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STABILIZED INSECTICIDAL PHOSPHORODITHIOATES IN POWDERS OF SUBSTANTIALLY IRON-FREE KIESELGUHR WITH LOW VOLATILE SOLVENT OILS

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ABSTRACT OF THE DISCLOSURE

Insecticidal powders of the wettable or dispersible type containing a phosphorodithioate as the active biocide having critically improved stability on storage are formed by combining the active biocide with a solvent oil of low volatility and substantially iron-free kieselguhr.

Background of the invention

Insecticides such as the phosphorodithioic acid derivatives have a pronounced tendency to decompose, with the formation of thiol groups. When such decomposition occurs, in formulations containing these insecticides, the formulations acquire an offensive smell and suffer a considerable loss of activity due to substantial reductions in the content of the active constituent. Various methods have previously been proposed for deodorising insecticides of this type, but these methods have usually relied on the removal of the offensive thiol compounds formed on decomposition, and have done nothing to maintain the activity of insecticidal compositions by inhibiting the decomposition itself.

The same problem has been encountered in the preparation of powders containing these insecticides. When deodorised Malathion, for example, is added to conventional carrier materials, such as silica flour, there is an accelerated return of the odor, indicating that the active constituent is being quite rapidly decomposed.

Summary of invention

The present invention provides an insecticidal powder in which the content of the active ingredient remains substantially constant over a long period, and is characterised by a particulate carrier composed of substantially iron-free kieselguhr, or diatomaceous earth, impregnated with a phosphorodithioate insecticide in the presence of a solvent oil of low volatility.

Where a wettable or dispersable powder is required, an inert wetting and/or dispersing or suspending agent may be incorporated in the product.

It is preferred that the kieselguhr should be of high absorptivity. It has been observed that kieselguhrs containing considerable amounts of iron and of low absorptivity do not provide carriers on which the insecticide is stable. It is found that the kieselguhr should preferably be of white or nearly white grade. A brick red color indicates a high iron content, high density and low absorptivity and such material is unsuitable for the purposes of the invention. A buff color indicates a lower iron content, a lower density and a higher absorptivity, and a material more suitable for use according to the invention. It is also preferable for the kieselguhr to have a low moisture content, and to be uncalcined.

Although the iron content of an initially iron-rich kieselguhr can be reduced by washing it with acid and/or a chelating agent such as oxine or 8-hydroxyguinoline, the resulting material is for the purposes of the invention markedly inferior to a kieselguhr which in its original condition has a very low iron content.

It has further been found that the fluffier and lighter the kieselguhr, the better the performance and it is therefore preferable that the structure of the kieselguhr should be maintained as far as possible. Thus heavy milling for the purpose of producing very fine particles reduces the effectiveness of the kieselguhr, although a light milling is permissible.

It has been observed that kieselguhrs which are substantially iron-free, and have the other preferred properties indicated above, nevertheless differ somewhat in the extent to which they stabilise these insecticides. Accordingly, when a stabilised powder is to be prepared, it is desirable that several grades of suitable kieselguhr should be tested in order that the most effective available grade should be employed.

It should also be noted that the insecticide is not stable, even on the preferred carrier, in the absence of the solvent oil of low volatility, which exerts a protective action. Aromatic or predominantly aromatic oils are preferred, for example those sold under the trademarks Shellsol E and Shellsol R. As alternatives, long-chain alcohols can be employed but are dear and less stable, while chlorinated hydrocarbons are often too volatile.

Kieselguhr is a relatively expensive material, and it is economically undesirable that it should form the bulk of a dilute powder. This is particularly important in the case of dusting powders. Accordingly, a preferred modification of the invention is a powder which comprises an insecticidal powder as hereinbefore defined, in admixture with an inert, solid particulate extender.

A number of solids available at low cost are not suitable for use as extenders in that they increase the rate of decomposition of the insecticide, not being completely inert. The preferred extender is pumice powder, which is relatively cheap and does not affect the stability of the insecticide on the carrier. It should be noted that the insecticide must be applied to the carrier first, and the extender added later. The desired stabilisation will not be achieved if powdered pumice is added to the kieselguhr before the insecticide is applied.

The preferred method for the production of wettable and dispersable powders, which should always be followed if the maximum stability is to be achieved, requires that the wetting and/or suspending agents should only be added after the application of the insecticide to the carrier and the drying of the resulting powder. Heavy milling of these powders is undesirable, although light milling is not harmful. The shelf life of powders prepared in this way is not quite as good as that of the dusting or other powder formulated without wetting or suspending agents, but is adequate for normal purposes.

EXAMPLES

The following are examples of the preparation of stabilised powders according to the invention, in which all quantities are quoted in parts by volume.

Example 1

Deodorised Malathion, 75 parts by volume, was mixed with 25 parts Shellsol E (an 84% aromatic solvent of low volatility) and applied to a powdered, substantially iron-free California kieselguhr of low moisture content and high absorptivity, to give a powder of nominal 25% content of Malathion.

The resulting dry powder was subjected to accelerated storage tests by being maintained for long periods at elevated temperatures in sealed containers. It was found that at temperatures of up to 55° C. the odor did not return in a period of 6½ months. This observation was confirmed by analysis, which showed that the content of

Malathion, originally 25.1%, was still 25.0±0.1% after the same time at 55° C.

Identical results from the standpoint of stability were obtained when "Malathion Technical" was used as the active constituent.

Example 2

The 25% concentrate prepared as in Example 1 was mixed with dry Italian pumice dust to yield powders of various Malathion contents below 25%. All the low dilution powders so prepared had excellent stability when stored at elevated temperatures. The content of a powder initially 3.7% remain unchanged after 8 months hot storage.

Example 3

Deodorised Malathion ("Kypfos 93D), 75 parts, is added to 25 parts of Shellsol E and thoroughly mixed. The solution is then sprayed onto a powdered, substantially iron-free kieselguhr of high absorptivity (Johns Manville "Celite"), while being mixed in a trough mixer, the quantity of insecticide applied being sufficient to achieve a final active content of 25% Malathion.

The result is a dry powder with a faintly peraffinic odor, which stores well, remaining substantially free from offensive odors for at least 4½ months in a sealed container at 55° C.

To produce a wettable powder, a powdered wetting agent, for example Arylan AS40, and a powdered suspending agent, for example Dispersol AT, are mixed with the powder at this stage to give a content of 0.5% wetting agent and 3% suspending agent. If a 25% active content material is required, the quantity of insecticide applied to the carrier must be increased to compensate for the dilution which occurs when the wetting agent is added. The wettable powders so produced remain free from offensive odors for about 3½ months under the conditions specified.

If the solvent is omitted from the above formulation, the odor returns in 24–48 hours under the test conditions mentioned above. Similar instability also occurs if a different absorbent carrier is substituted for the iron-free kieselguhr. The proportion of solvent here described is the most advantageous, and an increase or decrease in this proportion leads to a gradual decrease in shelf life.

It will thus be seen that the invention provides powders which are more stable than conventional formulations of the insecticides concerned. Moreover, if an insecticide is employed which has previously been deodorised, for example an oxidative process, and preferably by the process of U.S. 3,275,499, a powder is obtained which will remain inoffensive in odor for long periods. Dusting powders according to the invention are eminently suitable for the protection of stored grain without contamination thereof.

What is claimed is:

1. A stabilized insectical powder comprising: a phosphorodithioate insecticide; a solvent oil of low volatility; and a particulate carrier composed of substantially iron-free kieselguhr; said insecticide being in admixture with said oil and supported on and stabilized by said carrier.

2. A powder according to claim 1 in which said oil is predominantly aromatic.

3. A powder according to claim 1 in which said insecticide and said oil are present in the proportions of about 75 parts by volume of said insecticide to 25 parts by volume of said oil.

4. A dilute insecticidal powder comprising: a phosphorodithioate insecticide; an aromatic solvent oil of low volatility; a particulate carrier composed of a substantially iron-free kieselguhr; and an inert solid particulate extender; said carrier being impregnated with said insecticide and said oil in admixture whereby said insecticide is stabilized against decomposition, and said impregnated kiselghur being admixed with said extender.

5. A powder according to claim 4 in which said extender is pumice powder.

6. An insecticidal dusting powder comprising O,O-dimethyl S-(1,2-dicarbethoxyethyl) phosphorodithioate; an aromatic solvent oil of low volatility in admixture with said insecticide; and a particulate carrier consisting of pulverulent substantially iron-free kieselguhr impregnated with said insecticide and oil and stabilizing said insecticide.

7. A dusting powder according to claim 6 in admixture with a particulate extender comprising pumice powder.

8. A wettable insecticidal powder comprising a phosphorodithioate insecticide; an aromatic solvent oil of low volatility in admixture with said insecticide; a particulate carrier composed of substantially iron-free kieselguhr impregnated with said insecticide and oil and stabilizing said insecticide; and a powder wetting agent or dispersing agent in admixture with said impregnated carrier.

9. A method of preparing a stabilised insecticidal powder comprising the steps of: admixing a phosphorodithioate insecticide with a solvent oil of low volatility; impregnating pulverulent substantially iron-free kieselguhr with said insecticide and oil mixture; and agitating said impregnated kieselguhr until said mixture is fully absorbed.

10. A method according to claim 9 in which said impregnated kieselguhr is admixed with a wetting agent or dispersing agent and a wettable powder formed thereby.

11. A stabilized insecticidal powder as claimed in claim 1 in which said oil is predominantly aromatic, said insecticide and oil are present in the proportions of about 75 parts by volume of said insecticide to 25 parts by volume of said oil and said powder comprises between about 4 to 25% by volume of said insecticide.

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