UNITED STATES PATENT OFFICE.

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SOLIDIFIED OIL AND PROCESS OF MAKING SAME.

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To all whom it may concern:

Be it known that I, CARLETON ELLIS, a citizen of the United States, residing at Montclair, in the county of Essex and State of New Jersey, have invented certain new and useful Improvements in Solidified Oils and Processes of Making Same, of which the following is a specification.

This invention relates to solidified oil compositions and to the process of making same and relates particularly to the solidified pine oils made from long leaf pine oil products and similar material and the solid product derived from cresylic acid and similar carbolic acid material. Ordinary pine oil, for example, is a liquid insoluble in water and having no power of itself of readily emulsifying with water. If however, the pine oil is mixed with soap solutions, emulsions may be obtained. By the use of concentrated soaps, such as oleate of potash, the pine oil becomes more or less miscible with water and certain preparations known as soluble pine oils which are of this character are now on the market.

The objeect of the present invention is to put pine oil and similar oils into a solid form and particularly into a form in which they are only slightly soluble in water, but which by the action of water, become slowly soluble or miscible thus becoming capable of distributing themselves in fine particles throughout a large body of water. The ordinary long leaf pine oil may be solidified by the incorporation with it of stearic acid and subsequent addition of caustic soda, to produce a soap in a concentrated condition. In the absence of more than a very small amount of water, practically that amount which just suffices to put the caustic soda in solution, and at an elevated temperature, the pine oil dissolves the sodium stearate, and on cooling solidifies to a hard solid material. On the other hand if oleic acid is used with caustic potash, for example, as the saponifying agent, a liquid material is derived. This is usually of a dark grown color due to the use of red oil or commercial oleic acid, while the stearic acid products are usually white and even transparent if the proportion of stearic acid soap is not too great and if the temperature has been sufficiently high to effect a perfect solution.

As an illustration to 14 pounds of pine oil and 1 pound of stearic acid commingled and incorporated by heat, when the stearic acid has thoroughly dissolved in the pine oil, ¼ pound caustic soda dissolved in slightly more than its own weight of water is added and the mixture stirred thoroughly, the heating being carried up preferably nearly to the boiling point of the pine oil in order to secure proper incorporation. At temperatures of for example, 40° or 50° C., a sort of coagulation occurs and a very unsatisfactory product results. If the temperature is carried to say 100° C., the product is usually firm and somewhat opaque. If however the temperature is carried up to nearly the boiling point of the pine oil, a clear transparent material may be produced. It is desirable however in actual practice to use steam jacketed kettles for manufacturing the product and for that reason, a temperature of 100° C. to 135° C. is perhaps more feasible. It is also desirable, after the material has been incorporated, to chill the product so as to prevent segregation which often occurs on slow cooling. This is very readily done in a jacketed kettle by substituting cold water in the jacket for the steam previously used for heating. The product obtained however if desired may be cast in molds which preferably should be chilled, and also preferably should be exposed a short time to the air or to a vacuum before shipment, in order to develop a high degree of firmness which such air or vacuum exposure tends to bring about. The pine oil product made in this manner is fairly soluble. If a small block of material is placed under running water, it may dissolve in from 20 to 30 minutes. For some purposes, such a high grade of solubility is undesirable and to reduce the solubility, I incorporate with the pine oil, for certain purposes, various oils such as refined mineral oils or crude petroleum. For example, in the above formula, if crude petroleum is substituted for one-third or one-half of the pine oil mentioned, a relatively slightly soluble material is secured. This is of importance as an insectifugal material for repelling and destroying mosquitos and the like on ponds or small bodies of water, inasmuch as large cakes of the material may

be placed in the water and as these slowly dissolve, the pine oil and crude petroleum oil spread over the surface of the water, the insectifugal odors of the pine oil driving the mosquitoes from the neighborhood and the crude petroleum oil effectually sealing the surface of the water so as to prevent reproduction of the mosquitos. Of course, various mineral oils may be used instead of crude petroleum oil and an oil of about .865 specific gravity derived from petroleum, is a very useful material for this purpose. Waxes act in a somewhat similar manner as respects reduction of solubility but are more expensive and are therefore more limited in application. Crude paraffin wax including scale wax and the like are however fairly cheap and may be included in varying amounts to secure various degrees of solubility. For example, in the formula above mentioned, the incorporation of 25% of scale wax reduces the solubility of the material and 50% makes the product very hard and slightly soluble; with the addition of 75%, the material becomes practically too insoluble to be of any great value for most applications. On the other hand, with the addition of 50% to 75% mineral oil, very oily solid products may be secured which have certain very desirable properties. In lieu of waxes, naphthalene may be employed to a greater or less extent. For example, in the above formula comprising pine oil, stearic acid and caustic soda, 5%, 10% and upward to say 40 or 50% of naphthalene may be incorporated. With the lower percentages, very little crystallization is evident on cooling, but with the larger percentages, crystallization is so much in evidence that the masses oftentimes are somewhat crumbly especially after water has acted upon the material for a short time. These solidified oils may be given various colors by the use of oil soluble dyes. They may be combined if desired with filling or weighting materials as for example, if it is desired to have the cake heavier than water, such substances as talc, infusorial earth, oxid of iron, etc., may be employed, while, if it is desired to have the cake lighter than water, such materials as sawdust, wood flour, starch, sugar and the like may be made use of. Also other essential oils may be included if it is desired to give special odors or special medicinal or other properties to the product. Of course, greases such as stearin, tallow, etc., degras, wool grease, lanolin, various resins such as colophony, dammar, sandarac, copal, Pontianak, guayule and similar resins, may be incorporated to a greater or less extent in accordance with the character of the product desired for special applications. Also rubber products particularly the crude Pontianak and guayule gums containing both the rubber and resinous material. Metallic soaps such as aluminum palmitate, zinc stearate and the like may be added as desired.

In the solidification of creosote, to produce a solid composition for preparing sheep dip and for similar purposes, the formula may be varied as follows: cresylic acid 10 pounds, stearic acid, 1 pound, caustic soda ¼ of a pound. This gives a product of moderate solubility which may be rendered more soluble by the incorporation of potassium oleate or sulfonated oils. Similarly with the pine oil, should it be desired to secure an especially soluble product, sulfonated oil in its concentrated liquid or solid forms, or potassium oleate or any similar soap material, may be introduced to advantage, care being taken to not use such proportions as to cause liquefaction.

The process may be applied to various oils such as oil of cedar, oil of cedar leaf, oil of camphor, oil of lemongrass and oils of birch tar, cajeput, mace, nutmeg and the like to secure a solid product. In order to produce a clear product it is desirable to heat the oil after incorporation of the soap to a temperature above the boiling point of water, until the water is eliminated, care being taken to agitate the oil well during this process as the boiling is oftentimes violent. If alcohol is used instead of water as the solvent for the caustic material, the operation may take place at a low temperature. This is especially the case with methyl alcohol which boils at 66° C. In the case of certain essential oils which have rather low boiling points and which are injured by subjection to high temperatures, the use of an alcoholic solvent is to be recommended.

In case of cresylic acid or other carbolic acid materials, the solidification may be made such that the product may be cut out into the shape of tablets for medicinal uses and may be made so firm as to be capable of supporting a coating of some other material. The addition of a little more alkali than that actually required for the combination with the fatty acid employed is usually desirable in the solidification of cresylic acid material. In the case of pine oil as will appear from the foregoing formula, 25% of caustic soda has been used reckoned on the amount of stearic acid, while the actual saponification requires only about 20%. This additional quantity of alkali is however not objectionable and is rather desirable in order to rapidly complete saponification. The material may however be made substantially neutral by using the exact equivalent of alkali. The stearic acid may be replaced by still stock to advantage in some cases.

The present invention therefore involves the process of melting or incorporating the essential oils to be solidified, with freely saponifiable soap-forming fatty acids, adding

thereto, the requisite amount of alkali, such as caustic soda, carbonate of soda, potash or whatever the alkali may be, dissolved in preferably just sufficient water to maintain the alkali in solution, combining this, preferably hot, with the oil, preferably hot, carrying the freely saponifiable fatty acid and agitating to effect perfect incorporation and substantial solution of the soap, preferably followed by the step of heating the oil to a temperature sufficient to expel a substantial portion or all of the water whereupon the mass preferably is rapidly chilled to insure proper setting and to overcome segregation.

It will be evident to those skilled in the art, from the information herein set forth, that various modifications may be made under the present invention to produce bodies of different proportions and suited for many different applications, and I do not wish to limit myself strictly to the invention as herein set forth and wish to apply or invoke the doctrine of equivalency so far as same may be herein applicable, all in view of the present state of the art and with due recognition of the heretofore employed methods of making soluble or solidified oils in various ways, by the use in some cases of soaps either to produce emulsions or miscible oils in a manner foreign to the idea of the present invention, but in some cases employing materials more or less similar in character to produce bodies having different properties or characteristics from those herein described. The products made under my invention are characterized by their peculiar colloidal condition manifesting itself in various ways. Some of the compositions hereunder for example are readily fusible when first formed but become difficultly fusible on standing. Seemingly the soap, forming as it does in the oil itself exerts by nascent action some peculiar colloiding influence on the oil and it is probably because of this condition of nascency that firmer, sounder products are obtained. Especially does this seem a more potent factor if after reaction is well under way the mass is quickly chilled, thereby possibly preventing the formation of molecular soap complexes.

By pine oil, or oil of pine, as used herein, I refer particularly to the volatile oils derived from genus *Pinus* and more specifically refer to spirits of turpentine, Russian turpentine, and long leaf pine oil.

I claim:—

1. The herein described firm solid insectifugal composition comprising an essential oil and a modicum of a soap of stearic acid with sufficient additional water-insoluble material to substantially reduce the solubility of the solidified product in water, said composition being prepared at a temperature above 100° C. whereby it becomes clear and transparent.

2. The herein described firm, solid insectifugal composition comprising an essential oil and a modicum of a sodium soap of stearic acid with sufficient additional water-insoluble material to substantially reduce the solubility of the solidified product in water, said composition being prepared at a temperature above 100° C. whereby it becomes clear and transparent.

3. The herein described solid insectifugal composition comprising pine oil, naphthalene and a modicum or sodium stearate so blended as to constitute a clear and transparent mass.

4. The herein described solid insectifugal composition comprising pine oil, naphthalene and a modicum of sodium stearate incorporated with a non-volatile oil to substantially reduce the solubility of the solidified product in water; the components of said composition being so blended as to constitute a clear and transparent cake.

In testimony whereof I have affixed my signature in presence of two witnesses.

CARLETON ELLIS.

Witnesses:
NATHANIEL L. FOSTER,
JAMES EMOLT.