

- (54) **PESTICIDAL FORMULATION**
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- (60) Provisional application No. 60/006,346, filed on Nov. 8, 1995.
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- (58) **Field of Search** 424/405, 408-409,
424/717-421; 514/30, 29, 28

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(57) **ABSTRACT**

The present invention is directed to a pesticidal composition of a water-soluble pesticide and processes for its manufacture. More specifically, the invention relates to a soluble granule (SG) pesticidal formulation comprising a water-soluble pesticide and a water-soluble filler. The present invention provides a pesticidal formulation with efficacy equal to the corresponding liquid formulation, yet with improved handler safety, such as lower eye irritation.

11 Claims, No Drawings

PESTICIDAL FORMULATION

CROSS REFERENCE TO RELATED APPLICATIONS

This application is a continuation of U.S. Ser. No. 09/146,463, filed Sep. 3, 1998, ABD, which is a continuation of U.S. Ser. No. 08/744,809, filed Nov. 6, 1996, ABD, which claims priority under 35 U.S.C. § 119(e) from U.S. Ser. No. 60/006,346, filed Nov. 8, 1995.

FIELD OF THE INVENTION

The present invention relates to pesticidal compositions comprising a water-soluble pesticide and process for their manufacture. More specifically, the invention relates to soluble granule (SG) formulations comprising a water-soluble pesticide and a water-soluble filler.

BACKGROUND OF THE INVENTION

Emulsifiable concentrate (EC) agricultural formulations of pesticides are well known in the art. These formulations generally comprise emulsifying and dispersing agents in addition to the pesticide to give a uniform solution. However, these agricultural formulations suffer numerous drawbacks because they are liquid. In particular, liquid pesticidal formulations must be handled carefully to minimize safety concerns, particularly dermal and ocular irritation.

Although solvent-free pesticidal compositions are known, most solvent-free pesticidal formulations have lower bioactivity than the corresponding emulsifiable concentrate formulations. Thus, one skilled in the art frequently has been forced to choose between using a solvent to give a more bioactive formulation but with higher dermal and ocular toxicity, or having a less active solid formulation.

The present invention overcomes the drawbacks of liquid formulations by taking advantage where possible of the water solubility of the pesticide to furnish a composition in divided form so that it will dissolve in water.

Certain methods for preparing water-soluble granules of pesticides are known in the art. PCT Publication No. WO 90/12503 discloses the formation of alkaline salts of soluble acidic or phenolic pesticides to give a granular composition. U.S. Pat. No. 4,511,395 discloses wetttable herbicidal powder compositions which comprise a swelling hydrous silicate clay. EPO Publication No. EP 0,111,112 discloses pesticide granules comprising an inert carrier and a pesticide, and which are coated with finely divided silica.

The use of lactose as a solid filler to make tablets is well known in the pharmaceutical industry. (See e.g. Lerk, C. F., "Consolidation and Compaction of Lactose" *Drug Development and Industrial Pharmacy*, 19, 2359 (1993); "Role of Ingredients and Excipients in Developing Pharmaceuticals" *Manufacturing Chemist*, p. 32 (April 1994). Nevertheless, the use of lactose or other water-soluble fillers as agricultural inert ingredients is not common in formulating pesticides for crop protection. Hudson and Tarwater (Reduction of Pesticide Toxicity by Choices of Formulation, J. L. Hudson and O. R. Tarwater, in *ACS Symposium series #371, Pesticide Formulations*, B. Cross and H. B. Scher, eds.) discuss the effect the choice of formulation on toxicity, and state (on p. 129) that the ocular irritation can be very different for different formulations of the same active ingredient, and that changes in the other toxicity categories may occur.

table powders (WP), water dispersible granules (WDG) or dry flowables (DF), but they do not even mention soluble granule (SG) formulations.

The soluble granule (SG) formulation of the present invention has numerous advantages over the corresponding liquid formulation of a given pesticide. Because it is a solid, the SG formulation exhibits improved ease of handling relative to the liquid formulation. Nevertheless, when dissolved in water the SG formulation provides a clear solution which is aesthetically pleasing. The SG formulation also minimizes the use of ingredients of environmental concern by eliminating the need for volatile organic compounds and employing fillers which are biologically derived, thereby diminishing the potential environmental impact. In addition, formulations of higher concentration (hence lower cost per kg of active ingredient formulated) can be achieved without any loss in biological activity with respect to a liquid formulation. Because the dried granules in the SG formulation are of roughly constant bulk density, the granules may be measured by volume, thus making them equally convenient to liquid formulations in ease of use.

More importantly, however, use of the instant SG formulation is less risky to personnel than the corresponding liquid formulation. Risk of exposure is generally evaluated under the guide: risk=hazard×exposure. Because the instant SG formulation is less toxic, the hazard is decreased. In addition, the probability of contact is lower when the material is in a solid rather than a liquid form and hence the degree of exposure is also reduced. The present invention also affords relatively hard non-dusty granules, thereby even further diminishing the risk of exposure to dust from the composition. Also, the present invention may be practiced without the use of mineral fillers which contain crystalline silica, a known carcinogen. Thus, the present invention diminishes the risk of exposure and provides greater handler safety, especially with regard to ocular irritation, than a liquid formulation.

Accordingly, the present invention provides a relatively safe, bionatural delivery system for a pesticide which is water soluble at the concentration at which it is employed.

SUMMARY OF THE INVENTION

The present invention relates to a pesticidal composition of a water-soluble pesticide and processes for its manufacture. More specifically, the invention relates to a soluble granule (SG) pesticidal formulation comprising a water-soluble pesticide and a water-soluble filler. The present invention provides a pesticidal formulation with efficacy equal to the corresponding liquid formulation, yet with improved handler safety, such as lower eye irritation.

DETAILED DESCRIPTION OF THE INVENTION

The present invention is directed to an pesticidal composition comprising a water-soluble pesticide and processes for its manufacture. More specifically, the invention relates to a soluble granule (SG) formulation comprising a water-soluble pesticide, such as emamectin benzoate, and a water-soluble filler, such as lactose.

The pesticidal composition of the present invention comprises: a water-soluble pesticide; and a water-soluble filler.

The present pesticidal composition may further comprise: a wetting surfactant; and/or a dispersing surfactant. Optionally, a defoaming agent may also be present.

erably the water-soluble filler is selected from: lactose, sucrose, glucose, and the like. Preferably the wetting surfactant is selected from: sodium N-methyl-N-oleyl taurate, sodium N-methyl-N-palmityl taurate, sodium N-methyl-N-oleoyl taurate, sodium dioctyl sulfosuccinate and other sodium alkyl sulfosuccinates, sodium lauryl sulfate, alpha-(p-nonylphenyl)-omega-hydroxy poly(oxyethylene) with an average of 8–12 moles of ethylene oxide, and alpha-(p-octylphenyl)-omega-hydroxy poly(oxyethylene) with an average of 7–12 moles of ethylene oxide. Preferably the dispersing surfactant is selected from: sodium alkyl naphthalene sulfonate, sodium naphthalene sulfonate, calcium lignosulfonate, sodium lignosulfonate, and ammonium lignosulfonate. Preferably the defoaming agent is selected from: methylated silicones, polyorganosiloxane, and 2-ethylhexanol.

More preferably the water-soluble pesticide is emamectin or emamectin benzoate. More preferably the water-soluble filler is lactose. More preferably the wetting surfactant is sodium N-methyl N-oleyl taurate. More preferably the dispersing surfactant is sodium alkyl naphthalene sulfonate. More preferably the defoaming agent is polyorganosiloxane.

The pesticidal composition of the present invention may be provided in the form of a wettable powder but preferably is in the form of a water-soluble granule. Similarly, the pesticidal composition of the present invention may be provided as an aggregate, a matrix, or a monolith, such as a brick, pellet, tablet, stick, film, sheet, and the like. Preferably, the pesticidal composition of the present invention is embedded in a water-soluble matrix or monolith. It also will be appreciated by one skilled in the art that the present invention further includes encasement of the instant pesticidal compositions in a water-soluble package, such as a pouch, sachet, bag, capsule, and the like.

The pesticidal compositions of the present invention comprise 0.1 to 90% by weight of a water-soluble pesticide, preferably emamectin benzoate; and 30 to 99.9% by weight of a water-soluble filler, preferably lactose (not to the exclusion of other ingredients).

It will be appreciated by one skilled in the art that the sum of the proportions of the water-soluble pesticide and the water-soluble filler are not greater than 100% by weight and that the exact concentrations of the components may vary depending on the presence of impurities.

In a more preferred embodiment, the pesticidal compositions of the present invention comprise 0.1 to 60% by weight of a water-soluble pesticide, preferably emamectin benzoate; 40 to 99.9% by weight of a water-soluble filler; 0 to 50% by weight of a wetting surfactant; 0 to 50% by weight of a dispersing surfactant; 0 to 5% by weight of a defoaming agent (not to the exclusion of other ingredients).

It will be appreciated by one skilled in the art that the sum of the proportions of the water-soluble pesticide, the water-soluble filler, the wetting surfactant, the dispersing surfactant and the defoaming agent are not greater than 100% by weight and the exact concentrations of the components may vary depending on the presence of impurities.

In one embodiment of the present invention the formulation contains about 0.1 to 90% by weight of the water-soluble pesticide.

Preferred pesticidal compositions of the present invention comprise 0.1 to 60% by weight of emamectin benzoate; 40 to 99.9% by weight of lactose; 0 to 25% by weight of sodium N-methyl N-oleyl taurate; 0 to 10% by weight of sodium alkyl naphthalene sulfonate; and 0 to 3% by weight of

More preferred pesticidal compositions of the present invention comprise 1 to 50% by weight of emamectin benzoate; 40 to 99% by weight of lactose; 0 to 10% by weight of sodium N-methyl N-oleyl taurate; 0 to 2% by weight of sodium alkyl naphthalene sulfonate; and 0 to 1% by weight of polyorganosiloxane (not to the exclusion of other ingredients). Even more preferred pesticidal compositions are those which may comprise 1 to 20% by weight of emamectin benzoate.

Even more preferred pesticidal compositions of the present invention comprise 1 to 20% by weight of emamectin benzoate; 60 to 99% by weight of lactose; 5 to 10% by weight of sodium N-methyl N-oleyl taurate; 0.5 to 2% by weight of sodium alkyl naphthalene sulfonate; and 0 to 0.5% by weight of polyorganosiloxane (not to the exclusion of other ingredients). Still more preferred pesticidal compositions are those which comprise 1 to 10% by weight of emamectin benzoate.

It will be appreciated by one skilled in the art that the sum of the proportions of emamectin benzoate, lactose, sodium N-methyl N-oleyl taurate, sodium alkyl naphthalene sulfonate and polyorganosiloxane are not greater than 100% by weight and that the exact concentrations of the components may vary depending on the presence of impurities.

Especially preferred pesticidal formulations are as follows:

Soluble granules comprising about 5.3% by weight of emamectin benzoate; about 86% by weight of lactose; about 7.5% by weight of sodium N-methyl N-oleyl taurate; about 1% by weight of sodium alkyl naphthalene sulfonate; about 0.1% by weight of polyorganosiloxane.

The present invention further includes a process for preparing a soluble granule formulation which process comprises:

- (1) forming a powder blend of a water-soluble pesticide and a water-soluble filler;
- (2) adding water to the blend;
- (3) extruding the wet blend through a die to form granules; and
- (4) drying the granules to remove the water.

The present invention is further directed to a soluble granule formulation prepared by such process.

In a preferred embodiment, the process for preparing a soluble granule formulation comprises:

- (1) forming a powder blend of a water-soluble pesticide, a water-soluble filler, a wetting surfactant and a dispersing surfactant;
- (2) adding water to the blend;
- (3) extruding the wet blend through a die to form granules; and
- (4) drying the granules to remove the water.

Optionally, in this process a defoaming agent may be added to the powder blend.

The present invention is further directed to a soluble granule formulation prepared by such process.

Specifically exemplifying the present invention is the process comprising the steps of:

- (1) forming a powder blend of a emamectin benzoate and lactose;
- (2) adding water to the blend;
- (3) extruding the wet blend through a die to form granules; and
- (4) drying the granules to remove the water.

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- (1) forming a powder blend of a emamectin benzoate, lactose, sodium alkyl naphthalene sulfonate, sodium N-methyl N-oleyl taurate, and polyorganosiloxane;
- (2) adding water to the blend;
- (3) extruding the wet blend through a die to form granules; and
- (4) drying the granules to remove the water.

The present invention is further directed to a soluble granule formulation of emamectin benzoate prepared by such process.

The present invention is further directed to a delivery system for water-soluble pesticides, in particular, emamectin benzoate, which comprises soluble granules and the use thereof.

The present invention further provides a method for administering the water-soluble pesticide comprising:

- (1) formulating the water-soluble pesticide as a soluble granule;
- (2) mixing with water a portion of the soluble granule sufficient to give the desired amount of active ingredient of the water-soluble pesticide; and
- (3) applying the pesticide/water mixture.

The present invention is further directed to a pesticide/water mixture prepared by such process.

The application of the pesticide/water mixture may be conducted by methods well known in the art, including, spraying, misting, dripping, infusing, injecting, irrigating, and the like.

The term "water-soluble pesticide" as used herein includes any biologically active pesticidal agent or crop protection chemical which is water soluble at the concentration in which it is employed (i.e. the agent is water soluble at the volume used in field application). Such water-soluble pesticides include certain fungicides, herbicides, such as plant growth regulators, and insecticides, such as nematocides, anti-helmentics, and miticides. Exemplary water-soluble pesticides which may be employed in the present invention include: the fungicides: blasticidin-S, kasugamycin, and hymexanol; the herbicides: acifluorfen, glyphosate, and glufosinate; the plant growth regulators: gibberellic acid, maleic hydrazide and dikegulac; and the insecticides: acephate, emamectin, and emamectin benzoate. A particularly preferred water-soluble pesticide is emamectin, or an agriculturally acceptable salt thereof, such as the salt formed with benzoic acid, salicylic acid, gallic acid, benzenesulfonic acid, hydrochloric and citric acid, especially emamectin benzoate. One skilled in the art will readily appreciate that these pesticides exhibit sufficient water solubility that they will dissolve when mixed with water at the labeled use rate. For example, commercial label use rates of acephate are as high as 1.33 lb (0.60 kg) in a minimum of 3 gallons (11.4 liters) of water. Because 650 g of acephate is soluble in 1 liter of water, the acephate is fully soluble in water at its maximum label use rate. Similarly, emamectin benzoate is soluble at about 100 ppm in water. Thus, at a typical use rate 3.4 g of emamectin benzoate will be soluble in about 34 liters of water.

The term "water-soluble granule" as used herein is intended to include any granular or pelletized compositions which permit the active ingredient to be in solution after dissolving in water. It is not necessary for all of the ingredients of the water-soluble granule to be in solution as long as the active ingredient itself is in solution. Thus, the present invention includes within its scope water-soluble granules in which some of the ingredients may be relatively insoluble. The actual physical form of the instant pesticidal composition

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The term "water-soluble filler" as used herein includes any water soluble or water dispersible agent which may be employed to dilute the pesticide. Preferred water-soluble agents include those which are biologically derived. Appropriate water-soluble fillers include lactose, glucose, fructose, mannose, mannitol, sucrose, such as confectioner's sugar, black sugar, brown sugar, soft brown sugar, other sugars or saccharides, microcrystalline cellulose, powdered cellulose, calcium phosphate(s), inorganic water-soluble salts, and the like, and mixtures thereof. Examples of lactose which can be used in the present invention include hydrated α -lactose, anhydrous α -lactose, hydrated β -lactose, anhydrous β -lactose, and the like, and mixtures thereof.

Any surfactants can be used as the surfactant in the present invention, as long as it will emulsify the desired pesticide, or the pesticide when mixed with water. Examples of such surfactants include glycerine fatty acid esters, sucrose fatty acid esters, sorbitan fatty acid esters, fatty acid salts, alkyl sulfates, alkylbenzene sulfonic acid salts, alkyl aryl ethers and polyoxyethylenated products thereof, ethylene oxide addition products of higher alcohols, polyoxyethylene polyoxypropylene glycol, lignin sulfonic acid salt, polyoxyethylene styrylphenyl ether, polyoxyethylene alkyl esters, alkyl aryl sulfates, and the like. The surfactants may be used singly or in a suitable combination. Surfactants which can uniformly mix with the desired pesticide or the pesticides and water are preferred but it is not always necessary to use such surfactants. It is sufficient that surfactants be dissolved in water when the pesticidal composition is diluted with water.

The term "wetting surfactant" as used herein includes any agent which may be employed to enhance the wetting properties of the formulation such as sodium N-methyl-N-oleyl taurate, sodium N-methyl-N-palmityl taurate, sodium N-methyl-N-oleoyl taurate, sodium dioctyl sulfosuccinate and other sodium alkyl sulfosuccinates, sodium lauryl sulfate, alpha-(p-nonylphenyl)-omega-hydroxy poly(oxyethylene) with an average of 8-12 moles of ethylene oxide, and alpha-(p-octylphenyl)-omega-hydroxy poly(oxyethylene) with an average of 7-12 moles of ethylene oxide.

The term "dispersing surfactant" as used herein includes any agent which may be employed to enhance the dispersion of the formulation when mixed in water such as sodium alkyl naphthalene sulfonate, sodium naphthalene sulfonate, calcium lignosulfonate, sodium lignosulfonate, and ammonium lignosulfonate.

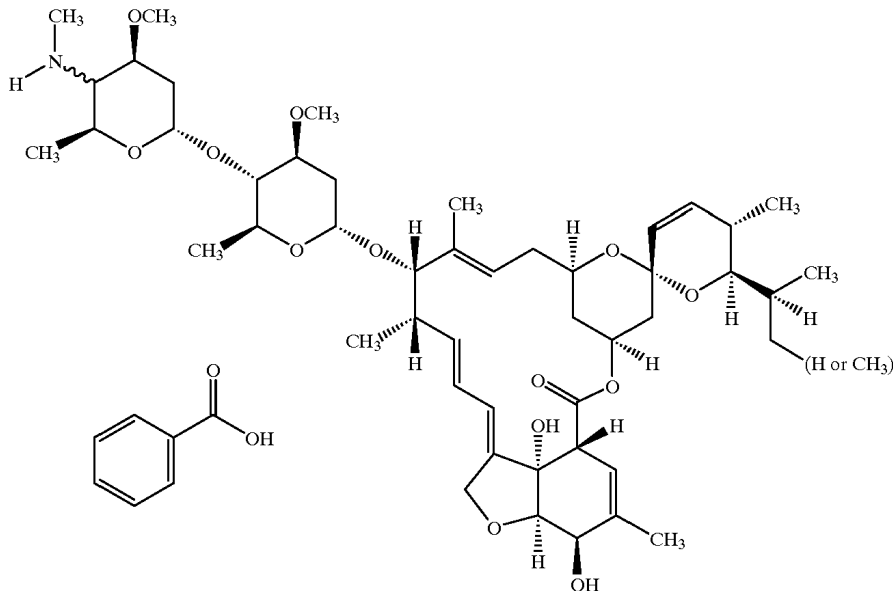
The term "defoaming agent" as used herein includes any agent which may be employed to reduce the tendency of the formulation to induce the generation of foam when added to water such as methylated silicones, polyorganosiloxane, and 2-ethylhexanol.

In addition to the pesticide, the water-soluble filler, the wetting surfactant, the dispersing surfactant and the defoaming agent, the instant pesticidal compositions may also appropriately contain stabilizers, synergists, coloring agents, etc.

The pesticidal compositions of the present invention are generally spread after diluting with water. One skilled in the art will appreciate that when diluted with water, the dilution magnification of the pesticidal composition varies depending upon the kind of pesticide, the kind of harmful organism to be controlled, the kind of crops to be treated, the kind of blight to be mitigated, the kind of weeds to be controlled, the time period for treatment, the method of application, etc. and

A preferred water-soluble pesticide for use in the formulations of the present invention is emamectin benzoate. Certain avermectin B1a/B1b compounds which have activity as agricultural insecticides are disclosed in U.S. Pat. No. 4,310,519 (issued Jan. 12, 1982). The compound 4"-deoxy-4"-epi-methylamino avermectin hydrochloride is disclosed in U.S. Pat. No. 4,874,749 (issued Oct. 17, 1989) as having properties as an agricultural insecticide. Stable salts of 4"-deoxy-4"-epi-methylamino avermectin B1a/B1b are disclosed in U.S. Pat. No. 5,288,710 (issued Feb. 22, 1994).

In particular, U.S. Pat. No. 5,288,710 discloses the benzoate salt of 4"-deoxy-4"-epi-methylamino avermectin B1a/B1b (emamectin benzoate) which has the structure, e.g.:



This compound may be readily prepared by the methodology describe therein. The other ingredients in the subject formulations are either commercially available, or are readily prepared by methods known in the art.

The use of the present formulations will be readily apparent to one skilled in the art. In particular, formulations comprising emamectin benzoate may be utilized e.g. as described in U.S. Pat. No. 5,288,710.

The pesticidal compositions of the present invention may be used to exterminate or control harmful organisms or regulate plant growth.

The pesticidal compositions of the present invention are particularly useful in combating agricultural pests that inflict damage upon crops while they are growing or while in storage. The compounds are applied using known techniques as sprays, dusts, emulsions and the like, to the growing or stored crops to effect protection from such agricultural pests.

For example, the compound emamectin and the agriculturally acceptable salts thereof, in particular, emamectin benzoate exhibit significant parasiticidal activity as anthelmintics, ectoparasiticides, insecticides and acaricides, in human and animal health and in agriculture.

Emamectin and the agriculturally acceptable salts thereof are useful against insect pests of stored grains such as *Tribolium* sp., *Tenebrio* sp., and of agricultural plants such as spider mites, (*Tetranychus* sp.), aphids, (*Acyrtosiphon* sp.); against migratory orthopterans such as locusts and

nematodes and plant parasites such as *Meloidogyne* sp. which may be of importance in agriculture. These pesticides are also active against other plant pests such as the southern army worm and Mexican bean beetle larvae. These pesticides also have activity against *Dirofilaria* in dogs; *Namatospiroides*, *Syphacia*, *Aspicularis* in rodents; the arthropod ectoparasites of animals and birds such as ticks, mites, lice, fleas, and blowfly; in sheep *Lucilia* sp.; biting insects and such migrating dipterous larvae as *Hypoderma* sp. in cattle; *Gastrophilus* in horses; and *Caterebra* sp. in rodents. In addition these compounds are also active against parasitic worms known as helminths. Helminthiasis is a prevalent and serious economic problem in domesticated

animals such as swine, sheep, horses, cattle, goats, dogs, cats and poultry. Among the helminths, the group of worms described as nematodes causes widespread and often times serious infection in various species of animals. The most common genera of nematodes infecting the animals referred to above are *Haemonchus*, *Trichostrongylus*, *Ostertagia*, *Nematodirus*, *Cooperia*, *Ascaris*, *Bunostomum*, *Oesophagostomum*, *Chabertia*, *Trichuris*, *Strongylus*, *Trichonema*, *Dictyocaulus*, *Capillaria*, *Heterakis*, *Toxocara*, *Ascaridis*, *Oxyuris*, *Ancylostoma*, *Uncinaria*, *Toxascaris* and *Parascaris*. Certain of these, such as *Nematodirus*, *Cooperia* and *Oesophagostomum* attack primarily the intestinal tract while others, such as *Haemonchus* and *Ostertagia*, are more prevalent in the stomach while still others such as *Dictyocaulus* are found in the lungs. These pesticides are also useful against parasites which infect humans. The most common genera of parasites of the gastro-intestinal tract of man are *Ancylostoma*, *Necator*, *Ascaris*, *Strongyloides*, *Trichinella*, *Capillaria*, *Trichuris*, and *Enterobius*. Other medically important genera of parasites which are found in the blood or other tissues and organs outside the gastrointestinal tract are the filarial worms such as *Wuchereria*, *Brugia*, *Onchocerca* and *Loa*, *Dracunculus* and extra intestinal stages of the intestinal worms *Strongyloides* and *Trichinella*. The compounds are also of value against arthropods parasitizing man, biting insects and, other dipterous pests causing annoyance to man. These pesticides are also active against household pests such as the cockroach, *Blattella* sp.,

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