap. Although such dispersions are not

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as difficult to prepare as those mentioned above, they still have the disadvantage of requiring the transportation of large quantities of water whenever the dispersions are shipped from the manufacturer to the ultimate consumer. Also such dispersions are rather alkaline which makes them somewhat unsuited for some uses.

A considerable saving on freight charges and thus a reduction in the cost to the ultimate consumer could be effected if it were possible to provide metal soap compositions in a dry form which could readily and easily be dispersed in water. As was pointed out above, ammonia and various alkylamines are fairly effective dispersing agents for metal soaps; however, in view of their volatility it is apparent that they would be quite unsuitable for preparing dispersible metal soap compositions in dry form. Other dispersing agents which have been employed require the use of specialized dispersing equipment or of specialized and often complicated procedures in preparing metal soap dispersions. Consequently all of this has necessitated preparation by the manufacturer of the aqueous dispersions as such, and has thus required the transportation of large quantities of water when the dispersions have been shipped to the ultimate consumer. Although the advantages of having in a dry powdered form a metal soap composition which anyone may readily and easily disperse in water are quite obvious, no one appears to have been able heretofore to produce a commercially practicable composition of this type.

It is the object of this invention to provide water-dispersible metal soap compositions in dry, powdered form.

A further object of the invention is to provide dry, powdered metal soap compositions which may easily be dispersed in aqueous media to form stable, concentrated aqueous dispersions of metal soaps.

Other objects of the invention will in part be obvious and will in part appear hereinafter.

We have discovered that the above and other objects of the invention may be realized by admixing a pulverulent metal soap with a small amount of an alkylolamide prepared by reacting a fatty acid with an alkylolamine. Such a composition in the dry powdered form is readily and easily dispersible in aqueous media.

In preparing the dry compositions of our invention any pulverulent metal soap of a saturated fatty acid containing from 10 to 22 carbon atoms may be employed. The metal soaps which we employ are preferably prepared by double decom-

position processes wherein water-soluble soaps of the desired fatty acids and water-soluble inorganic salts of the desired metals are reacted together in an aqueous solution to form the desired metal soaps which, since they are water-insoluble, precipitate from the solution. Metal soaps produced in such a manner precipitate in a powdery form and after being separated from the aqueous solution and dried, usually need not be ground or further sub-divided before being utilized in preparing the products of our invention. In one instance, however, the metal soap employed in preparing the products of our invention is preferably produced by direct reaction between an inorganic salt of the desired metal and the desired fatty acid or a fatty material containing the desired fatty acid. This is the case when a soap of lithium is prepared. Since lithium is so closely related physically and chemically to sodium and potassium it would seem that lithium soaps would be water-soluble; however, that is not the case and in most instances, as in this specification, lithium soaps are included in the class of so-called "metal soaps" in view of their insolubility in water.

The fatty acids employed in preparing the metal soaps used in our novel compositions are the saturated fatty acids containing from 10 to 22 carbon atoms. In many cases instead of using the free fatty acids themselves, glycerides containing such fatty acids may be employed. Since in many cases glycerides contain varying amounts of unsaturated fatty acids and saturated fatty acids containing less than 10 carbon atoms, metal soaps prepared employing glycerides as the source of fatty acids will often contain varying amounts of such acids. Such metal soaps may be employed in preparing the products of our invention, however, as long as they contain sufficient saturated fatty acids of 10 to 22 carbon atoms to give soaps which have a powdery form or which on being ground will be in a powdery form. Excellent examples of such metal soaps are the so-called metal "palmitates" of commerce. The metal palmitates of commerce instead of being produced from palmitic acid as would be supposed are actually produced from the mixture of fatty acids contained in coconut oil. However, even though the metal palmitates contain some fatty acids other than saturated fatty acids containing from 10 to 22 carbon atoms, they all either have a powdery form or are capable of being reduced to a powdery form by grinding. Thus they are quite suitable for use in preparing the products of our invention.

As specific examples of metal soaps suitable for use in preparing the products of our invention there may be mentioned aluminum stearate, zinc stearate, calcium stearate, barium stearate, lithium stearate, magnesium stearate, lead stearate, copper stearate, the corresponding metal palmitates, etc. As is well known, the commercial metal stearates contain fatty acid chains of somewhat varying carbon content depending upon the source of the fatty material. In most cases, however, chain lengths of 16 and 18 carbon atoms will predominate.

Alkylolamines containing from 2 to 7 carbon atoms may suitably be used in producing the alkylolamides to be employed in preparing the water-dispersible compositions of our invention. As examples of such amines there may be mentioned monoethanolamine, diethanolamine, 1-amino - 2 - ethyl-3-ethanolaminoisopropanol, 1-amino - 2 - ethyl - 3-methylaminoisopropanol, 1-amino - 3 - dimethylaminoisopropanol, ethanol ethylenediamine, 1-amino-3-methylaminoisopropanol, N-ethyl N'-ethanol ethylenediamine, N,N'-diethanol ethylenediamine, isopropanol ethylenediamine, diisopropanolamine, monopropanolamine, dipropanolamine, 1,3-diaminoisopropanol, ethyl ethanolamine, butyl ethanolamine, ethanol diethylenetriamine, ethanol propylenediamine, etc.

In preparing the alkylolamides which are to be incorporated into the compositions of our invention, an alkylolamine of the type described above is amidified with a fatty acid containing from 8 to 22 carbon atoms. When the alkylolamine which is being employed is a polyamine, it is preferred to amidify only one of the amine groups with a fatty acid containing from 8 to 22 carbon atoms and to amidify the remaining reactive amine groups with an organic acid containing from 2 to 5 carbon atoms. In any case it is preferred that the alkylolamine be substantially completely amidified. Details of preparing such amides may be found in U. S. Patents Numbers 2,340,881 and 2,425,393 and for the sake of brevity such details will not be repeated here.

The ratio of the alkylolamide to the metal soap in the compositions of our invention will vary slightly depending upon the concentration of the metal soap which will be desired in the dispersions later prepared from the dry dispersible products of the invention with a slightly higher proportion of the amide being employed as the concentration of the metal soap is increased. Ordinarily an amount of the amide equal to from about 5% to about 25% of the weight of the metal soap will be sufficient although larger amounts may be employed if desired since there is no critical upper limit. Likewise amounts smaller than 5% may be employed, e. g. 1% although for best results it is preferred to use at least 5% of amide.

In preparing the dry dispersible products of our invention, the metal soap and the alkylolamide may be admixed in any suitable manner. The dry metal soap may be thoroughly mixed with the amide after the amide has been rather finely divided, e. g. by grinding, if the particular amide being employed is a solid, or the metal soap may be mixed with an amide which is in the liquid form or which has a melting point which is not excessively high, e. g. not over 150° C., and may thus easily be converted to the liquid form and the resulting liquid admixed with the metal soap. If the product obtained is not already in more or less of a powdery form, it is preferred to grind it or otherwise finely subdivide it.

Instead of mixing an alkylolamide with a dry metal soap, the compositions of the invention may be prepared in conjunction with the manufacture of the metal soaps. When the compositions of the invention are so prepared, the metal soap may be precipitated in the presence of the amide or the amide may be admixed with the wet metal soap just after it is precipitated. After the wet metal soap and the amide are thoroughly mixed, the resulting composition is dried, and then preferably ground or otherwise finely divided to more or less of a powdery form.

Although some of the alkylolamides employed in preparing the compositions of the invention are liquids, this does not affect the dry character of the products. The metal soaps appear to more or less adsorb or absorb such amides, and thus in physical appearance there is very little if any difference between the dry metal soaps

themselves and the dry easily dispersible products of our invention. It is to be understood that the term "dry" as used herein with reference to the water-dispersible compositions of our invention does not necessarily means that the compositions are completely anhydrous although they may be if so desired. By the term "dry" we mean that they have a dry appearance and feel in the ordinary sense of the word.

In employing the compositions of our invention to prepare aqueous dispersions of metal soaps, no special mixing or dispersing equipment is required. The dry dispersible products may be merely added to an aqueous medium or vice versa and the products then dispersed in the aqueous medium by means of moderate but thorough agitation. If desired the dry compositions or the aqueous medium, as the case may be, may be added a small amount at a time with each portion being thoroughly admixed before the next is added. Also, if desired the aqueous medium may be heated slightly, e. g. to about 60–70° C., to aid in the dispersing.

The concentration of metal soap to be incorporated into an aqueous dispersion may vary considerably. Preferably not more than about 40% of metal soap is incorporated into an aqueous dispersion. The lower limit may be as low as the particular user may desire or it may be whatever concentration may be desirable for the particular application involved. In most cases, however, it is preferred to have at least about 5% of metal soap in the aqueous dispersions.

Metal soap dispersions prepared from the compositions of the invention may be employed to waterproof, mildewproof, etc. textiles, leather, paper, concrete, etc. They may also be used as defoamers or used for any other purpose where an aqueous dispersion of a metal soap would be useful.

Although we are particularly concerned with the production of the dry, water-dispersible metal soap compositions, the scope of our invention includes, of course, not only those compositions but also the aqueous dispersions produced therefrom.

For a fuller understanding of the nature and objects of the invention reference may be had to the following examples which are given merely to illustrate further the invention and are not to be construed in a limiting sense, all parts given being by weight.

Example I

Twenty-five parts of an amide prepared by reacting diethanolamine with sufficient coconut oil to substantially completely amidify the diethanolamine were admixed with 100 parts of dry, powdered aluminum stearate. The amide, which was an amber liquid, was slowly added to the aluminum stearate while the aluminum stearate was rapidly agitated. The resulting white, dry, powdery composition was very easily dispersible in water in large proportions. A 20% dispersion of the composition was a stable, thin fluid. A 50% dispersion of the composition (one to one ratio of the composition to water) was a viscous stable fluid. These dispersions were prepared merely by adding the proper amount of water in each case to a quantity of the dispersible composition and then thoroughly agitating the mixture.

Example II

Five parts of the amide employed in Example I were admixed with 100 parts of aluminum stearate in the same manner as in Example I to give a powdery mixture which was readily dispersible in water. A 20% aqueous dispersion of the mixture prepared in the same manner as in Example I was very stable but was not quite as fluid as the 20% dispersion of Example I.

Example III

Twenty-five parts of the amide of Example I were admixed with 50 parts of aluminum stearate in the same manner as in Example I to give a powdery mixture which was easily dispersible in water. Twenty parts of the mixture admixed with 80 parts of water in the same manner as in Example I gave a very fluid, stable dispersion.

Example IV

An alkyloldiamide was prepared from amino ethyl ethanolamine by amidifying one of the amino groups with commercial oleic acid and amidifying the other amino group with acetic acid. The amide was an amber, slushy liquid. Twenty-five parts of this amide were heated until completely liquid and then slowly added with rapid agitation to 100 parts of dry, powdered aluminum stearate resulting in a white, dry, powdery mixture which was readily dispersible in water to give fluid, concentrated aqueous dispersions. A 20% aqueous dispersion prepared in the same manner as in Example I was fluid and stable.

Example V

Diethanolamine was amidified with commercial stearic acid giving a light tan solid melting at about 40° C. Twenty-five parts of the product obtained were heated until liquid and then slowly added with thorough agitation to 100 parts of dry, powdered aluminum stearate giving a dry, powdery composition. This composition was readily dispersible in water in large concentrations to give fluid aqueous dispersions. Twenty parts thereof dispersed in 80 parts of water in the same manner as in Example I gave a stable, fluid dispersion.

Example VI

One hundred parts of dry, powdered zinc stearate were admixed with 25 parts of the amide of Example I in the same manner as in Example I. The resulting powdery composition was readily dispersible in water. A 20% aqueous dispersion of the composition, prepared as in Example I, was a thin, stable fluid.

Example VII

One hundred parts of dry, powdered zinc stearate were admixed with 5 parts of the amide of Example I in the same manner as in Example I giving a dry, powdery product which was readily dispersible in water. A 20% aqueous dispersion of the product prepared as in Example I was a very thick, stable fluid.

Example VIII

Fifty parts of dry, powdered zinc stearate were admixed with 25 parts of the amide of Example I in the same manner as in Example I giving a dry, powdery composition which was easily dispersible in water. A 20% aqueous dispersion of the composition prepared as in Example I was a thick, stable fluid.

Example IX

One hundred parts of dry, powdered aluminum stearate were admixed with 25 parts of an amide prepared by amidifying monoethanolamine with coconut oil fatty acids. The amide, which was

a tan solid with a melting point of about 52° C., was heated until liquid and then slowly added to the dry, powdered aluminum stearate with thorough agitation. The resulting dry, powdery composition was easily dispersible in water to give stable, concentrated, fluid dispersions. Twenty parts thereof dispersed in 80 parts of water in the same manner as in Example I resulted in a stable fluid dispersion.

Example X

Twenty-five parts of the amide of Example I were admixed with 100 parts of dry, powdered calcium stearate in the same manner as in Example I. A dry, powdery composition which was easily dispersible in water to give stable aqueous dispersions was obtained. A 20% aqueous dispersion of the product prepared as in Example I was fluid and stable.

Example XI

One hundred parts of dry, powdered magnesium stearate were admixed with 25 parts of the amide of Example I in the same manner as in Example I giving a dry, powdery product. A 20% aqueous dispersion of the product prepared as in Example I was very thick and stable.

Example XII

One hundred parts of dry, powdered lithium stearate were admixed with 25 parts of the amide of Example I in the same manner as in Example I giving a dry, powdery composition. A 20% aqueous dispersion of the composition prepared as in Example I was fluid and stable.

Example XIII

Twenty-five parts of the amide of Example I and 100 parts of dry, powdered barium stearate were admixed as in Example I. The resulting dry, powdery product was easily dispersible in water. A 20% aqueous dispersion of the product, prepared as in Example I, was fluid and stable.

From the above descriptions and examples it is quite apparent that our invention provides dry metal soap compositions which are highly suitable for forming any desired kind of an aqueous metal soap dispersion.

The dry, water-dispersible products of our invention may also have included therein other solid constituents, such as plasticizers, dyes, pigments or other coloring matter, etc.

Having described our invention, what we claim as new and desire to secure by Letters Patent is:

1. As a new composition of matter, a solid comprising an intimate mixture of a water-insoluble soap of a saturated fatty acid containing from 10 to 22 carbon atoms and an alkylolamide, said alkylolamide being prepared by substantially completely amidifying an alkylolamine containing from 2 to 7 carbon atoms, each molecule of said amide containing at least one fatty acyl radical containing from 8 to 22 carbon atoms, said composition of matter being characterized by the property of being readily dispersible in aqueous media and adapted to form free-flowing concentrated aqueous dispersions with aqueous media.

2. As a new composition of matter, a solid comprising an intimate mixture of a water-insoluble soap of a saturated fatty acid containing from 10 to 22 carbon atoms and an alkylolamide, said alkylolamide being prepared by substantially completely amidifying an alkylolpolyamine containing from 3 to 7 carbon atoms, each molecule of said amide containing at least one fatty acyl radical containing from 8 to 22 carbon atoms, said composition of matter being characterized by the property of being readily dispersible in aqueous media and adapted to form free-flowing concentrated aqueous dispersions with aqueous media.

3. As a new composition of matter, a solid comprising an intimate mixture of a water-insoluble metal stearate and an alkylolamide, said alkylolamide being prepared by substantially completely amidifying an alkylolamine containing from 2 to 7 carbon atoms, each molecule of said amide containing at least one fatty acyl radical containing from 8 to 22 carbon atoms, said composition of matter being characterized by the property of being readily dispersible in aqueous media and adapted to form free-flowing concentrated aqueous dispersions with aqueous media.

4. As a new composition of matter, a solid comprising an intimate mixture of a water-insoluble aluminum soap of a saturated fatty acid containing from 10 to 22 carbon atoms and an alkylolamide, said alkylolamide being prepared by substantially completely amidifying an alkylolamine containing from 2 to 7 carbon atoms, each molecule of said amide containing at least one fatty acyl radical containing from 8 to 22 carbon atoms, said composition of matter being characterized by the property of being readily dispersible in aqueous media and adapted to form free-flowing concentrated aqueous dispersions with aqueous media.

5. As a new composition of matter, a solid comprising an intimate mixture of aluminum stearate and an alkylolamide, said alkylolamide being prepared by substantially completely amidifying an alkylolamine containing from 2 to 7 carbon atoms, each molecule of said amide containing at least one fatty acyl radical containing from 8 to 22 carbon atoms, said composition of matter being characterized by the property of being readily dispersible in aqueous media and adapted to form free-flowing concentrated aqueous dispersions with aqueous media.

6. As a new composition of matter, a pulverulent solid comprising an intimate mixture of a water-insoluble soap of a saturated fatty acid containing from 10 to 22 carbon atoms and an alkylolamide, said alkylolamide being prepared from an alkyloldiamine containing from 3 to 7 carbon atoms by amidifying one of the two amine groups with a fatty acid containing from 8 to 22 carbon atoms and amidifying the other amine group with an organic acid containing from 2 to 5 carbon atoms, said composition of matter being characterized by the property of being readily dispersible in aqueous media and adapted to form free-flowing concentrated aqueous dispersions with aqueous media.

7. As a new composition of matter, a pulverulent solid comprising an intimate mixture of a water-insoluble soap of a saturated fatty acid containing from 10 to 22 carbon atoms and an alkylolamide, said alkylolamide being prepared by substantially completely amidifying diethanolamine with coconut oil fatty acids, the alkylolamide being present in the mixture in an amount of between about 1% and about 50% based upon the weight of the water-insoluble soap, said composition of matter being characterized by the property of being readily dispersible in aqueous media and adapted to form free-flowing concentrated aqueous dispersions with aqueous media.

8. As a new composition of matter, a pulverulent solid comprising an intimate mixture of a

metal stearate and an alkylolamide, said alkylolamide being prepared by substantially completely amidifying diethanolamine with coconut oil fatty acids, the alkylolamide being present in the mixture in an amount of between about 1% and about 50% based upon the weight of the metal stearate, said composition of matter being characterized by the property of being readily dispersible in aqueous media and adapted to form free-flowing concentrated aqueous dispersions with aqueous media.

9. As a new composition of matter, a pulverulent solid comprising an intimate mixture of aluminum stearate and an alkylolamide, said alkylolamide being prepared by substantially completely amidifying diethanolamine with coconut oil fatty acids, the alkylolamide being present in the mixture in an amount of between about 1% and about 50% based upon the weight of the aluminum stearate, said composition of matter being characterized by the property of being readily dispersible in aqueous media and adapted to form free-flowing concentrated aqueous dispersions with aqueous media.

10. As a new composition of matter, a pulverulent solid comprising an intimate mixture of a water-insoluble soap of a saturated fatty acid containing from 10 to 22 carbon atoms and an alkylolamide, said alkylolamide being prepared by substantially completely amidifying monoethanolamine with coconut oil fatty acids, the alkylolamide being present in the mixture in an amount of between about 1% and about 50% based upon the weight of the water-insoluble soap, said composition of matter being characterized by the property of being readily dispersible in aqueous media and adapted to form free-flowing concentrated aqueous dispersions with aqueous media.

11. As a new composition of matter, a pulverulent solid comprising an intimate mixture of a metal stearate and an alkylolamide, said alkylolamide being prepared by substantially completely amidifying monoethanolamine with coconut oil fatty acids, the alkylolamide being present in the mixture in an amount of between about 1% and about 50% based upon the weight of the metal stearate, said composition of matter being characterized by the property of being readily dispersible in aqueous media and adapted to form free-flowing concentrated aqueous dispersions with aqueous media.

12. As a new composition of matter, a pulverulent solid comprising an intimate mixture of aluminum stearate and an alkylolamide, said alkylolamide being prepared by substantially completely amidifying monoethanolamine with coconut oil fatty acids, the alkylolamide being present in the mixture in an amount of between about 1% and about 50% based upon the weight of the aluminum stearate, said composition of matter being characterized by the property of being readily dispersible in aqueous media and adapted to form free-flowing concentrated aqueous dispersions with aqueous media.

13. As a new composition of matter, a pulverulent solid comprising an intimate mixture of a water-insoluble soap of a saturated fatty acid containing from 10 to 22 carbon atoms and an alkylolamide, said alkylolamide being prepared by substantially completely amidifying amino ethyl ethanolamine, one amino group thereof being amidified with oleic acid and the other amino group being amidified with acetic acid, the alkylolamide being present in the mixture in an amount of between about 1% and about 50% based upon the weight of the water-insoluble soap, said composition of matter being characterized by the property of being readily dispersible in aqueous media and adapted to form free-flowing concentrated aqueous dispersions with aqueous media.

14. As a new composition of matter, a pulverulent solid comprising an intimate mixture of a metal stearate and an alkylolamide, said alkylolamide being prepared by substantially completely amidifying amino ethyl ethanolamine, one amino group thereof being amidified with oleic acid and the other amino group being amidified with acetic acid, the alkylolamide being present in the mixture in an amount of between about 1% and about 50% based upon the weight of the metal stearate, said composition of matter being characterized by the property of being readily dispersible in aqueous media and adapted to form free-flowing concentrated aqueous dispersions with aqueous media.

15. As a new composition of matter, a pulverulent solid comprising an intimate mixture of aluminum stearate and an alkylolamide, said alkylolamide being prepared by substantially completely amidifying amino ethyl ethanolamine, one amino group thereof being amidified with oleic acid and the other amino group being amidified with acetic acid, the alkylolamide being present in the mixture in an amount of between about 1% and about 50% based upon the weight of the aluminum stearate, said composition of matter being characterized by the property of being readily dispersible in aqueous media and adapted to form free-flowing concentrated aqueous dispersions with aqueous media.

16. An aqueous metal soap dispersion comprising water and the product of claim 1.

17. An aqueous metal soap dispersion comprising water and the product of claim 3.

18. An aqueous metal soap dispersion comprising water and the product of claim 7.

19. An aqueous metal soap dispersion comprising water and the product of claim 10.

20. An aqueous metal soap dispersion comprising water and the product of claim 13.

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