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2,799,599

## LUSTERED FUR HAIRS AND METHOD FOR PRODUCING SAME

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This invention relates particularly to a new and improved method of treating furs and a new and improved composition for such purpose, and a new and improved coated fur or other material. The invention relates particularly to the lustering of fur coats. The invention is not limited to the treatment of furs, as it applies to the treatment of other fiber materials and even non-fiber materials.

In cleaning and renovating a fur coat, it is common practice to clean the coat, and then to apply various oil and wax compositions to the hair side of the coat, and then to treat the fur coat by heating it, preferably under pressure, and also while simultaneously combing the fur hairs.

The purpose of such treatment is to increase the gloss and texture of the fur hairs and to water-proof them. This operation is generally known as lustering.

According to one feature of my invention, I replace the vegetable and mineral oils and the waxes which have been used for this purpose, either in whole or in part, by an organo-silicon polymer, which is preferably a silicone. This silicone is preferably a liquid at normal room temperature of 20° C.-25° C.

The silicones are well-known poly-organo-siloxanes which contain the unit—R<sub>2</sub>SiO—, in which "R" represents an alkyl or aryl group. These silicones and other organo-silicon polymers are described in many standard text-books, such as "A Text-Book of Organic Chemistry" by Schmidt & Rule, published in 1947 by D. Van Nostrand Company Inc. These organo-silicon polymers or polyorganosiloxanes may contain the group R(H<sub>2</sub>Si—O—)<sub>x</sub> and the invention includes polyorganosiloxanes other than the silicones, which have the desired property.

These organo-silicon compounds may be made by combining silicon dioxide with methyl or ethyl groups derived from alcohols, or with ethylene chloride or phenol. Both straight-chain or ring-type organic molecules may be combined with the silicon dioxide. These materials are stable, inert, and strongly water-repellent.

One illustrative example of my invention, to which the invention is not limited, is stated below in which the proportions are by weight.

## Example

Ingredient:	Parts
A. Silicone -----	10
B. Beeswax -----	5
C. Stearic acid -----	10
D. Morpholine -----	3
E. Water -----	50
F. Added water -----	25
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Ingredient A is a silicone which has a viscosity of one hundred centipoises at ordinary room temperature of 20° C.-25° C. It is a well-known commercial product. It is a pourable liquid at 20° C.-25° C.

Ingredient C may be ordinary commercial stearic acid. The general formula of stearic acid is C<sub>18</sub>H<sub>36</sub>O<sub>2</sub>. The

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stearic acid exemplifies a class of fatty acids which preferably have at least thirteen carbon atoms and which are solids at 20° C.-25° C.

Hence this preferred class includes, as additional examples of such preferred class which can be used for the purposes of my invention, the following:

Tridecylic acid, CH<sub>3</sub>(CH<sub>2</sub>)<sub>11</sub>COOH, whose melting point is 51° C.

Myristic acid, CH<sub>3</sub>(CH<sub>2</sub>)<sub>12</sub>COOH, whose melting point is 58° C.

Pentadecylic acid, CH<sub>3</sub>(CH<sub>2</sub>)<sub>13</sub>COOH, whose melting point is 54° C.

Palmitic acid, CH<sub>3</sub>(CH<sub>2</sub>)<sub>14</sub>COOH, whose melting point is 64° C.

Margaric acid, CH<sub>3</sub>(CH<sub>2</sub>)<sub>15</sub>COOH, whose melting point is 59.9° C.

Arachidic acid, CH<sub>3</sub>(CH<sub>2</sub>)<sub>20</sub>COOH, whose melting point is 77° C.

Behenic acid, CH<sub>3</sub>(CH<sub>2</sub>)<sub>20</sub>COOH, whose melting point is 84° C.

I can also use hydrogenated fatty acids.

Ordinary commercial stearic acid usually contains another fatty acid.

The stearic acid of commerce which I can use contains 45% of stearic acid and 55% of palmitic acid and I may use a grade of commercial stearic acid which has a melting point of 53° C.-55° C.

Ingredient B, which is optional, exemplifies a class of waxes which are preferably solids at 20° C.-25° C., such as Japan wax, montan wax, candelilla wax, and many others. These other waxes may be substituted for the beeswax. I can use waxes which are not saponified in the composition, or which are wholly or partially saponified therein.

Morpholine, O:(C<sub>2</sub>H<sub>4</sub>):NH, is an amine, which like other amines, produces salts or soaps with long straight chain fatty acids. One mole of morpholine reacts with one mole of the fatty acid to produce the respective salt or soap with the formation of one mole of water as a by-product.

Morpholine is strongly basic. It is freely miscible with water, boils at 128° C. and it is a good emulsifying agent.

The stearic acid may be fused at 60° C. and maintained fused at this temperature.

An aqueous solution of morpholine, which is also at 60° C., is added slowly to the fused stearic acid, with constant stirring, while the temperature is maintained at 60-70° C. The reaction is exothermic. This will form the morpholine-stearic acid salt or soap which is the water soluble neutral salt. This soap is dissolved in the water of the aqueous solution.

If the aqueous solution of this morpholine-stearic acid soap is heated up to 130° C., in air under normal pressure of 760 millimeters of mercury, the solution is evaporated and the high temperature decomposes the dissolved soap, the morpholine is liberated and evaporated, and the stearic acid is substantially regenerated, remaining as a dry and water-insoluble residue.

In one test, 100 grams of said grade of stearic acid were fused, and this fused stearic acid was reacted under the above mentioned conditions with 33 grams of morpholine, which had been dissolved in 133 grams of distilled water. The weight of the aqueous morpholine-stearic acid soap solution was 266 grams.

This solution was gradually evaporated at said temperature by heating from 110° C. up to 130° C., under said standard pressure. The weight of the residue was 104 grams.

The colloidal soapy mass of the original morpholine stearic acid soap solution, was converted to a clear oily liquid at 130° C., which is above the boiling point of

morpholine. In the last stage of the reaction, there is no odor of morpholine. This residue was substantially the original stearic acid.

While the invention is not limited to the use of a morpholine soap in the composition which is applied to the fur or other material, the use of a morpholine soap is preferred for practical purposes, because it is particularly selective and effective for the desired purpose. The morpholine which is liberated from the decomposed soap is readily volatile at moderate temperature.

If beeswax is used, this contains a substantial percentage of compounds which react with the morpholine to produce additional soaps. I can use either the yellow beeswax of commerce or the white beeswax which is described at page 1282 of the 1947 edition of *The Dispensary of the United States of America*. Beeswax contains a substantial percentage of cerotic acid. It may have an acid value of 17 to 24. These beeswax soaps are in the composition which is applied to the fur.

While I prefer to use morpholine or other water-soluble volatile organic base which is readily volatile at the lustering temperature, the invention is not limited to the use of morpholine. Thus, the morpholine may be replaced by an equivalent amount of ammonia, alkyl and other amines, whose soaps or additional compounds are also similarly decomposed by heat.

The purpose of adding a water-repellant wax or waxes is to improve the body of the composition and the lustering effect and the water-proofing effect.

Other conditioning ingredients may be added, such as vegetable and mineral oils, paraffin, lecithin, lanolin, glycols, anti-static agents, etc.

According to one embodiment of making the composition, which is highly preferred, the anhydrous silicone and the anhydrous wax (if the wax is used) are mixed with the fused anhydrous stearic acid at about 65° C., if said grade of commercial stearic acid is used, until a homogeneous fluid and initial fused mixture is formed which is anhydrous or substantially anhydrous.

The three parts of morpholine are separately dissolved in the 50 parts of water.

This aqueous morpholine solution is slowly added to said initial fluid mixture with constant stirring, while maintaining a temperature of 65–70° C. This may be done in air at normal pressure of 760 millimeters of mercury. This produces a stable composition. The aqueous solution of the morpholine or other base, when added, is preferably at the same temperature as the initial fluid mixture.

I can use a molar proportion of morpholine to combine with all the fatty acid or all of the mixture of fatty acids, or I can use a greater or less proportion than the molar proportion.

Thus, in the specific example, a substantial part of the morpholine reacts with the beeswax to produce a soap or soaps so that a substantial proportion of the stearic acid, or of the mixture of fatty acids in the commercial stearic acid, remains in uncombined form in the composition or is in the form of an acid soap. This is a stable emulsion or dispersion in which the uncombined acid or acids and other uncombined materials are finely dispersed. This composition is a soft paste at 20° C.–25° C.

This composition may be diluted with the additional 25 parts of water, by stirring at 20° C.–25° C.

If additional agents, such as oils, lecithin, etc. are used, these are added to the initial fused mixture, prior to adding the aqueous solution of morpholine or other amine or ammonia thereto.

Optionally, the silicone is mixed with the fatty acid material at 60° C., to form an initial fluid mixture, the aqueous solution of morpholine is added thereto at 60° C. to make an initial emulsion, and this initial emulsion is mixed with a separately made aqueous emulsion of the beeswax, oils, lecithin, etc. This separately made emul-

sion may be made with a part of the stearic acid material and a part of the morpholine.

The molecular proportion of the morpholine may be from 50% to 100% of the total fatty acid material which is reacted with the morpholine. This proportion may be calculated upon the entire weight of the material which reacts with the morpholine, including the reactive material of the beeswax. This applies if the morpholine is replaced by an equivalent material. In either case, the non-soap ingredients, including some of the stearic acid, are dispersed in the emulsion.

Various pre-formed aqueous silicone emulsions are on the market, and such pre-formed aqueous emulsions can be used, if compatible.

A composition which is thus made, is applied to the cleaned fur, as by brushing at 20° C.–25° C. The composition is applied to the base of the fur and its hairs or to the hairs alone.

The fur is then heated, optionally pressed under heat or combed under heat and pressure, using well-known hand appliances or machinery for this purpose. This is done in an atmosphere of air under normal pressure of 760 millimeters of mercury. Any conventional lustering procedure may be used.

The temperature of the fur and of the applied aqueous composition is gradually raised. Before the water of the composition is wholly evaporated, the soap is decomposed, and the fatty acid or fatty acids are regenerated and the morpholine is liberated and it is wholly evaporated.

A thin coating of the composition may be applied to the fur hairs, which may be simultaneously combed and heated to decompose the morpholine soap and to drive off the morpholine, and to regenerate the stearic acid or other fatty acid.

There is little or no loss of the silicone. The silicone and the deposited water-insoluble fatty acid or acids and other water-insoluble deposited material provide a very intimate and stable coating on the base and hairs of the fur. The particles of silicone are coated with the other material or materials, which act as a binder. The result is much superior than if the silicone or an ordinary aqueous emulsion thereof is applied by brushing. The regenerated stearic acid or other fatty acid material, when thus formed and deposited in situ, acts as a vehicle and binder which is intimately physically combined with the silicone, and this intimate physical mixture is very adherent and stable and it minimizes the rubbing off of the silicone.

While oleic acid can be used as the fatty acid material or as a part thereof, it is highly preferable to use fatty acid material which has a melting point above 25° C., and which preferably has a melting point of at least 50° C., so that it remains unfused and does not soften under ordinary conditions of wear.

When the composition is used to luster a fur, the fur hairs retain their original color.

While it is preferred to use a silicone as a lustering water-proofing agent, the invention applies to other water-proofing materials which may be oils, waxes, etc.

I believe that the coating composition has anti-static properties, so that the fur hairs do not clump because of static electricity.

As one example, the finished composition is a soft paste which is slightly pourable at 20° C.–25° C. In this example, the finished composition is an aqueous dispersion of a water-insoluble organo-silicon polymer, in which the dispersing agent is a water-soluble morpholine soap which is decomposed when said composition is heated in order to liberate and drive off the morpholine and to combine the acid radical of said soap with water of said dispersion, in order to regenerate the original acid which corresponds to said acid radical. This polymer is sub-

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stantially non-volatile at the temperature which is required to decompose said soap.

I claim:

1. A combination method of lustering fur hair which consists in making a mixture of an oily silicone and a fused, free fatty acid, said free fatty acid having at least 13 carbon atoms in its chain, mixing said mixture while said free fatty acid is in fused form with an aqueous solution of morpholine to react only a part of said fused fatty acid with the dissolved morpholine in said aqueous solution to form the morpholine soap of said fatty acid in a coating product which is a soft aqueous paste, enough of said free fatty acid being left unreacted and enough water being provided in said aqueous solution to provide said soft aqueous paste, applying said soft aqueous paste to the fur hair, and heating and combing said aqueous paste on said fur hair to evaporate its water and to decompose said morpholine soap and to regenerate the free fatty acid thereof and to drive off the morpholine which results from the decomposition of the morpholine soap before all the water of said aqueous paste is evaporated, said aqueous paste being thus heated at a temperature at which said fur hair retains said oily silicone and the free fatty acid in said paste and said regenerated fatty acid.

2. A combination method of lustering fur hair, which consists in making a substantially anhydrous mixture of an oily silicone and fused beeswax and fused free stearic acid, said mixture consisting of substantially 10 parts of said silicone and substantially 5 parts of beeswax and substantially 10 parts of free stearic acid, mixing said mixture with an aqueous solution of substantially 3 parts of morpholine in substantially 50 parts of water at substantially 65° C.-70° C. to form a coating product while reacting a part of said morpholine with only a part of said free and fused stearic acid and while reacting the remainder of said morpholine with a part of said fused beeswax to form the corresponding morpholine soap, enough water being used in said aqueous solution of morpholine to provide a coating product which is a soft aqueous paste at 20° C.-25° C., said coating product containing free stearic acid and also containing said morpholine-stearic acid soap and also containing said soap of morpholine formed from the saponifiable material of the beeswax, applying said soft aqueous paste to the fur hair, and heating and combing said aqueous paste in the fur hair to evaporate the water of said paste and to decompose said morpholine soaps and to regenerate the acidic ingredients of said soaps and to drive off the morpholine which results from the decomposition of said morpholine soaps before all the water of said aqueous paste is

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evaporated, said aqueous paste being heated at a temperature at which said fur hair retains said oily silicone and the unsaponified material in said paste and said regenerated acidic ingredients.

3. A method of lustering fur hair, which consists in applying a soft aqueous paste to the fur hair, said aqueous paste containing the morpholine soap of a fatty acid which has at least 13 carbon atoms and which is a solid at 25° C.-30° C., said paste also containing an oily silicone, said morpholine soap and said oily silicone being uniformly intermixed and dispersed in said aqueous paste, and combing and heating said aqueous paste on said fur hair to decompose said morpholine soap and to regenerate the acidic component of said soap and to drive off the morpholine which results from said decomposition and to evaporate the water of said paste, said morpholine soap being decomposed and said morpholine being driven off before the water of said paste is wholly evaporated said paste being thus heated at a temperature at which said oily silicone and said regenerated acidic component are retained on said fur hair.

4. Fur which has lustered fur hairs, said fur hairs having surface coatings which consist substantially of an intimate mixture of 10 parts of an oily silicone, substantially 5 parts of beeswax and substantially 10 parts of stearic acid, said stearic acid being the regenerated stearic acid of an aqueous solution of a morpholine soap of stearic acid decomposed in the presence of water on said fur hairs.

5. Fur which has lustered fur hairs, said fur hairs having surface coatings whose major ingredients is an oily silicone, said surface coatings also including the regenerated fatty acid of a morpholine soap, said fatty acid having at least 13 carbon atoms, said oily silicone and said regenerated fatty acid being in the form of an intimate mixture on said fur hairs, said regenerated fatty acid being the regenerated acid of an aqueous solution of a morpholine soap of said fatty acid and decomposed in the presence of water on said fur hairs.

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