

Malaya

PATENT SPECIFICATION

1,167,875

NO DRAWINGS.

Date of Application (No. 45459/66) and filing Complete Specification: 11 Oct., 1966.

Application made in United States of America (No. 498,153) on 19 Oct., 1965.

Complete Specification Published: 22 Oct., 1969.

1,167,875



Index at acceptance:—A5 E1C5B; C1 A(H11G, H12A, H12C, H12D, H12X, N11).

International Classification:—A 01 n 11/04.

COMPLETE SPECIFICATION.

Dispersions of Cupric Hydroxide.

We, KENNECOTT COPPER CORPORATION, a corporation organized under the laws of the State of New York, one of the United States of America, of 161 East 42nd Street, City and State of New York, United States of America, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to dispersions of cupric hydroxide. More particularly, the invention relates to aqueous dispersions of finely divided, phosphate-process cupric hydroxide. Such dispersions are useful for biocidal purposes, particularly for fungicidal applications.

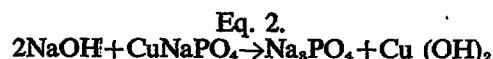
Although cupric hydroxide has been referred to frequently in the literature, it is not a simple, well-defined chemical. When a base, such as sodium hydroxide, is added to a solution of a water-soluble copper salt, for example, copper sulfate, a blue, gelatinous precipitate is formed that gradually turns black in color. This precipitate is not a stable or simple material, and apparently consists of a mixture of the hydroxide, cupric oxide hydrate, and cupric oxide.

The nature of such a precipitate changes with time, temperature and drying. Such changes proceed, for example, in the fungicidal mixture known as Bordeaux Mixture, that is prepared by mixing together copper sulfate and lime. This mixture is freshly made before application, in order to be effective, because of the chemical changes that occur in it over a period of time.

Attempts to avoid such undesirable changes in composition, as occur in Bordeaux Mixture, led to the development of the so-called fixed coppers, for example, tribasic copper sulfate, copper oxychloride

and copper-zinc chromate, and that can be stored, mixed with water or dusted when needed for use.

United States Reissue Patent No. 24,324, granted May 28, 1957, discloses a process for making a stable cupric hydroxide. This process comprises reacting substantially equal molar amounts of copper sulfate and trisodium phosphate, to obtain a copper-containing precipitate. Sodium hydroxide is then added in an amount sufficient to convert a major portion of the precipitate to cupric hydroxide. The addition of sodium hydroxide regenerates trisodium phosphate. The reaction may be characterized as follows:



The process is continued by alternately adding copper sulfate and sodium hydroxide. As many as 15 or 20 of these additions may be made in producing a batch of cupric hydroxide by this process. A dry, solid product is obtained by separating the solids from the mother liquor, washing, drying, and grinding.

It has now been found that the many variables in this multistep process may be adjusted over wide ranges to produce phosphate-process cupric hydroxide products of differing characteristics. These variables include not only factors such as temperature, concentrations, ratios and pH, but also other factors such as the presence of other reactants. For example, in some cases it may be desirable to produce a cupric hydroxide product that contains a minor but appreciable amount of phosphate. In such

a case, calcium hydroxide may be substituted for at least a part of the sodium hydroxide, in the production process, so that the final product includes calcium phosphate as an inert diluent.

The products of this process may therefore differ in composition and characteristics, but cupric hydroxide materials produced by precipitation from copper sulfate with trisodium phosphate followed by reaction with sodium hydroxide are identified collectively hereinafter by the general term, "phosphate-process cupric hydroxide". It will be understood that these products contain a substantial amount of cupric hydroxide, together with detectable phosphate, and, usually, detectable sulfate.

A typical phosphate-process cupric hydroxide product, produced in accordance with the method of United States Reissue 24, 324, as described generally above, is characterized by being a water-insoluble material, having a very fine particle size. In contrast with previously available copper hydrate products, it is stable in dry, powdered form, and can be stored over extended periods of time without change in color or loss in its useful properties.

Prior art copper hydrate products have not only tended to be unstable in chemical characteristics, chemical composition and color, but they also have typically been

incapable of forming practically useful, stable aqueous suspensions for a variety of reasons. For example, dispersions and mixtures of copper hydrate products in water are often not flowable at solids concentrations in excess of 25% by weight of the mixture. This has limited the use of such products in biocidal and other applications.

One object of the present invention is to provide aqueous dispersions of finely divided cupric hydroxide, useful for a variety of practical applications, particularly in the fungicidal field and having commercially acceptable stability as to both chemical and physical properties.

A related object of the invention is to provide such dispersions that contain fungicidally-effective amounts of cupric hydroxide, and that are sufficiently stable that the cupric hydroxide will not settle out to form hard conglomerates, even after storage over prolonged periods of time or under adverse conditions, and that are flowable, even at extremely high solids contents, to facilitate handling.

Following the procedure that is described in U.S. Reissue No. 24,324, a phosphate-process cupric hydroxide product was prepared and dried to a powder. The approximate chemical analysis specifications of this product can be stated as follows:

Chemical Specifications

% By Weight

Copper Content (expressed as metallic copper)	53.0—64.00
Phosphorus Content (as P_2O_5)	2.0—7.0 or higher; preferably 3—4
Sulfate Content (as SO_4)	0.5—3.0
Free Water Content	1—5

Products made in accordance with these specifications are stable for indefinite periods of time when stored at temperatures that do not exceed 120°F. The powder is light blue in color, and has an acicular alternate crystal form. The dry powder has a true specific gravity of approximately 4.0, and a bulk density of from 5 to 15 lbs. per cubic foot. The individual particles are in the colloidal size range, that is, within the size range from 0.001 micron up to 1 micron. If permitted to stand in saturated air at 15°C., the moisture content of the dried powder will rise gradually until an equilibrium is attained at 8.9% or higher.

Considerable practical difficulties have been encountered in the past in preparing and in utilizing aqueous suspension of cupric hydroxide. Depending upon the material being used, and upon other pertinent factors including the hardness of the water,

the pH of the water, the presence or absence of a protective material such as soap or starch, and the amount of solid material attempted to be kept in suspension, there often occurred unpredictable, uncontrollable, and undesirable demonstrations of instability, for example, settling out of the solid particles, formation of a hard gel, formation of agglomerates, or, in some instances, the formation of hard cakes. Such demonstrations of instability have hampered commercial exploitation of products of these kinds.

It has now been found that substantially uniform aqueous dispersions of finely divided cupric hydroxide, of commercially acceptable stability, can be made by dispersing previously prepared cupric hydroxide in an aqueous vehicle under carefully regulated pH conditions, where the pH of the dispersion is in the range of from 7.0

to 9.5 and preferably where the hardness of the aqueous vehicle is not greater than the equivalent of 180 ppm CaCO_3 . Such dispersions may be prepared simply by mechanically dispersing phosphate-process cupric hydroxide in an aqueous vehicle. In some applications dispersing agents may be added to give greater dispersion. Examples of suitable dispersing agents are alkali metal salts of polymeric carboxylic acids, alkali metal salts of lignin-sulfonic acids, alkyl sulfates, sulfated fatty alcohols and sulfonated naphthalenes. If desired, protective colloids may be added to prevent agglomeration. To facilitate spraying of the dispersions, small but effective amount of wetting agents may also be added. Such dispersions are attractive for a variety of applications when the content of cupric hydroxide, by weight, is at least 0.1% and preferably in the range from 0.1 to 60%; and are fungicidally effective and economically attractive with a content of phosphate-process cupric hydroxide in the range from 0.5% to 40%. For shipment in concentrated form to a point of local use, stable dispersions containing at least 10% to 20%, and preferably at least 33% by weight of phosphate-process cupric hydroxide are desirable and can be made in accordance with the present invention.

The invention accordingly consists in a stable mixture having a pH of from 7.0 to 9.5, comprising at least 0.1% by weight of finely divided cupric hydroxide and an aqueous vehicle.

Phosphate-process cupric hydroxide, in accordance with the specifications set forth

above, ordinarily will contain phosphate sulfate ions, in amounts that can be regulated by careful control of the proportions of the respective reactants, that are used in the manufacturing process. For fungicidal applications, a phosphate content of about 5% by weight of solids, dry basis, is advantageous and desirable in many cases.

Beyond the limits of the preferred pH range of 7.0 to 9.5, conditions are unfavorable for stability. At low pH values, chemical changes occur and the copper goes into solution. At high pH values, chemical changes also occur and the cupric hydroxide loses its distinctive characteristic as it converts to copper oxide.

The following specific examples will illustrate several preferred embodiments of the invention.

EXAMPLE I

Stable dispersions were prepared, in this example, utilizing as a dispersing agent a highly purified sodium lignin sulfonate, anionic in aqueous solution.

In this and the following examples, the dispersions were prepared by adding all of the ingredients successively to a predetermined quantity of the aqueous vehicle, and agitating. The dispersant was, and always should be, added to and mixed with the aqueous vehicle before the addition of phosphate-process cupric hydroxide. The aqueous vehicle was selected to have an initial hardness not greater than the equivalent of 180 ppm CaCO_3 . The pH of the dispersion was adjusted, as necessary, to be in the range from 7.5 to 9.5.

The following dispersions were prepared:

TABLE I

Dispersion No.	% Phosphate-Process Cupric Hydroxide, based on the entire mixture	% Dispersing Agent*	Viscosity** of Dispersion	% Protective Colloid (Water-soluble Lecithin)
1	10	1	10,000	—
2	33.3	1	33,000	1
3	33.3	1	26,000	1
4	33.3	2		

* By weight, based on total inorganic solids, dry basis; used in all subsequent examples also, as to the dispersant.

** Units, centipoises, as measured on a Brookfield viscometer using a T-type spindle at 2.5 rpm; used throughout except where otherwise specified.

In this and all subsequent examples, all percentages are by weight.

Lecithin was added in this and some subsequent examples, for dispersions having

high solids contents. It serves as a protective colloid, and inhibits agglomeration.

All of the dispersions described in Table I exhibited excellent stability.

EXAMPLE II

Excellent dispersions were also made, using as a dispersing agent the sodium salt of a polymeric carboxylic acid, which is an anionic liquid in its commercial form.

TABLE II

Dispersion No.	% Phosphate-Process Cupric Hydroxide	Dispersing Agent, based on dry inorganic solids	Viscosity of Dispersion	% Protective Colloid (Water-soluble Lecithin)
5	10.0	1	—	—
6	33.3	1.5	5,400	—
7	33.3	1.5	3,300	1%
8	33.3	2.0	8.4*	—
9	33.3	2.0	179*	1%
10	37.3	1.6	—	—
11	45.1	1.5	—	—
12	58.0	6.0	above 10,000	—

* As measured on the Brookfield viscometer using an RV type spindle at 50 rpm.

Density measurements were made on dispersions No. 11 and 12, respectively, and were: 1.34 grams per milliliter; and 1.45 grams per milliliter.

All of the dispersions were stable over prolonged periods of time. The dispersions containing lecithin exhibited good resistance

to the formation of agglomerates under unfavourable storage conditions.

EXAMPLE III

Additional dispersions were made up, using as the dispersing agent a commercially available sulfonated naphthalene:

TABLE III

Dispersion No.	% Phosphate-Process Cupric Hydroxide	% Dispersing Agent, based on dry inorganic solids	Viscosity of Dispersion
13	10	1	—
14	33.3	1	45,000
15	33.3	1.5	27,000
16	33.3	2.0	7,600

The above dispersions were acceptably stable when stored at ordinary room temperatures over long periods of time. These dispersions demonstrate the effect of the dispersing agent in reducing viscosity.

EXAMPLE IV

Additional formulations of aqueous dispersions, that demonstrate the use of a variety of dispersants, are as follows:

TABLE IV

5	Dispersion No.	% Phosphate-Process Cupric Hydroxide	Identity of Dispersing Agent	% of Dispersing Agent, based on dry inorganic solids	Viscosity*
	17	33.3	Technical protein colloid — sold by Swift and Company under the trade identification of Swift 2185	3	380,000
10	18	33.3	same	5	400,000
	19	33.3	Tallow dimethyl benzyl ammonium chloride, sold by Onyx chemical under the trademark Ammonyx 856	1	510,000
15	20	33.3	same	2	520,000
	21	33.3	Sodium salt of polymerized alkyl aryl sulfonic acid, sold by R. T. Vanderbilt under the trademark Darvan 1	1	28,000
20	22	33.3	same	2	26,000
	23	33.3	Diethanolamide of special fraction of coconut fatty acids, sold by Scher Brothers under the trademark Schercomide SPO	1	33,000
25	24	33.3	same	2	37,000
	25	33.3	Sodium salt of condensed mononaphthalene sulfonic acid	1	30,000
	26	33.3	same	2	32,000

* T-type spindle on a Brookfield viscometer at 2.5 rpm.

30 All of the above dispersions were characterized by satisfactory stability.

Wetting agents are useful additives for the dispersions, to facilitate spraying. Care must be exercised, however, in the selection of the wetting agent and in the amount used, to avoid foaming and increased viscosity.

FUNGICIDAL APPLICATIONS

Comparative growth studies have shown that dispersions made in accordance with the present invention are effective mycotic agents against organisms that are of economic importance in agriculture, as follows:

45	Organism	Significance
	<i>Cercospora Musae</i>	Organism Causing Leaf Spot on Bananas (Sigatoka)
	<i>Monilia Roeri</i>	Organism Causing Pod Rot of Cocoa
	<i>Alternaria Solani</i>	Organism Causing Early Blight on Tomatoes
50	<i>Chaetomium Globosum</i>	Standard F. F. H. A. Mildew Resistance
	<i>Corynebact. Insidiosum</i>	Causative Agent of Alfalfa Wilt
	<i>Helminthospor. Turcicum</i> Pass ...	Causative Agent of Corn Leaf Blight
	<i>Phytophthora Citrophthora</i>	Causative Agent of Citrus Brown Rot
	<i>Phytophthora Infestans</i>	Causative Agent of Potato Late Blight
55	<i>Streptomyces Scabies</i>	Causative Agent of Potato Scab
	<i>Diplodia Zeae</i>	Corn Ear and Stalk Rot
	<i>Colletotrichum Lindemuthianum</i> ...	Cause of Anthracnose in Beans
	<i>Verticillium Albo-Atrum</i>	Causative Agent of Cotton Wilt
	<i>Fusarium Oxysporum</i>	Cause of Wilts in Potato, Muskmelon, etc.

Dispersions made in accordance with the present invention have been demonstrated to be effective in the control of many other troublesome agricultural conditions, in addition to those identified above, such as, for example, crown rot of the banana, cercospora leaf spot of celery and sugar beets, and pear fire blight.

In wet climates, one or more materials can be added to the aqueous fungicidal dis-

persions that are made in accordance with this invention, to promote retention of the cupric hydroxide particles on the plant sites where applied. The following example is illustrative.

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EXAMPLE V

Stable aqueous dispersions were made up with phosphate-process cupric hydroxide, for evaluation for fungicidal activity, as follows:

TABLE V

Dispersion No.	% Phosphate-Process Cupric Hydroxide	% Dispersing Agent	% Emulsified Petroleum Wax
27	1.4	1	10
28	2.9	1	10
29	5.7	1	10
30	5.7	1	—

The dispersing agent used was isooctyl phenyl polyethoxy ethanol, a nonionic material.

The emulsified wax was prepared as an emulsion before addition to the dispersion. It was prepared to contain approximately 51% by weight of solids, comprising a mixture of paraffin wax, microcrystalline wax, and petroleum. The emulsion was milky in appearance and had a weight of 8 lbs. per gallon. It was stable in the presence of acids and electrolytes. The percentage of emulsified wax, that was added to each of the above dispersions, represents the wax emulsion, at 51% solids, rather than the amount of wax alone.

Each of the dispersions identified above was evaluated for effectiveness as a fungicide for the control of *Monilia* on Cacao, in a controlled experiment providing comparative data relative to other effective, commonly used fungicides, as well as against the customary check plot in which no fungicide was employed. A significant reduction in the incidence of diseased pods was observed in those cases where each of the dispersions in this example was employed. Those dispersions that included the wax exhibited a greater sticking power or residuary action than the dispersion that did not contain wax.

By way of further example, residual, phosphate-process cupric hydroxide, after application as a spray of one of the dispersions mentioned above in this Example, or other, more concentrated dispersions, at a concentration in the range from about 1.6 to about 3.3 micrograms (dry basis) per square centimetre of leaf surface, effectively inhibits the germination of *Cercospora Musae*, the cause of Sigatoka disease of

bananas. This is a significantly higher inhibitory effect than is obtained with Bordeaux Mixture.

An unexpected advantage of fungicidal compositions, that are made in accordance with the present invention, is that they exhibit the characteristic of high retention on plants under rainy conditions, even when the dispersion does not include wax or other "sticking" agent. This may be attributable to the extremely small particle size of the copper hydroxide particles. It is also affected by the use of wetting agents. By observing plants after application of the dispersion, it has been found that an aqueous vehicle may, upon drying, contract and tend to clump the particles of solids into concentrated sites. The addition of a wetting agent helps to overcome this tendency.

Although erosion of the cupric hydroxide spray deposit does occur during rainfall, reduction in disease control does not deteriorate at the same rate as might be indicated by the reduction in the presence of copper. This is a completely unexpected advantage of preparations made in accordance with the present invention, and has been observed in many applications. This phenomenon is believed to be attributable to the fact that the residual particles of the phosphate-process cupric hydroxide are so finely divided that they are extremely difficult to remove, and are also highly effective in destroying fungus spores before they can induce infection.

EXAMPLE VI

The following procedure was employed in evaluating dispersions of phosphate-process cupric hydroxide, in the control of early blight on tomatoes.

Phosphate-process cupric hydroxide powder was blended with a 0.5% aqueous solution of a nonionic detergent. This blend was then diluted with water to form three different solutions containing the phosphate-process cupric hydroxide equivalent to 0.7, 2.1, and 6.3 lbs. of the cupric hydroxide per hundred gallons of dispersion, respectively. Dispersions of tribasic copper sulfate were made up at the same time, in substantially the same way, and at the same concentrations, for comparative purposes.

These dispersions were applied to test

plantings of Bonny Best tomato plants by spraying. The spray was allowed to dry on the leaves for about one hour after application. Evaluations were then made of retention of the active ingredient during rainy conditions, and of fungicidal effectiveness.

The phosphate-process cupric hydroxide fungicides were considerably more effective in controlling early blight than the tribasic copper sulfate fungicides, as shown by the data that is summarized in the following table.

	Treatment Applied	Dose Lbs. 100 Gal.	Control After Rainfall (Inches)				
			0"	$\frac{1}{2}$ "	1"	2"	4"
30	Phosphate-process $\text{Cu}(\text{OH})_2$ dispersion	0.7	% 75	% 77	% 74	% 75	% 59
		2.1	86	85	85	79	59
		6.3	91	86	87	86	88
	Tribasic CuSO_4 dispersion	0.7	43	50	20	28	44
		2.1	52	41	39	69	22
		6.3	45	60	57	45	45

Other stable suspensions of phosphate-process cupric hydroxide have been prepared by grinding together, in the dry state, the phosphate-process cupric hydroxide and a material such as carboxymethylcellulose or hydroxyethylcellulose. These form excellent suspensions when the dispersing agent is at a concentration of about 1% to 2% by weight. However, these dispersions are not quite as satisfactory for application through fine spray nozzles as some of the other suspensions that have previously been described.

Wetting agents are often included in fungicidal dispersions to obtain an initial good distribution of the cupric hydroxide particles on the plant surfaces to which they are applied, that is, to promote desirable deposition and adhesion.

GENERAL

In preparing suspensions, for example, fungicidal concentrates or formulations, in accordance with the present invention, although the pH of the suspension may have any value within the range from 7.0 to 9.5 for acceptable stability, it is preferably in the range from 8 to 9. The hardness of the aqueous vehicle should be not greater than the equivalent of 180 ppm CaCO_3 for best results.

To improve the stability of a suspension, a dispersant should be employed. Preferably, an anionic dispersant with low foaming tendencies should be used. The primary purpose of a dispersant is to reduce or to eliminate agglomeration of the particles. A

secondary and very valuable purpose of the dispersant is to reduce the viscosity of the suspension. This is particularly important with respect to concentrated suspensions, containing up to 70% by weight of phosphate-process cupric hydroxide, prepared for shipment to a point of use, for dilution there to use strength.

A wetting agent is a valuable addition to a suspension, for lowering surface tension, and for obtaining a uniform distribution of particles on a surface to which the suspension is applied. Frequently, a single material may serve as both a dispersing agent and a wetting agent.

Wax or wax-like additives are used to promote good adhesion of the solid particles to the surface to which the suspension is applied. Such additives preferably are used in the form of emulsions. The wax material may be an animal, vegetable or mineral wax, or a synthetic wax, such as, for example, a polyethylene wax. When a wax additive is employed, the use of a protective colloid usually produces no additional advantage. Acrylic emulsion additives are also useful and for present purposes can be considered to be wax-like additives. Other materials, such as, for example, synthetic latices, caseins, natural latices, and more complex polymeric materials, for example, modified alkyd resins, can also be employed for the same purpose as wax emulsions.

The amount of wax employed may be very small. Amounts in the range from 10% to 50% of wax, based upon the weight of phosphate-process cupric hydroxide in the

suspension, are satisfactory, although larger amounts may be used, as shown in Example V.

WHAT WE CLAIM IS:—

- 5 1. A stable aqueous dispersion having a pH of from 7.0 to 9.5 comprising at least 0.1% by weight of previously prepared, finely divided, cupric hydroxide and an aqueous vehicle.
- 10 2. A stable dispersion in accordance with claim 1 including a dispersing agent.
3. A stable dispersion in accordance with claim 1 or 2 wherein the pH of the mixture is in the range from 8 to 9.
- 15 4. A stable dispersion in accordance with any of claims 1 to 3 wherein the aqueous vehicle has a hardness not greater than the equivalent of 180 ppm CaCO_3 .
- 20 5. A stable dispersion in accordance with any of the preceding claims, wherein the cupric hydroxide consists of particles in a colloidal size range.
6. A stable dispersion in accordance with any of the preceding claims, wherein the dispersing agent is an anionic dispersing agent.
- 25 7. A stable dispersion in accordance with any of the preceding claims, wherein the dispersing agent is an alkali metal salt of a polymeric carboxylic acid, an alkali metal
- 30

salt of a lignin sulfonic acid, an alkyl sulfate, a sulfated fatty alcohol, or a sulfonated naphthalene.

8. A stable dispersion in accordance with any of the preceding claims including a protective colloid effective to inhibit agglomeration. 35

9. A stable dispersion in accordance with claim 8 wherein the protective colloid is a water soluble lecithin. 40

10. A stable dispersion in accordance with any of the preceding claims wherein the amount of cupric hydroxide present is from 0.1% to 60% by weight of the dispersion.

11. A stable dispersion in accordance with any of the preceding claims including at least 10% by weight of the cupric hydroxide, dry basis, of an emulsified wax. 45

12. A fungicidal composition comprising as the active ingredient a stable aqueous dispersion in accordance with any of claims 1—10. 50

13. A stable, substantially uniform aqueous dispersion substantially as hereinbefore described with reference to any one of examples I—V. 55

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Printed for Her Majesty's Stationery Office by Burgess & Son (Abingdon), Ltd.—1969.
Published at The Patent Office, 25 Southampton Buildings, London, W.C.2,
from which copies may be obtained.

