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[73] Assignee **The United States of America as represented by the Secretary of Agriculture**

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[54] **METHOD FOR APPLYING LONG CHAIN FATTY ACID PHENYLMERCURY FUNGICIDES IN WAX**
3 Claims, No Drawings

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[51] **Int. Cl.** **B44d 1/09,** D06m 13/50
[50] **Field of Search** ..... 117/138.5, 139.5 CQ, 139.5 F, 121, 135.5; 424/291; 106/15 AF

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ABSTRACT: This invention relates to a process for applying long chain fatty acid phenylmercuric fungicides. Many long chain fatty acid phenylmercuric salts are available as fungicides but application procedures have been difficult and expensive. This invention relates to a method whereby these compounds can be applied in conjunction with a wax finish, in a single bath application whereby the fungicide remains in the wax finish component of fabric treatments for water repellency and fungicidal activity.

METHOD FOR APPLYING LONG CHAIN FATTY ACID PHENYLMERCURY FUNGICIDES IN WAX

A nonexclusive, irrevocable, royalty-free license in the invention herein described, throughout the world for all purposes of the United States Government, with the power to grant sublicenses for such purposes, is hereby granted to the Government of the United States of America

Many long chain fatty acid phenylmercuric salts are available as fungicides. However, application procedures have been difficult and expensive. These compounds are insoluble in water and form water soluble complexes with triethanolamine or zirconyl acetate. This invention relates to a method whereby these compounds can be applied in conjunction with a wax finish in single bath applications whereby the fungicide remains in the wax finish component of fabric treatments for water repellency and fungicidal activity.

Phenylmercuric compounds (oleate, stearate, naphthenate, etc. will melt and blend completely with paraffin wax. Paraffin wax emulsions have long been used to finish off fabrics after fungicidal treatment. By the process of this invention, the blended paraffin and phenylmercuric salt is emulsified, the same as with paraffin alone, and the emulsion diluted with water to give any concentration of phenylmercuric salt and paraffin desired.

In the preferred process of this invention, fabric treatment consists of conventional padding with the paraffin-phenylmercuric salt emulsion to give the desired add-on. The padded fabric is dried at 120° –130° C. for about 5 minutes. Drying temperatures must be over 110° C. in order to remove water and volatiles and to cause the phenylmercuric salt-paraffin emulsion to fuse and form a protective fiber coating. The dry emulsion treatment fabric is then padded with an aqueous solution of aluminum-acetate and again dried at 120° –130° C. for 5 minutes to remove any ammonia and reduce solubility of any residual emulsifier.

The following examples illustrate but do not limit the scope of this invention.

EXAMPLE 1

One hundred grams of paraffin, 50 grams of stearic acid, and 50 grams of phenylmercuric stearate are melted together (115° C.). One hundred milliliters of 28 percent ammonium hydroxide (NH_4OH) are added to 200 ml. of distilled water, and this solution added to the melt, with vigorous stirring, keeping the temperature between 70° –80° C. throughout the addition. A uniform paste emulsion results. Stirring is continued (70° –80° C.) until the uniform paste is thoroughly emulsified, the temperature is then allowed to drop to room temperature, while stirring is continued. The paste emulsion concentrate contains 20 percent paraffin and 10 percent phenylmercuric stearate.

EXAMPLE 2

One hundred grams of paraffin, 167 grams of 30 percent phenylmercuric oleate liquid and 50 grams of stearic acid are mixed and heated to 70° C. until the paraffin and acid dissolve to form a clear liquid. Two hundred ml. of distilled water and 100 ml. of 28 percent NH_4OH are mixed and added to the clear liquid when cooled to 60° C. The mix is vigorously stirred until the mixture cools to room temperature. A paste emulsion results containing 20 percent paraffin AND 10 percent phenylmercuric oleate. This emulsion is further diluted with water for application.

EXAMPLE 3

An emulsion using
100 grams of paraffin
50 grams phenylmercuric stearate
50 grams stearic acid
200 grams distilled water
100 grams 28 percent ammonium hydroxide
was prepared as in example 2 to give 500 grams paste emulsion (20 percent paraffin and 10 percent phenylmercuric salt).

EXAMPLE 4

Seventy grams of the paste emulsion from example 3 were diluted to 1000 grams with distilled water at 120° .F. This results in an emulsion containing 1.4 percent paraffin and 0.7 percent phenylmercuric stearate. This emulsion was padded onto 2 yards of canvas duck to give a 60 percent wet pickup, resulting in an add-on of 0.84 percent paraffin and 0.42 percent phenylmercuric stearate. The mercury content is 35 percent of the phenylmercuric stearate, resulting in 0.15 percent mercury (Hg) add-on. This treatment on duck shows 26.5 percent retained strength after 2 week soil burial, whereas an untreated control duck sample had rotted in 1 week.

EXAMPLE 5

Two hundred ten grams of paste emulsion from example 3 were diluted to 1000 grams with distilled water at 120° F. and stirred to produce a uniform emulsion. A 2-yard sample of canvas duck was padded and oven dried at 130° C./4 minutes. This 1000 grams of emulsion contains 4.2 percent paraffin and 2.1 percent phenylmercuric stearate. Application was made for a 60 percent wet pickup, resulting in an add-on of 2.52 percent paraffin and 1.20 percent phenylmercuric stearate. Since 3.5 percent mercury composes phenylmercuric stearate, the mercury metal add-on was 0.42 percent (Hg). This fabric was subjected to soil burial. At the end of three weeks burial, the treated sample showed 24.4 percent retained breaking strength (tensile), while the untreated control duck had rotted out in one week.

EXAMPLE 6

A paraffin phenylmercuric oleate emulsion using 100 grams paraffin
167 grams phenylmercuric oleate (30 percent salt) containing 17.98 g. of Hg metal
50 grams stearic acid
200 grams distilled water
100 grams 28 percent ammonium hydroxide
was prepared as in example 2. Emulsification occurs readily and easily with vigorous stirring, and a cream white paste emulsion results. It contains 16.2 percent paraffin and 27.0 percent of the phenylmercuric liquid, or 8.1 percent of the phenylmercuric oleate (dry basis). Since phenylmercuric oleate contains 36 percent mercury, the emulsion contains 2.91 percent mercury.

EXAMPLE 7

One hundred grams emulsion described in example 6 were diluted with 400 grams hot distilled water (50° C.). This uniform, well mixed, emulsion contains 1.6 percent phenylmercuric oleate (57 percent Hg) and 3.24 percent paraffin. With a 55 percent wet pickup, a 2-yard × 9-inch sample of canvas duck was treated through conventional pad and cure, to give an add-on of 0.31 percent Hg and 1.78 percent paraffin, the paraffin and phenylmercuric compound as a blend in the wax finish. A colorless fabric with good hand and no discoloration was produced.

EXAMPLE 8

Fungicidal activity of the sample treated in example 7 was evaluated through soil burial. The treated sample showed no strength (tensile) loss for 2 weeks, while the control rotted out in one week. At the end of four weeks burial, the treated sample was intact and still retained 3.1 percent tensile strength. The active fungicide is protected by the water repellency of the wax, and as the finish slowly disintegrates through oxidation and UV absorption in weathering, the mercury compound is actively in contact with surface micro-oranisms. These wax-phenylmercuric compound emulsions are as stable as paraffin emulsions alone and can be used in place of pure paraffin emulsions to attain the usual water repellency with an added fungicidal effect. It is also an advantage to have a phenylmercuric fatty acid fungicide in the wax water repellent finish, as

well as in the water phase of certain textile treatments, giving complete fungicidal protection from the surface of the wax finish throughout the entire fabric. These paraffin-phenylmercuric compound emulsions require no organic solvents and eliminates these in procedure, thus reducing cost and increasing safety from fire hazards with solvent systems. In prior art, the application of phenylmercuric oleate, stearate, etc. required organic solvent systems for solvent or emulsion application. This process or procedure eliminates organic solvents entirely, with solubility of the phenylmercuric compound being effected in melted paraffin with excess stearic acid and direct emulsification with aqueous ammonia. Particle size and stability of these produced emulsions were excellent for storage and application.

EXAMPLE 9

An emulsion was prepared as in example 1 using:
100 grams paraffin
165 grams phenylmercuric naphthenate (18.1 percent Hg)
50 grams stearic acid
200 grams distilled water (50° C.)
100 grams 28 percent ammonium hydroxide
Emulsification occurs immediately. The stock emulsion contains 16.2 percent paraffin and 26.8 (Hg). percent phenylmercuric naphthenate or 4.85 percent mercury (Hg).

EXAMPLE 10

One hundred grams of the emulsion prepared in example 9 were diluted with 300 grams of hot distilled water (50° C.). This emulsion contains 4.0 percent paraffin and 1.21 percent mercury. This emulsion was padded and cured onto canvas duck to give a 50 percent wet pickup, resulting in add-ons of 2.0 percent paraffin and 0.60 percent mercury. Cure was effected in the oven at 50° C. for 5 minutes. Treatment was colorless and fabric hand good.

EXAMPLE 11

Soil burial with phenylmercuric naphthenate-paraffin emulsion treated canvas duck. The treated fabric (example 10) was subjected to soil burial with an untreated canvas duck control. At the end of one week the untreated duck was rotting and showed 5.7 percent retained strength while the treated sample showed 100 percent retained tensile strength. At the end of two weeks the control had disintegrated while the treated sample showed 100 percent retained tensile strength. At the end of 5 weeks the treated sample showed 47 percent retained strength and was in very good condition.

I claim:

1. A process for rendering a fabric water repellent and fungicidal comprising:
   a. heating together about 2 parts of paraffin wax, about 1 part of a fungicidal, water-insoluble, long chain fatty acid phenylmercuric salt, and about 1 part stearic acid, at a temperature of about 115° C. to form a molten mixture in which the phenylmercuric salt is blended with the wax;
   b. adding to the mixture from (a) a solution consisting of about 1.7 to 2.0 parts of 28% ammonium hydroxide and about 4 parts of distilled water with vigorous stirring while maintaining the temperature at about 70° C. to 80° C. to form a uniform paste emulsion in which the phenylmercuric salt remains in the nonaqueous phase thereof;
   c. cooling the emulsion from (b) to about room temperature;
   d. diluting the emulsion from (c) with distilled water at a temperature of about 50° C. in the ratio of about 3 to 14.3 parts of water per part of the emulsion;
   e. padding a fabric with the diluted emulsion of (d); and
   f. drying the padded fabric from (e) at a temperature of about from 120° C. to 130° C. for about 5 minutes to produce a fabric having a wax finish with the phenylmercuric salt as a component thereof thereby to impart both water repellency and fungicidal activity to the fabric.

2. The process of claim 1 wherein the phenylmercuric salt is phenylmercuric stearate.

3. The process of claim 1 wherein the phenylmercuric salt is phenylmercuric oleate.

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