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HEAT-TREATED MAGNESIUM SOAP
COMPOSITION

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The present invention relates to a method of heat-treating magnesium soaps and the resulting products and derivatives thereof.

The magnesium soaps of saturated fatty acids are insoluble in most of the common organic solvents. For example, magnesium stearate, the most important commercially of this class of compounds, is insoluble in methanol, ethanol, turpentine, benzene, toluene and the petroleum solvents such as petroleum ether, naptha and kerosene. This unfortunate characteristic greatly limits the commercial utilization of the magnesium soaps due to the resulting difficulties in applying the soaps to the materials under treatment, especially where a thin film of the soap is desired. In U. S. Patent No. 2,356,313 the soaps of divalent metals including magnesium are disclosed in admixture with petroleum solvents as low temperature lubricants. The patent teaches that these soaps may be dispersed in a solvent at room temperature or temperatures elevated "to about 100° F. or 150° F." It is further stated that while mixtures containing less than 30% of the soap tend to separate, this defect is not fatal for many lubricating purposes inasmuch as the metallic soap alone will have some lubricating effect.

German Patent No. 698,653 discloses the heat-treating of the soaps of di- and trivalent metals for several hours at temperatures between 100 and 250° C. prior to dissolving them in solvents to produce cleaning agents. It is stated that the ordinary metallic soaps of this type yield solutions in hydrocarbon solvents of higher viscosity than solutions formed from the same metallic soaps after the prescribed heat-treatment. The concentration of the heat-treated soaps in such solutions may be as high as 40% without the solutions becoming viscous or gelatinous according to this patentee. The only working example in this patent describes the solution of a heat-treated aluminum soap of train fatty acids. Although magnesium is included in the general list of suitable metals for the soap, it is doubtful that experiments were conducted with magnesium soaps of saturated fatty acids because they do not form solutions at 20° C., let alone viscous or gelatinous solutions, in solvents of the types mentioned. Moreover, the results obtained in the present invention do not correspond with the generalities set forth in the aforementioned German patent insofar as magnesium soaps are concerned for the lower limit of 100° C. does not appear critical for heat-treating purposes and clear gels or opaque pastes rather than thin solutions in hydrocarbon solvents were obtained at soap concentrations considerably lower than 40%.

An object of the invention is to provide a magnesium soap of improved properties.

A second object of the invention is to provide a substitute for ester waxes.

A third object of the invention is to provide a process for rendering magnesium soaps soluble in organic solvents.

A fourth object of the invention is to provide a process for rendering magnesium soaps more stable in solution.

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A fifth object of the invention is to provide improved polish bases and polishes.

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

5 The present invention is concerned with a process for heat-treating magnesium soaps of fatty acids by heating them in the molten state until a substantial reduction in viscosity of the material occurs. It also comprises the heat-treated magnesium soaps and mixtures thereof with waxes and/or liquid solvents of the group consisting of hydrocarbons and halogenated hydrocarbons.

10 The invention accordingly comprises the several steps and the relation of one or more of such steps with respect to each of the others thereof, which will be exemplified in the method hereinafter disclosed, and the scope of the invention will be indicated in the claims.

15 It has been discovered that unusual changes of a nature not yet understood occur when a magnesium soap is heated above its melting point for a period sufficient to cause a substantial reduction in the viscosity of the molten soap. Once this has been accomplished, the solubility characteristics of the heat-treated metallic soap are entirely different from the original material, at least where certain organic solvents are involved. These changes 20 may be due to a reorientation of the molecular arrangement of the magnesium soap or they may be due to the production of small amounts of solubilizing agents from the fatty acid radicals. In support of the latter theory, it is to be noted that the heat-treatment changes the color of white magnesium stearate powder to a light tan or amber, possibly as a result of slight or partial decomposition of the stearate radical; however, this has not been determined with certainty and we do not wish to be bound to any theory concerning the changes.

25 Many advantages accrue from the novel heat-treatment as it provides substitutes for expensive natural waxes and also permits the formation of stable solutions or gels of magnesium soaps in solvents in which untreated magnesium soaps are insoluble at room temperature and form 30 only unstable solutions, pastes or gels at substantially elevated temperatures. While the heat-treated soap may be substituted for an equal proportion of one of the hard, high-melting and costly natural waxes, such as carnauba and candelilla waxes, equally good, if not better, results are secured by mixing a cheap extender such as petroleum wax with the metallic soap. Another very desirable feature of the compositions disclosed herein is that polishes 35 consisting of the heat-treated magnesium soap in a solvent produce a transparent, shiny, continuous film without laborious rubbing or polishing. Thus, it is only necessary to apply a thin coating of the polish and allow the solvent to evaporate. Where color is a consideration, as is often the case for wax products, the light, almost neutral color of these heat-treated soaps is a distinct advantage.

40 In heat-treating magnesium soaps according to the present invention, it is necessary only to heat the soap to its melting point until the reduction in viscosity of the resulting liquid occurs. This conversion can be expedited by heating the soap to a temperature considerably above its melting point; however, it is best to keep the temperature below the point of substantial decomposition in order to avoid a product having a dark color or undesirable odor. The melting point is the critical 45 lower limit of the heat-treatment for heating magnesium soaps, for extended periods at temperatures only slightly below the melting point has failed to yield products having the striking qualities of those treated at or above the melting point. The soap may be heated either alone or in admixture with other substances such as solvents 50 or waxes. It is thought that the presence of such other materials slows down the conversion to some extent,

but this point has not been fully investigated. An advantage in heat-treating the metallic soap alone is that the change in viscosity is more readily observed than when diluents, such as waxes, are present. Once the soap has been heat-treated, it is not necessary to heat a mixture of this soap with a wax and/or a solvent to the heat-treating temperature in order to form a stable solution or mixture. However, it is generally desirable to use moderately elevated temperatures to expedite dispersing the soap in the wax or solvent.

As is apparent from the examples below, the unusual effects of the heat-treatment seem to be confined to magnesium soaps alone because similar results are not obtained with other polyvalent, water-insoluble metallic soaps. The magnesium soaps which are suitable for the purposes of the present invention include those having saturated fatty acid radicals containing from 8 to 22 carbon atoms or, more specially, the caprylic, capric, lauric, myristic, palmitic, stearic, arachidic and behenic acid soaps or mixtures thereof. Magnesium stearate is the preferred species and has been used throughout the examples to provide comparative results. Any of the conventional sources of these fatty acid radicals may be utilized in manufacturing the soaps in known manner from ordinary or hydrogenated animal, vegetable or marine oils, fats or waxes, or from fatty acids split therefrom.

The heat-treated magnesium soaps form stable gels or solutions with hydrocarbon and halogenated hydrocarbon solvents. Mixtures of these soaps and waxes have similar solubility characteristics. Suitable solvents include mineral spirits, naphtha, Stoddard solvent, benzene, toluene, xylene and terpene hydrocarbons such as alpha-pinene, beta-pinene, dipentene and turpentine. In addition, the halogenated hydrocarbons exemplified by methylene chloride, chloroform, carbon tetrachloride, and trichlorethylene, to name only a few, may be used. Mixtures of the aforementioned solvents are entirely satisfactory for most purposes. Where it is desired to heat-treat the magnesium soaps in situ, it is preferred to select a solvent having a boiling point of about at least 5° C. above the melting point of the soap in order to permit the use of simple equipment. However, the soap may be heat-treated in the presence of highly volatile solvents of low boiling point by employing pressure equipment to prevent excessive losses of the solvent at the prescribed temperature. Stable gels containing 10 to 100 parts by weight of heat-treated magnesium soap per 100 parts of solvent may be produced according to this invention but those having 10 to 35 parts of the soap appear to be the most useful.

The heat-treated magnesium soaps of the present invention are compatible with all of the hydrocarbon and ester waxes. These include, inter alia, ozokerite, paraffin and scale waxes as well as carnauba, candelilla, montan and like waxes. While a particular purpose of the invention is to provide new magnesium soaps to serve as extenders or substitutes for the high melting, hard and brittle carnauba wax, they may be blended with other waxes to adjust the properties of the mixture in regard to melting point, color and hardness. In particular, various mixtures of 100 parts by weight of wax and from 40 to 400 parts (60 to 200 parts being preferred) of heat-treated magnesium soap show great promise as substitutes for carnauba wax.

It is also possible to form stable gels by dispersing the above mixtures of heat-treated magnesium soaps and wax in one or more of the aforementioned hydrocarbon and halogenated hydrocarbon solvents. In making these gels, the soap-wax mixture is usually heated to its melting point in the presence of the solvent and an untreated magnesium soap may be heat-treated in situ. For each 100 parts by weight of solvent, from 15 to 100 parts of the soap-wax mixtures described in the preceding paragraph may be employed, but the range of 30 to 70 parts

of these solids in 100 parts of solvent is preferred for most purposes.

For a fuller understanding of the nature and objects of the invention, reference should be had to the following examples which illustrate the invention. All parts are given in terms of weight unless otherwise stated:

Example I

A sample of magnesium stearate which had been prepared by the precipitation method was gradually heated. At about 140° C. the white powder fused into a water-white viscous liquid. While being maintained at this temperature, the color gradually changed to light amber. After about 3 minutes it was observed that the consistency of the liquid thinned, then the fused mass was allowed to cool to room temperature. The product was a hard, brittle, translucent, waxy solid of a light amber color. Since this material was found to fracture in the same manner as carnauba wax, an empirical test for this feature was devised in which a thin knife blade $\frac{3}{16}$ of an inch wide was forced point foremost into the center of the material being tested to a maximum depth of 1 inch. Although the dimensions of the test samples varied from about $\frac{3}{4}$ to 2 inches, the size and shape of the sample appeared to have no effect on their fracturing characteristics. When the heat-treated magnesium stearate was tested in this manner, a sharp, clean fracture occurred.

Example II

	Parts
Fused magnesium stearate of Example I	15
Stoddard solvent (155–205° C. boiling range)	85

The particles of fused magnesium soap were stirred into the solvent and heated to approximately 150° C. for about 5 minutes. An almost clear yellowish solution of the consistency of water resulted. Upon cooling, a substantially transparent firm gel of an amber tinge formed and the solvent displayed no apparent tendency to sweat or bleed from the gel. This gel was applied to a smooth surface and quickly dried to an even, continuous, clear film of high luster without rubbing, thereby indicating its utility for polishes and polish bases.

Example III

	Parts
Magnesium stearate	15
Stoddard solvent	85

Example II was repeated using ordinary powdered magnesium stearate in place of the heat-treated variety and heating only to the 120 to 130° C. range. A hazy, colorless thin fluid was obtained. After cooling to room temperature, a firm whitish paste resulted and a slight separation of the solvent was noted. On storage at room temperature a large quantity of the solvent sweated out and the paste became granular and much more opaque. The opacity indicated separation of magnesium stearate from the paste.

Example IV

	Parts
Magnesium stearate	30
Petroleum solvent (149–210° C. boiling range)	70

The materials listed were heated to about 150° C. for approximately 7 minutes to heat-treat the magnesium stearate in situ, then the slightly hazy liquid was cooled as before. A very firm, slightly hazy, almost colorless gel was the product.

Example V

	Parts
Magnesium stearate	30
Petroleum solvent (149–210° C. boiling range)	70

These substances were mixed at room temperature to produce a white opaque suspension of the metallic soap, then heated to 65° C. for about 7 minutes. The mass

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was cooled to yield a white opaque paste of the consistency of butter.

Example VI

	Parts
Magnesium stearate.....	30
Petroleum solvent (149-210° C. boiling range).....	70

This batch was heated to 100° C. for 7 minutes and a paste similar to the product of Example V was obtained.

Example VII

	Parts
Magnesium stearate.....	20
Petroleum solvent 149/210° C.....	80

A white opaque thick suspension was obtained by heating the above ingredients to 65° C. for about 7 minutes and a white opaque paste resulted upon cooling.

Example VIII

	Parts
Zinc stearate.....	30
Petroleum solvent 149/210° C.....	70

The procedure of Example IV was carried out with these materials and a white opaque paste rather than a gel was produced.

Example IX

	Parts
Calcium stearate.....	30
Petroleum solvent 149/210° C.....	70

When the substances listed were treated according to Example IV, the opaque dispersion became substantially transparent at 150° C. and yielded a clear stiff gel. However, a very heavy seepage of solvent from the gel was observed indicating that the gel lacked the stability of the magnesium soap gel of Example IV.

Example X

	Parts
Barium stearate.....	30
Petroleum solvent 149/210° C.....	70

Upon repeating the process of Example IV with the above ingredients, the results were the same as in Example IX save that the bleed of solvent from the gel, although large, was somewhat less than from the calcium stearate gel.

From the results obtained in the above examples, it is clear that heat-treating magnesium stearate at a temperature above its melting point produces unusual results in connection with the solubility of this material in solvents and in the stability of the resulting gels. Similar results were not obtained in heating magnesium stearate to a temperature only 10 to 20° C. lower than the critical figure of 140° C. In most of the examples, heating to 150° C. was employed to maintain uniformity for purposes of comparison inasmuch as all of the metallic soaps tested are molten at that temperature. It is also significant that this conversion or change in magnesium soaps did not occur in other closely related polyvalent metallic soaps as stable gels were not formed.

Example XI

	Parts
Magnesium stearate.....	60
Crude white scale wax (51° C. M. P.).....	40

To the molten wax at 120° C. the powdered magnesium soap was gradually added with stirring and the mixture was a thick opaque white paste. The temperature was raised to 140° C. which resulted in a considerable reduction in viscosity accompanied by a clarification and slight yellowing of the fluid mass. These changes occurred over a period of 5 minutes. When allowed to cool, the product bore little resemblance to scale wax for it was a hard, brittle solid of light amber color and this material had a melting point of 116° C. (A. S. T. M. paraffin wax method) and split with a

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clean fracture when tested with a knife blade as in Example I. This contrasted sharply with the crude scale wax which did not split upon penetration of the knife blade. In comparison with carnauba wax, this new product is somewhat softer, much lighter in color, translucent instead of opaque, possesses a far higher melting point, but fractures in the same manner. It was apparent that the new composition could be substituted for the more expensive carnauba wax in many applications with superior results. 25 parts of the magnesium stearate-wax mixture were dissolved in 75 parts by weight of Stoddard solvent at a temperature of approximately 120° C. Upon cooling, the clear fluid mass became a slightly hazy, stiff gel which displayed no tendency to lose solvent. This gel had the characteristics of an excellent polish.

Example XII

	Parts
Lithium stearate.....	60
Crude white scale wax.....	40

The wax and lithium soap formed a very stiff, white, opaque paste when stirred together at 120° C. The temperature was gradually increased until the paste liquified and became clear at 210° C. In reaching this conversion point, there appeared to be a considerable decomposition of the lithium stearate and the fluid, although fairly transparent, was of a dark brown color. Upon cooling, a coffee colored opaque solid having a melting point of 193° C. was obtained. This material was softer than that of Example XI and did not fracture when tested with the knife blade.

Example XIII

	Parts
Zinc stearate.....	60
Crude white scale wax.....	40

This mixture was a white, opaque, water-thin fluid at 120° C. It was heated up to 200° C. without observing any change in viscosity or transparency. Cooling to room temperature yielded an opaque, grayish-white, waxy material. While it was found to be of substantially the same hardness as the product of Example XI, it did not split upon the penetration of the knife point and had a melting point of only 82° C. After dissolving 25 parts of this mixture of zinc soap and wax in 75 parts by weight of Stoddard solvent at 110° C., it was found that a precipitate formed during cooling, leaving a supernatant liquid.

Example XIV

	Parts
Barium stearate.....	60
Crude white scale wax.....	40

The initial thick, opaque, white paste formed by mixing the above substances turned brown while it was being heated gradually to a maximum of 190° C., thereby indicating that some decomposition had taken place. When cooled, the dirty-gray solid was found to have a melting point above 160° C. Although this material was harder than the product of Example XI, it did not fracture like that product or carnauba wax when tested with the knife mentioned in Example I. It was found that 25 parts of this material would not dissolve completely in 75 parts by weight of Stoddard solvent at 110° C. Upon cooling this mixture, it was observed that a heterogeneous mixture was obtained with a greater proportion of solids concentrated at the bottom than at the top.

Example XV

	Parts
Magnesium stearate.....	50
Crude white scale wax.....	50

The powdered stearate was stirred into the wax while the latter was molten at 120° C. The resulting mixture was a very thick, opaque white dispersion. The temperature was then increased and the mass maintained at or above 140° C. until the mixture thinned to medium vis-

cosity and became substantially transparent with only a slight haziness remaining. After cooling to room temperature, the resulting solid (melting point 105° C.) was observed to be practically identical in appearance but slightly softer than the product of Example XI. Similar fracturing characteristics were also found.

Example XVI

	Parts
Lithium stearate -----	50
Crude white scale wax -----	50

To the wax at 120° C., the powdered lithium stearate was added with thorough mixing. A very stiff white paste was formed and the temperature was increased slightly with continual agitation until the paste liquified and became almost clear at about 210° C. In reaching this temperature, increasing darkening of the paste occurred as the temperature rose; this was thought to be due to increasing decomposition of the lithium stearate by the relatively high degree of heat required to dissolve the metallic soap. The brown fluid was converted by cooling into a coffee-colored solid with a melting point of 185° C. This material was somewhat softer than the corresponding wax-magnesium stearate mixture and did not fracture in the knife blade test.

Examples XI to XVI, inclusive, showed that heating a magnesium soap mixed with wax caused different changes than in the case of other metallic soap-wax mixtures even when all are heated above the melting point of the soap used. The various products differ in respect to hardness, color, melting point, solubility, stability in solvents, translucency and fracturing characteristics. Of these soap-wax mixtures, only those containing magnesium soap appeared suitable in substantially all respects and superior in some as substitutes for natural ester waxes, especially carnauba wax.

Example XVII

	Parts
Magnesium stearate -----	40
Crude white scale wax -----	60

Upon repeating the heat treatment of Example XI with the quantities of materials listed, the initial thick, viscous, opaque fluid became clear and thin while being maintained at 140° C. The translucent product melted at 93° C. and was found to be slightly softer than the 50-50 mixture of Example XV. However, all other characteristics appeared to be substantially the same.

Example XVIII

	Parts
Magnesium stearate -----	30
Crude white scale wax -----	70

In duplicating the heat treatment at 140° C. with this mixture, the opaque dispersion of medium viscosity turned into a thin substantially transparent fluid which was then cooled to a very pale amber, translucent solid having a melting point of 82° C. The knife test of Example I produced a sharp clean fracture. Although this product was softer than the more concentrated magnesium stearate mixtures, its properties did not resemble those of crude scale wax.

Similar wax mixtures were prepared containing smaller quantities of heat-treated magnesium stearate. Data obtained from the resulting products indicated that these samples retained too many of the characteristics of scale wax to serve as adequate substitutes for carnauba and similar ester waxes.

Example XIX

	Parts
Magnesium stearate -----	20
Crude white scale wax -----	10
Candelilla wax -----	6
Petroleum solvent, 149/210° C. -----	64

After heating the solvent to 100° C. in a beaker the

solids listed were added and mixed thoroughly by prolonged stirring with the temperature constant; then the milky liquid was allowed to cool to room temperature. The cooled product was a soft, white, opaque paste. A layer of sediment was observed at the bottom of the beaker containing the soft, white, opaque paste; this sediment was magnesium stearate powder.

Example XX

Example XIX was repeated exactly except that the wax and stearate dispersion was heated to 140-150° C. until the mixture became clear. Upon cooling a light amber, clear, firm gel was obtained and no tendency toward bleeding of the solvent was observed.

Since certain changes in carrying out the above process and certain modifications in the composition which embody the invention may be made without departing from its scope, it is intended that all matter contained in the above description shall be interpreted as illustrative and not in a limiting sense.

It is also to be understood that the following claims are intended to cover all the generic and specific features of the invention herein described and all statements of the scope of the invention, which, as a matter of language, might be said to fall therebetween; and that they are intended to be inclusive in scope and not exclusive, in that, if desired, other materials may be added to our novel compositions of matter herein claimed without departing from the spirit of the invention. Particularly it is to be understood that in said claims ingredients or components recited in the singular are intended to include compatible mixtures of said ingredients wherever the sense permits.

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

1. A composition of matter which comprises a homogeneous mixture of a wax, and a heat-treated magnesium soap of a saturated fatty acid containing from 8 to 22 carbon atoms, the soap having been heated above its melting point for a period sufficient to substantially reduce its viscosity in the liquid state.

2. A composition of matter which comprises a homogeneous mixture of 100 parts by weight of a wax and from 40 to 400 parts by weight of a heat-treated magnesium soap of a saturated fatty acid containing from 8 to 22 carbon atoms, the soap having been heated above its melting point for a period sufficient to substantially reduce its viscosity in the liquid state.

3. A composition of matter which comprises a homogeneous mixture of 100 parts by weight of a hydrocarbon wax and from 60 to 200 parts by weight of heat-treated magnesium stearate which has been heated above its melting point for a period sufficient to substantially reduce its viscosity in the liquid state.

4. A composition of matter which comprises a homogeneous mixture of a liquid solvent of the group consisting of hydrocarbons and halogenated hydrocarbons, a wax and a heat-treated magnesium soap of a saturated fatty acid containing from 8 to 22 carbon atoms, the soap having been heated above its melting point for a period sufficient to substantially reduce its viscosity in the liquid state.

5. A composition of matter which comprises a homogeneous mixture of a liquid hydrocarbon solvent, a hydrocarbon wax and heat-treated magnesium stearate which has been heated above its melting point for a period sufficient to substantially reduce its viscosity in the liquid state.

6. A composition of matter which comprises a homogeneous mixture of 100 parts by weight of a liquid solvent of the group consisting of hydrocarbons and halogenated hydrocarbons and from 15 to 100 parts by weight of solids comprising a wax and a heat-treated magnesium soap of a saturated fatty acid containing from 8 to 22 carbon atoms in which the quantity of soap

is from 40 to 400% of the weight of the wax, the soap having been heated above its melting point for a period sufficient to substantially reduce its viscosity in the liquid state.

7. A composition of matter which comprises a homogeneous mixture of 100 parts by weight of a liquid hydrocarbon solvent and from 30 to 70 parts by weight of solids comprising a hydrocarbon wax and heat-treated magnesium stearate in which the quantity of magnesium stearate is from 60 to 200% of the weight of the wax, the magnesium stearate having been heated above its melting point for a period sufficient to substantially reduce its viscosity in the liquid state.

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