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METALLIC SOAPS OF FATTY ACIDS AND ALPHA-HYDROXY ALKANOIC ACIDS

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This invention relates to polyvalent metal soaps capable of forming fluid solutions in organic solvents when dissolved therein in amounts up to 30% and to coating, impregnating, sealing and water-proofing compositions containing them. More particularly, the invention is directed to mixed polyvalent metal salts wherein both an alpha-hydroxy alkanoic acid containing from 8 to 12 carbon atoms and a higher fatty acid of about 10 to 22 carbon atoms are combined into an aluminum, zinc, calcium or magnesium soap composition possessing the property of forming low viscosity solutions in volatile hydrocarbon and chlorinated hydrocarbon solvents. The invention includes the soaps themselves and impregnating and water-proofing compositions containing them.

The coating and waterproofing properties of the polyvalent metal soaps, better known in the art as metallic soaps, has been well recognized and their application of these and similar uses is now firmly established. The most convenient method of applying such polyvalent metal soaps to various surfaces such as wood, paper, cloth, leather, masonry, wall-board, and the like, has been to make up a solution thereof in an organic solvent and to spray the same on the surface to be treated. Various volatile organic solvents have been used in the preparation of the polyvalent metal soap solutions, the more common ones including the aliphatic and aromatic hydrocarbons and their halogenated derivatives.

While the foregoing practice has found some success in the art, many difficulties have been encountered, inasmuch as when more than about 2% or 3% of some soaps are dissolved in some organic solvents, the resulting mass either thickens to such an extent or actually forms a solidified gel, as to render it substantially useless as a sprayable coating or waterproofing composition. While the thickened mass or solidified gel can be heated to render it less viscous, the application thereof in a heated state is occasionally dangerous due to the volatile and inflammable solvents used and is usually unsatisfactory for most purposes. Furthermore, the application of solutions containing only from 2% to 3% of the polyvalent metal soap is also highly commercially impractical in view of the low concentration of the solids in the solution, which factor additionally increases the cost of the product due to the large amounts of solvent necessary.

Various efforts have been made to solve the foregoing problems by increasing the soap concentration without undesirably decreasing the fluidity of the resulting solution. Peptizing agents having an acidic nature, such as acetic acid, phenol, tartaric acid, oxalic acid, and the like, have been used to increase the soap concentration in a solvent mixture without affecting the fluidity thereof. This proposal has proven to be undesirable commercially in view of the corrosive character imparted to the composition by such acidic agents. Another effort has involved the use of the ethanolamines, or the ethanolamine salts of the higher fatty acids, for the purpose of increasing the concentration of polyvalent metal soap

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solution. A solution produced according to this teaching has not found too widespread success.

A principal object of the present invention is to overcome the foregoing defects and other disadvantages.

Another principal object of the present invention is to provide an improved, relatively highly concentrated polyvalent metal soap solution of low viscosity.

A further principal object of the present invention is to provide an improved coating and waterproofing composition.

A still further principal object of the invention is to provide fluid polyvalent metal soap solutions containing up to 30% polyvalent metal soap concentrations.

It has now been found that the foregoing and other objects of the present invention may be achieved by the use of an alpha-hydroxy alkanoic acid containing from 8 to 12 carbon atoms, in conjunction with the fatty acid, or equivalent acid, used in the formation of the polyvalent metal soap. It has been found that the use of the alpha-hydroxy alkanoic acid remarkably reduces the viscosity of the resulting commercial compositions whereby readily sprayable solutions containing high percentages of metallic soaps can be prepared.

Representative alkanoic acids of the present invention would include as illustrative, but not as limitative, the following: alpha-hydroxy octanoic acid; alpha-hydroxy decanoic acid; alpha-hydroxy dodecanoic acid; 2-hydroxy-4-methyl-6,6-dimethyl heptanoic acid; 3-ethyl-2-hydroxy heptanoic acid; 6-methyl-2-hydroxy heptanoic acid; etc. It is to be particularly noted that in all these acids, the hydroxy group is attached to the carbon atom adjacent the carboxyl group.

In the preparation of the composition of the invention, any suitable water-insoluble polyvalent metal soap of a higher fatty acid or similar acid may be employed. Examples of such soaps include the polyvalent metal soaps of fatty acids containing from 10 to 22 or more carbon atoms, such as for example, the aluminum, calcium, magnesium, zinc, etc., soaps of capric, lauric, myristic, palmitic, stearic, arachidic, behenic, oleic, palmitoleic, lauroleic, myristoleic, linoleic, ricinoleic, and the like acids. Mixtures of acids such as hydrogenated fish oil fatty acids, hydrogenated cottonseed oil fatty acids, hydrogenated tallow fatty acids, hydrogenated soya bean fatty acids, and the like, are similarly usable. While any suitable polyvalent metal soap may be used in accordance with the invention, the aluminum soaps are preferred, and particularly aluminum stearate, palmitate or other saturated soaps of this metal.

The invention will be further described in greater detail by the following specific examples. It should be understood, however, that although these examples may set forth in particular detail some of the more specific features of the invention, they are given primarily for purposes of illustration of preferred preparations and evaluations and the invention in its broader aspects is not to be construed as limited thereto.

Example 1

A soap solution was prepared from 85 grams of 2-hydroxy-4-methyl-6,6-dimethyl heptanoic acid, 400 grams of "Hydrofol 51" (having the approximate composition: 7.5% C_{14}; 33.3% C_{16}; 27.2% C_{18}; 17.2% C_{20}; 14.8% C_{22}; 4.3% unsaturated acids); 100 grams of 98% sodium hydroxide and 1500 grams of water. This solution was heated to a temperature of approximately 65° C. and a second solution comprising 270 grams of alum and 2000 grams of water was added thereto. The aluminum soap precipitate which formed was filtered, washed on a laboratory crock and dried overnight at a temperature of approximately 75° C. The dried material was ground in a micropulverizer at a speed of approximately 4000 R. P. M. through a 1/8 inch r. p. screen.

A 20% solution in commercial toluene was found to be very fluid at room temperature and could be easily sprayed on fabrics, paper, wood shingles, concrete and other building materials as a waterproofing agent therefor.

A 20% solution in Stoddard solvent was very fluid and could be readily sprayed on textile materials, masonry, wall-board, asbestos, rockwool, and the like. The material was also readily soluble in benzene, xylene, carbon tetrachloride and similar aliphatic and aromatic hydrocarbons and their halogenated derivatives.

A laboratory analysis of the aluminum soap indicated the following: 10.10% ash, 9.22% washed ash; 0.88% water-soluble ash; 5.9 mgm. KOH acid value (hot acetone extraction method).

Example 2

A soap solution was prepared from 200 grams of alpha-hydroxy decanoic acid; 800 grams of "Hydrofol 45" (1.5% C_{14}; 28.0% C_{16}; 70.5% C_{18}; 6.0% unsaturated acids), 175 grams of 98% sodium hydroxide and 15,000 grams of water. This solution was heated to a temperature of approximately 65° C. and a second solution comprising 400 grams of zinc sulfate and 500 grams of water was added thereto. The zinc soap precipitate which formed was filtered, washed on a laboratory crock and dried overnight at a temperature of approximately 75° C. The dried product was ground in a micro-pulverizer at 4000 R. P. M. through a 1/8 inch r. p. screen. A laboratory analysis of the zinc soap indicated the following: 16.65% ash, 14.7% washed ash and 1.9% water-solubles. A 15% solution in commercial toluene was applied as a waterproofing agent to wood shingles, concrete, cement, and similar building materials.

Example 3

A soap solution was prepared from 200 grams of alpha-hydroxy decanoic acid, 800 grams of "Hydrofol 51," 212 grams of 98% sodium hydroxide and 15,000 grams of water. This solution was heated to a temperature of approximately 65° C. and a second solution comprising 540 grams of alum and 2500 grams of water was added thereto. The aluminum soap precipitate which formed was filtered, washed, and dried overnight in an oven maintained at a temperature of approximately 75° C. A laboratory analysis of the aluminum soap indicated: 10.05% ash; 9.18% washed ash; 0.87% water-soluble ash and 20.7 mgm. KOH acid value. A 30% solution in alpha-pinene was heated to 67° C. and was very thin and clear while hot. It was a readily sprayable waterproofing solution and could be sprayed on various surfaces such as wood, masonry, and the like. A 20% solution in Stoddard solvent was fluid and could be easily sprayed. A 25% solution in commercial toluene was similarly fluid and could also be readily sprayed. A 30% solution in commercial toluene was slightly thicker but still fluid.

The solubility of the aluminum soap was investigated further as follows: It was found to be clear, thin and readily sprayable in 15% solutions of petroleum ether, carbon tetrachloride, trichlorobenzene, cyclohexane, and other similar solvents.

Example 4

A soap solution was prepared from 100 grams of alpha-hydroxy decanoic acid, 17 grams of 98% sodium hydroxide and 4000 grams of water. This solution was heated to a temperature of approximately 45° C. and to it was added a second solution comprising 45 grams of zinc sulfate and 2500 grams of water. The zinc soap precipitate which formed was filtered, washed and dried overnight in an oven maintained at a temperature of approximately 75° C. A laboratory analysis of the zinc soap indicated the following: 14.95% ash; 13.37% washed ash; 1.58% water-soluble ash; and 1.6 mgm. KOH acid value.

A 15% solution in commercial toluene was heated to 85 °C., was very fluid and could be readily sprayed as a waterproofing agent. On cooling, the solution remained very thin and fluid and retained its sprayable characteristics.

Example 5

The following materials were employed to prepare the aluminum soap of alpha-hydroxy decanoic acid and mixed fatty acids:

800 grams of "Hydrofol 55"
200 grams of alpha-hydroxy decanoic acid
212 grams of 98% sodium hydroxide
540 grams of alum
18,500 grams of water.

Various methods of precipitation were used to form the aluminum soap. In the first method, the alum solution was added to the sodium soap. In the second method, the sodium soap solution was added to the alum solution; and in the third method; the sodium soap solution and the alum solution were added simultaneously to the precipitation tank.

All three methods were found to be satisfactory but the second method was found to produce a filter cake which was not too grainy and a smoother resulting product. The temperatures of precipitation were varied from about 50° to about 80° and all temperatures were found satisfactory although it was noted that a lower temperature occasionally prevented the formation of a precipitate which was too grainy.

Example 6

The procedures set forth in Example 1 were followed substantially as set forth therein with the exception that a single pressed stearic acid was substituted for the mixed fatty acids used therein. The resulting aluminum soap was very similar to that derived from Example 1.

Example 7

A soap solution was prepared from 100 grams of alpha-hydroxy capric acid, 400 grams of "Hydrofol 405," 100 grams of 98% sodium hydroxide and 16,000 grams of water. This solution was heated to a temperature of approximately 65° C. and a second solution comprising 540 grams of alum and 500 grams of water was added thereto. The aluminum soap precipitate which formed was filtered and washed and then dried overnight at a temperature of about 70° C. A laboratory analysis of the dried material indicated the following: 9.13% ash; 8.8% washed ash; 0.33% water-soluble ash; and 18.7 mgm. KOH acid value. A 10% solution in commercial toluene was used as a waterproofing agent and was sprayed on cotton, wool, asbestos, and like textile materials. A 15% solution in commercial toluene was applied to wood shingles, concrete, cement, masonry, and like building materials.

Example 8

A soap solution was prepared from 400 grams of "Emersol 110" (a mixture of C_{12} to C_{18} fatty acids), 100 grams of 2-ethyl-3-hydroxy hexanoic acid, 100 grams of 98% sodium hydroxide and 16,000 grams of water. This solution was heated to a temperature of approximately 75° C. and to it was added a second solution comprising 270 grams of alum and 1000 grams of water. The aluminum soap which precipitated was filtered and washed and then dried overnight in an oven at a temperature of approximately 80° C. Laboratory analysis indicated the following: 9.57% ash; 8.71% washed ash; 0.86% water-soluble ash; 2.26% moisture and volatile matter at 105° C. and 28.6 mgm. KOH acid value. 10% waterproofing solutions in commercial toluene were applied to concrete, cement, masonry, and like building materials.

The quantity by weight of alpha-hydroxy alkanoic acids used in proportion to the total acid in the preparation of the soap may be varied within wide limits. As little as

1% or less by weight of the alpha-hydroxy acid may be used and as high as up to 100% may be used, although the higher cost of such compositions renders them commercially and economically impracticable as coating, impregnating, sealing, or waterproofing agents. In general, it may be stated that compositions containing from about 5% to about 30% by weight of the alpha-hydroxy alkanoic acid, are economically and industrially most desirable.

As shown primarily in the examples, these metallic soaps are soluble up to as high as 30% in a very wide range of solvents without becoming too thick or gelatinous to use readily. It was observed that in many cases, aluminum stearate, when used without the addition of the alpha-hydroxy acid, could not form the thin and clear concentrated solutions which were sprayable, which solutions were made possible by the addition of the hydroxy acids during the soap formation.

This viscosity reducing characteristic created by the use of such an acid is also of value in decreasing the viscosity, when desired, of other compounded compositions such as greases, waxes, soaps, paints, varnishes, plastics, cosmetics, adhesives, rubber and the like.

Although we have described several specific embodiments of our inventive concept, we consider the same not to be limited thereto nor to the specific substances and compounds mentioned therein, but to include various other compounds and compositions of equivalent constitution as set forth in the claims appended hereto. It is understood, of course, that any suitable changes, modifications and variations may be made without departing from the spirit and scope of the invention.

What we claim is:

1. A soap composition comprising metal soaps of about 5% to 30% by weight of an alpha-hydroxy alkanoic acid containing from 8 to 12 carbon atoms and 95% to 70% by weight of a fatty acid containing from 10 to 22 carbon atoms, said metal being selected from the group consisting of aluminum, zinc, calcium and magnesium.

2. A composition according to claim 1 wherein the alpha-hydroxy alkanoic acid is alpha-hydroxy decanoic acid.

3. A composition according to claim 1 wherein the alpha-hydroxy alkanoic acid is 2-hydroxy-3-ethyl heptanoic acid.

4. A soap composition comprising an aluminum soap of about 5% to 30% by weight of an alpha-hydroxy alkanoic acid containing from 8 to 12 carbon atoms and 95% to 70% by weight of a fatty acid containing from 10 to 22 carbon atoms.

5. A water-proofing composition comprising a volatile hydrocarbon solvent containing up to 30% of a metal soap of about 5% to 30% by weight of an alpha-hydroxy alkanoic acid containing from 8 to 12 carbon atoms and 95% to 70% by weight of a fatty acid containing from 10 to 22 carbon atoms, said metal being selected from the group consisting of aluminum, zinc, calcium and magnesium.

6. A water-proofing composition according to claim 5 wherein the alpha-hydroxy alkanoic acid is alpha-hydroxy decanoic acid.

7. A water-proofing composition according to claim 5 wherein the alpha-hydroxy alkanoic acid is 2-hydroxy-3-ethyl-heptanoic acid.

References Cited in the file of this patent

UNITED STATES PATENTS

2,081,407	Minich	May 25, 1937
2,223,158	Licata et al.	Nov. 26, 1940
2,350,688	Licata et al.	June 6, 1944
2,453,520	Lang Kammerer	Nov. 9, 1948
2,618,596	Minich et al.	Nov. 18, 1952
2,626,897	Young et al.	Jan. 27, 1953
2,628,202	Allison et al.	Feb. 10, 1953