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WETTING-OUT COMPOSITIONS

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6 Claims. (Cl. 252-356)

Our invention relates to wetting-out compositions and is particularly concerned with such compositions which contain caprylic acid partial esters of triethanolamine together with certain agents which bring about marked enhancement of the wetting-out properties of said caprylic acid esters of triethanolamine.

The caprylic acid partial esters of triethanolamine, which have unusual and entirely unexpected wetting-out properties in themselves, may be produced by heating caprylic acid, its esters, halides or anhydrides or any other derivative which will condense with triethanolamine to form caprylic acid esters therewith, in the open atmosphere, under vacuo or in the presence of an inert gas such as carbon dioxide or nitrogen. We have, however, obtained especially satisfactory results by employing the reaction conditions described in our Patent No. 2,173,448, issued September 19, 1939, and it will, therefore, be understood that such represents a definitely preferred embodiment. As pointed out hereinabove, the caprylic acid esters of triethanolamine which are employed in the present invention are partial esters, that is, esters containing at least one, and preferably two, free or unesterified triethanolamine hydroxy groups.

As illustrative of methods of preparing the caprylic acid esters of triethanolamine, we admixed 1350 grams of 98% triethanolamine, 864 grams of technical caprylic acid, and 0.8 gram of potassium hydroxide, heated the mixture to about 125 degrees C. and then passed steam at 100 degrees C. directly into the reaction mixture, heat being applied to the latter to maintain it within the range of 170 degrees C.-250 degrees C. for 1½ hours. The reaction mixture was then cooled very quickly by immersion of the reaction flask in an ice bath, the passage of steam into the reaction mixture being continued until a temperature of about 125 degrees C. was reached. The steam was then shut off but the quick cooling or refrigeration was continued until a temperature of about 60 degrees C. or 70 degrees C. was reached after which the product was allowed to cool in the atmosphere. The final product comprised in the main the mono-caprylic acid ester of triethanolamine and was an oily, medium amber colored liquid. It may be washed with water to free it of excess or unreacted triethanolamine and may be salted out. If desired the caprylic ester of triethanolamine may be removed from the reaction mass by distillation in vacuo.

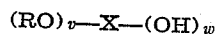
It will be appreciated that it is not necessary to employ pure triethanolamine or pure caprylic

acid, commercial sources of such compounds being entirely satisfactory. Furthermore, instead of using the free acid, esters thereof may be employed in the reaction, for example, mono-caprylin, dicaprylin, and the like. In other cases, acyl halides of caprylic acid may be employed, it being understood that, in such case, the reaction is carried out in the absence of moisture and preferably in the presence of a basic material to absorb the evolved halogen acid. In still other cases, the anhydride of caprylic acid may be utilized to produce caprylic acid esters of triethanolamine in accordance with general reaction conditions known in the art.

The caprylic acid partial esters are basic in reaction and may, therefore, be neutralized with acids or acidic materials of diverse character for various purposes such as, for example, to increase their water-solubility. Such acids and acidic substances include hydrochloric acid, sulphuric acid, phosphoric acids, tartaric acid, citric acid, malic acid, hydrobromic acid, lactic acid, phthalic acid, oleic acid, stearic acid, lauric acid, and the like.

In accordance with the novel features of our present invention as indicated hereinabove, we have found that the addition of certain materials, hereinafter described, which in themselves have substantially no wetting-out properties, to aqueous-containing baths markedly enhances the wetting-out properties of the caprylic acid esters of triethanolamine.

These addition or enhancing agents are the relatively high molecular weight esters of aliphatic polyhydroxy substances containing at least one free hydroxy group attached to the polyhydroxy nucleus. These substances may be represented by the general formula



wherein R is an acyl radical containing at least six carbon atoms, X is the carbon-hydrogen residue of an aliphatic polyhydroxy substance, and v and w are small whole numbers. The term "carbon-hydrogen residue" is here used in its ordinary chemical sense. Thus, for example, where glycerin, a polyhydroxy substance, is reacted with a fatty acid, such as lauric acid, to produce a mono-ester, the "carbon-hydrogen residue" of the polyhydroxy substance is



Of this class of compounds, we have found to

be particularly suitable those wherein the acyl radical contains from between six and fourteen carbon atoms and wherein the ratio of w to v is at least one. We have found that the mono-oleic acid ester of diethylene glycol, the coconut oil mono-fatty acid esters of diethylene glycol, the coconut oil mono-fatty acid esters of glycerin, and mono-caprylin are particularly efficacious for our present purposes. For a further disclosure of the general type of agents which we have found to enhance the effect or potency of certain classes of wetting-out agents, reference may be had to Reissue Patent No. 19,425.

In order to show the enhancing effect upon the wetting-out properties of caprylic acid esters of triethanolamine, we have utilized the Draves test, previously referred to, as a measure of said wetting-out properties. This test, as is well known, comprises suspending a skein of unscoured cotton of predetermined character and weight in a cylinder of water, a small weight attached to a string being affixed to the bottom of said skein. The length of time required for the skein to sink is taken as a measure of the potency of the particular wetting-out agent which has been dissolved or dispersed previously in the water in the cylinder, the shorter the length of time the more potent the wetting-out effect.

The addition of 0.1% of the monocaprylic acid ester of triethanolamine, prepared as described hereinabove employing the steam treatment, to water gave a sinking time of 1 minute and 25 seconds in the Draves test. By adding to water 0.075% of the caprylic acid ester of triethanolamine and 0.025% of monocaprylin, making a total of 0.1%, the sinking time was reduced to 57 seconds. The addition to water of 0.1% of monocaprylin gave no wetting-out test after 30 minutes, showing that the action of the monocaprylin is purely that of an enhancing agent. In other words, while the enhancing agent itself possesses substantially no wetting-out properties, it acts to increase markedly the wetting-out action of the caprylic acid esters of triethanolamine, notwithstanding the fact that the concentration or amount of the wetting-out agent is diminished.

Other Draves tests made with the monocaprylic acid ester of triethanolamine, prepared as described above, were as follows: 0.05% of the mono-caprylic acid ester of triethanolamine and 0.05% of the monolauric acid ester of triethylene glycol gave a sinking time of 1 minute and 12 seconds; 0.066% of said mono-caprylic acid ester of triethanolamine and 0.034% of the monolauric acid ester of triethylene glycol gave a sinking time of 1 minute and 15 seconds; 0.075% of said monocaprylic acid ester of triethanolamine and 0.025% of the monolauric acid ester of triethylene glycol gave a sinking time of 55 seconds. The monolauric acid ester of triethylene glycol itself, using 0.1%, gave a sinking time of several minutes.

It will be appreciated that the ratio of the caprylic acid esters of triethanolamine to the enhancing agents are subject to wide variation. It is particularly desirable, however, to employ the enhancing agent in proportions less than that of the triethanolamine ester.

While we have chosen the Draves test as a convenient means of illustrating the effect of these enhancing agents, the fact is that said enhancing agents also radically influence other valuable colloidal characteristics of the medium into which they are introduced.

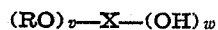
It will be apparent that neither the caprylic acid esters of triethanolamine nor the enhancing agents need be employed in the pure state and it will also be clear that admixtures of said esters with mixtures of different enhancing agents may likewise be utilized without departing from the spirit of our invention.

The enhancement of the wetting-out effect of caprylic acid esters of triethanolamine may be taken advantage of in treating baths containing an aqueous medium, with or without an additional substance, such as, for example, alkali, mordants, dyes, color discharging reagents, hydrogen peroxide, color reducing agents, and other reagents or substances commonly used in treating baths, and the treating bath so formed may be employed with improved results in many of the arts in which interface modification or wetting-out action is desired. For example, dyeing, bleaching, scouring, and otherwise treating fabrics and fibers and other materials in the treatment baths of this character are productive of excellent results. Also in the stuffing of leather, dyeing and otherwise treating furs, and in many other arts, a treating bath employing the materials of our invention may be used. In the flotation of ores, our invention may be used in connection with other reagents to modify the interface between the finely divided ore and the aqueous medium. The products of our invention may also be employed with effective results in cosmetic preparations such as cold creams, vanishing creams, lipsticks, skin lotions, and shaving creams of all types.

This application is a division of our earlier application, Serial No. 229,094, filed September 9, 1938, now Patent No. 2,239,997, issued April 29, 1941.

What we claim as new and desire to protect by Letters Patent of the United States:

1. Wetting-out compositions comprising a member selected from the group consisting of caprylic acid partial esters of triethanolamine and salts thereof, and including a proportion of a chemical compound having the formula



wherein R is an acyl radical containing at least six carbon atoms, X is the carbon-hydrogen residue of an aliphatic polyhydroxy substance, and v and w are small whole numbers.

2. The composition of claim 1, wherein R is a fatty acid acyl radical containing from 6 to 18 carbon atoms.

3. Wetting-out compositions comprising a member selected from the group consisting of caprylic acid mono-esters of triethanolamine and salts thereof, and including a lesser proportion of a fatty acid partial ester of a member selected from the group consisting of glycerol and glycols, the fatty acid radical of said partial ester containing from 6 to 18 carbon atoms.

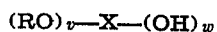
4. Wetting-out compositions comprising a member selected from the group consisting of caprylic acid mono-esters of triethanolamine and salts thereof, and including a proportion of a caprylic acid mono-ester of a member selected from the group consisting of glycerol and glycols.

5. Wetting-out compositions comprising (1) a member of the group consisting of the free reaction products and salts of reaction products having a light color and being characterized by substantial freedom from odor and resulting from the reaction of triethanolamine and a member selected from the group consisting of caprylic

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acid and esters thereof, at temperatures in excess of 100 degrees C. while passing steam through the reaction mass, and then rapidly cooling the reacting mixture while maintaining a steam atmosphere at least until partial temperature reduction has been effected, and (2) a proportion of a chemical compound having the formula



wherein R is an acyl radical containing at least six carbon atoms, X is the carbon-hydrogen residue of an aliphatic polyhydroxy substance, and v and w are small whole numbers.

6. Wetting-out compositions comprising (1) a

member of the group consisting of the free reaction products and salts of reaction products having a light color and being characterized by substantial freedom from odor and resulting from the reaction of triethanolamine and a member selected from the group consisting of caprylic acid and esters thereof, at temperatures in excess of 100 degrees C. while passing steam through the reaction mass, and then rapidly cooling the reacting mixture while maintaining a steam atmosphere at least until partial temperature reduction has been effected, and (2) monocaprylin.

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