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EFFLORESCENT COMPOSITION

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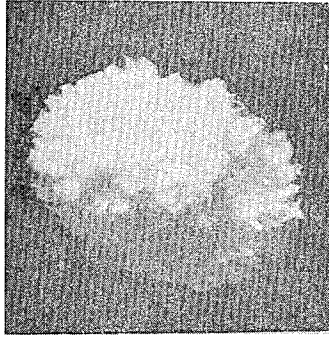


FIG. 1

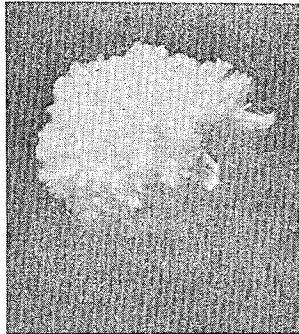


FIG. 2

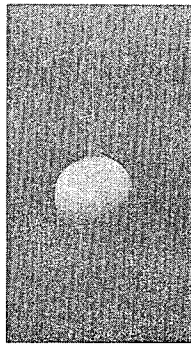


FIG. 3

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EFFLORESCENT COMPOSITION

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3 Claims

ABSTRACT OF THE DISCLOSURE

An efflorescent composition consisting essentially of an intimate mixture of (1) a substantially saturated aqueous solution of urea or of the sodium salt of a saturated fatty acid having from 6 to 10 carbon atoms in the carbon chain or of a mixture of urea and said fatty acid sodium salt, and (2) colloidal silicic acid.

This invention relates to a novel efflorescent composition, and more particularly to a shapable, coherent, initially smooth composition which, upon exposure to the air, slowly forms a growth of monocrystals which resemble hair.

It is known from British Patent 916,570 and French Patent 1,297,746 that a growth of monocrystals resembling hair may be produced on the surface of a hydrous soap composition containing a minor amount of sodium oleate, by incorporating into such soap compositions or applying to the outer surface of shaped articles made of such soap compositions a suspension of a gel, a sol or a solution of a gel-forming substance, and then exposing the soap composition to the air. The hair-like monocrystals which slowly grow out of the soap composition consist mainly, if not exclusively, of sodium oleate and reach a length of 1 to 1.5 cm. at the most. Thus, in order to produce a hair-like efflorescence on shaped articles in accordance with the prior art, it is necessary that the material on which the crystal growth forms be hydrous soap containing sodium oleate. Moreover, the monocrystalline growth is relatively short and not always uniform in density and length.

It is an object of the present invention to provide an efflorescent composition which does not require hydrous soap as a substrate.

Another object of the invention is to provide an efflorescent composition which, upon exposure to the air, forms a dense, uniform growth of relatively long hair-like monocrystals, not only on the surface thereof but throughout.

Still other objects and advantages of the instant invention will become apparent as the description thereof proceeds.

I have discovered that the above objects and advantages are achieved by a composition consisting essentially of an intimate mixture of (1) a solid powdered colloid, preferably colloidal silicic acid, and (2) a saturated or nearly saturated aqueous solution of urea or of a sodium salt of a saturated, relatively short-chain fatty acid whose carbon chain contains from 6 to 10, preferably 8, carbon atoms or of a mixture of urea and said fatty acid sodium salt. Thus, unlike in the case of the efflorescent compositions described in said British and French patents, the solid substrate in the composition according to the invention is not a commercial hydrous soap but rather a powdered colloid. The highly concentrated aqueous solution of urea or of the fatty acid sodium salt or of a mixture thereof acts as a binder for the dust-fine particles of the dry colloid powder, so that a plastic, barely shapable coherent mass is formed. Upon exposure to the air, a composition according to the present invention forms a monocrystalline growth resembling hair on the surface thereof, which

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reaches its maximum degree of efflorescence after one to two days, depending upon the ambient temperature and relative humidity.

The advantage of using urea or the sodium salt of a fatty acid of 6 to 10 carbon atoms or a mixture thereof as the efflorescent ingredient is not only that these substances are sufficiently soluble in water and form a heavy and decorative crystal growth, but also that they are odorless, non-poisonous and non-flammable and that their saturated or nearly saturated aqueous solutions are good binding agents for the powdered colloid ingredient.

I have further discovered that the appearance of the crystal growth may be influenced by varying the quantitative ratio of urea and fatty acid sodium salt. For instance, if the saturated or nearly saturated aqueous solution contains exclusively or predominantly a sodium salt of a fatty acid of 6 to 10 carbon atoms, the crystal growth is fine and somewhat curly and takes on the appearance of so-called "angel hair." On the other hand, if the aqueous solution contains predominantly urea, the crystal growth is largely in the form of long, straight "hair." Finally, if the solution contains exclusively urea as the efflorescent substance, the crystal growth is relatively short and coarse.

I have also determined that the appearance of the crystal growth may be influenced by adding to the saturated or nearly saturated aqueous solution of urea and/or the fatty acid salt minor amounts of a liquid organic substance which alters the surface tension or the interface tension of the aqueous solution. This class of liquid organic substances includes water-soluble anion-active wetting agents of the fatty alcohol sulfate and arylalkyl-sulfonate type, as well as a number of water-insoluble or sparsely soluble oils, such as dioctylphthalate, nonylphenol, dodecylphenol, liquid fatty alcohols and their esters, ethereal oils and perfume mixtures containing the same, and especially silicone fluids. For instance, the addition of nonylphenol causes the crystalline growth to be very fine and curly with very little sheen, somewhat similar to surgical cotton. On the other hand, the addition of a silicone fluid causes the crystalline growth to be particularly long, fine and virtually straight with a high sheen. With the aid of a surfactive additive of the type described above, it is also possible to obtain a very long and fine crystal growth from efflorescent compositions according to the present invention containing only urea as the efflorescent ingredient; some of these additives produce urea crystals which resemble surgical cotton, while others cause the urea crystals to be in the form of extended, fine, shiny fibers. As pointed out above, the efflorescing crystals of compositions according to the invention comprising only urea as the efflorescent ingredient take the form of relatively short, coarse needles when they are not modified with surfactive additives.

Efflorescent compositions pursuant to this invention in which the aqueous solution contains predominantly or exclusively a sodium salt of a fatty acid of 8 to 10 carbon atoms also exhibit the following surprising property: After the exterior crystalline growth has attained its maximum degree of efflorescence, the composition mass itself begins to expand due to the formation of an internal aggregate or felt-like system consisting of fine, hair-like crystals and colloid particles. When this latter process has reached its conclusion approximately four days after initial exposure to the air, the volume of the crystalline mass is from twenty to thirty times greater than the original volume of the composition before exposure to the air. For instance, when an efflorescent composition of this embodiment is shaped into a sphere having a diameter of 3 cm. and this sphere is exposed to the air under normal conditions for about

four days, a fluffy pompon with a silky sheen having a diameter of 8 to 9 centimeters is obtained.

On the other hand, if the composition contains predominantly or exclusively urea as the efflorescent ingredient, the composition itself does not expand in volume after the exterior crystal growth has reached its maximum degree of efflorescence, but instead undergoes a certain degree of shrinkage while the exterior crystal growth proceeds.

The hair-like crystal growth formed on a composition according to the present invention is essentially colorless. However, by means of an additional modification colored crystals may be caused to grow, namely, by neutralizing an organic basic dye, such as Astra Blue Base 4R (C.I. Solvent Blue 54), with a fatty acid of 8 to 10 carbon atoms, or by dissolving an organic acid dye, such as Benzobraun M (C.I. 22311 Direct Brown 2) or Sirius Lichtgelb (C.I. Direct Yellow 108), in a concentrated (about 40%) aqueous urea solution, and adding the dyes modified in this manner in small quantities to the saturated or nearly saturated solution containing the efflorescent ingredient which is later admixed with the dry colloidal substance to form the efflorescent composition.

In order to enhance the decorative and aesthetic effect, the efflorescent composition, which is colorless, may be modified by the addition of pigment dyes and perfume.

The efflorescent composition according to the present invention has utility in that it may be used for the manufacture of decorative efflorescent objects, especially for use at Christmas time and similar festive occasions.

The following examples further illustrate the present invention has utility in that it may be used for the understand it more completely. It should be understood, however, that the invention is not limited solely to the particular illustrative examples given below.

EXAMPLE 1

The following ingredients were charged into a mechanical mixer:

- 100 parts by weight of an aqueous solution of sodium caprylate containing 35% by weight caprylic acid, based on the total weight of the solution.
- 55 parts by weight of dry, dust-fine silicic acid powder obtained by precipitating sodium silicate from aqueous solution with a mineral acid, separating the precipitate by vacuum filtration, washing the filter cake with water, and then drying and grinding the filter cake.
- 1.2 parts by weight of pine-needle perfume.
- 0.1 part by weight of the pigment dye Indanthrene Brilliant Orange GR.

The ingredients were thoroughly admixed with each other, whereby a loose, pink-colored powder was obtained. The powder was rolled twice with the aid of a three-roller rolling mill of the type used in the manufacture of soap. The composition thus obtained was an intensively colored, viscous, plastic granulate from which compressed cylindrical blanks 28 mm. in diameter and 25 mm. in length were manufactured with the aid of a tablet press. The blanks were then shaped into spheres having a diameter of 30 mm. with the aid of a mechanical molding device, and each sphere was attached to a length of string.

Immediately thereafter, the spheres with string attached were individually hermetically sealed in polyethylene bags. Thus packaged, the spheres remained unchanged over a period of several months because the airtight envelope prevented evaporation of water.

When the spheres were removed from the polyethylene bags and suspended by the string from a suitable support so as to be exposed to the air all around, as shown in FIG. 3 of the attached drawing, snow-white silky crystals resembling hair begin to form on the surface of the spheres. After these crystals had attained a length

of about 20 mm., the color began to show through from the felt-like substrate with which had formed in the spheres themselves. At 20° C. and 60% relative humidity the efflorescent process was complete after about four days. The fluffy pompons formed thereby had an exterior diameter of 80-90 mm., and appeared as shown in FIGS. 1 and 2 of the attached drawing.

EXAMPLE 2

100 gm. of a mixed aqueous solution consisting of 75 gm. of an aqueous 35% solution of sodium caprylate and 25 gm. of an aqueous 45% solution of urea were admixed with 55 gm. of powdered silicic acid prepared as described in Example 1. The resulting mixture was combined with 0.5 gm. of a solution of 1 gm. of Astera Blue Base 4R (C.I. Solvent Blue 54) in 10 gm. of caprylic acid and with 3.0 gm. of silicone oil AR 350. The composition was then thoroughly mixed and rolled, and was thereafter pressed into cubes 2.5 cm. on the side. The cubes were immediately packed into individual sealed polyethylene bags. When the cubes were removed from the bags and suspended by a string so as to expose them to the air all around, a surface crystal growth in the form of blue, curly "hair" began to form, which ultimately reached a length of 2 to 3 cm. Thereafter, the body of the cubes swelled into a felt-like aggregate, so that after about two to three days of exposure to the air the cubes had changed into fluffy blue pompons of about 80 mm. in diameter.

EXAMPLE 3

100 gm. of an aqueous 35% solution of urea were shaken at room temperature with 50 mgm. of Benzobraun (C.I. 22311 Direct Brown 2), and then 1.5 gm. of coconut fatty alcohol sodium sulfonate were dissolved therein. Thereafter, the resulting solution was uniformly worked into a mixture consisting of 55 gm. colloidal silicic acid, 1.5 gm. lavender perfume and 2.0 gm. dioctylphthalate. The resulting mixture was pressed into tetrahedrons weighing about 20 gm. each. Upon exposure to the air the tetrahedrons sprouted a growth of very curly, pinkish-brown, hair-like crystals which attained a length of up to 8 cm.

When the acid dye was omitted the hair-like crystals growing out of the tetrahedrons were even longer and very much straighter.

EXAMPLE 4

70 mgm. of Sirius Lichtgelb (C.I. Direct Yellow 108) were dissolved in 100 gm. of a mixture consisting of 40 parts by weight of an aqueous 33% sodium caprylate solution and 60 parts by weight of an aqueous 40% urea solution. The resulting solution was homogeneously admixed with 50 gm. of powdered colloidal silicic acid which contained 2% by weight of dodecylphenol as the surfactive ingredient and 1% by weight of phenethyl alcohol as the perfume ingredient. The resulting mixture was pressed into variously shaped bodies, and these were exposed to the air. Hair-like light yellow crystals grew out of the surface of this composition; the crystal growth was similar to surgical cotton in texture and occurred in the form of numerous tufts all over the surface.

While the present invention has been illustrated with the aid of specific embodiments thereof, it will be readily appreciated that my invention is not limited to these particular embodiments, and that various changes and modifications may be made without departing from the spirit of the invention or the scope of the appended claims.

I claim:

1. A shapable, coherent efflorescent composition consisting essentially of an intimate mixture of (1) a substantially saturated aqueous solution of a member of the group consisting of urea, sodium caprylate and mixtures thereof, and (2) colloidal silicic acid powder, the weight ratio of (1) to (2) in said composition being about 2:1.

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2. A shapable, coherent efflorescent composition according to claim 1, wherein said substantially saturated aqueous solution contains about 0.1% by weight of a pigment dye and about 1.0% by weight of a perfume.

3. A shapable, coherent efflorescent composition according to claim 1, wherein said substantially saturated aqueous solution contains about 1 to 3% by weight of a liquid surfactive agent.

References Cited

UNITED STATES PATENTS

2,383,653 8/1945 Kirk ----- 252—313

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FOREIGN PATENTS

916,570 1/1963 Great Britain.

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