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(54) **Plant-protection compositions in the form of granules**

(57) Effervescent bioactive compositions comprise an acid herbicide or plant growth regulator and an alkali metal carbonate or bicarbonate

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SPECIFICATION

Plant-protection compositions in the form of granules

- The present invention relates to bioactive compositions consisting of effervescent granules containing an acid herbicide or growth regulator, an alkali metal carbonate or bicarbonate and, if appropriate, additives such as mould-release agents, binders and/or fillers.
- The herbicide acifluorfen sodium salt, marketed under the trademarks Tackle and Blazer and consisting of sodium 5-[2-chloro-4-(trifluoromethyl)-phenoxy]-2-nitro-benzoate, is known; acifluorfen is itself 5-[2-chloro-4-(trifluoromethyl)-phenoxy]-2-nitrobenzoic acid.
- The marketed formulations of acifluorfen sodium salt are compositions comprising water (76%) and only about 24% of herbicide as the main constituents.
- One object of the present invention is to produce dry herbicidal compositions which are readily dispersible in water and which contain a relatively high concentration of herbicidal active ingredient.
- On this point, it must be noted that, being hygroscopic, acifluorfen sodium salt is not in itself a suitable herbicidal concentrate in commercial terms.
- Another object of the present invention is to provide herbicidal compositions which are not in powder form. In fact, the people who apply pesticidal products are increasingly dissatisfied with compositions in powder form, because of the harmful effects and the inconvenience of handling powders (the need for a mask, staining, and the like).
- Furthermore, various kinds of water-dispersible pesticidal granules are well known, especially those called "dry flowables". In particular, effervescent granules have been proposed (French Patent 2,290,844 and British Patent 847,370), these granules comprising at least three essential constituents: the active ingredient, a carbonate and an acid capable of reacting with the carbonate.
- Another object of the present invention is to provide herbicidal compositions which are as simple as possible and contain a high proportion of active ingredient.
- Another object of the present invention is to provide dry herbicidal compositions which can easily be used to apply active ingredients such as acifluorfen sodium salt.
- It has now been found that these various objects can be achieved by virtue of the compositions according to the present invention.
- The present invention thus relates to bioactive pesticidal compositions, and more particularly herbicidal compositions, in the form of water-soluble granules, the said granules comprising 50 to 90% by weight of an acid herbicide or plant growth regulator, preferably an acid herbicide and about 10 to 40% by weight of an alkali metal carbonate or bicarbonate, the alkali metal being in particular potassium or, preferably, sodium, the said compositions containing also 0 to 10% by weight of impurities of the acid herbicide (such as isomers or manufacture by-products). The amount of acid active ingredient is preferably between 60 and 75% by weight.
- It must be clearly understood that the term "granules" is not to be taken in a restricted sense, and that it includes any other similar form such as grains, pellets or briquettes.
- In general, these granules have a minimum size of between 0.1 and 50 mm, preferably of between 0.3 and 3 mm.
- The amount of herbicidal active ingredient, relative to the carbonate, is such that the molar ratio carbonate or bicarbonate/acid active ingredient is between 1 and 3, preferably between 1.3 and 2.
- The compositions according to the present invention can contain minor amounts (e.g. 0.1 to 10% by weight) of other additives, such as lubricants or mould-release agents (e.g. 0.05 to 1% or less of aluminium stearate), stabilisers, binders, agents promoting cohesion of the granules (e.g. urea or cellulose derivatives) and agents promoting disintegration of the granules, or inert fillers, including water-insoluble inert fillers.
- The compositions according to the invention can also contain other additives which are useful for the application of the pesticide in question. Thus, it may be necessary to add surface-active agents or wetting or dispersing agents or any other product capable of promoting the penetration of the active ingredient into the plant.
- These surface-active agents can be emulsifying, dispersing or wetting agents of ionic or non-ionic type, or a mixture of such surface-active agents. Examples which may be mentioned are salts of polyacrylic acids, salts of lignosulphonic acids, polycondensates of ethylene oxide with fatty alcohols, fatty acids or fatty amines, substituted phenols (in particular alkylphenols or arylphenols), salts of sulphosuccinic acid esters, taurine derivatives (in particular alkyltaurates), phosphoric acid esters of condensates of ethylene oxide with alcohols or phenols, fatty acid esters of polyols, and derivatives of the above compounds containing sulphate, sulphonate or phosphate groups.
- Preferably, in the compositions according to the present invention, the grains are separate from one another and they can flow in bulk when poured.
- According to an advantageous variant, the compositions of the present invention do not contain any inert filler, in particular any water-insoluble inert filler.
- Acifluorfen is the active ingredient which is particularly appropriate to the invention, but it is also possible to use 5-[2-chloro-4-(trifluoromethyl)-phenoxy]-2-nitro-N-methanesulphonylbenzamide, 2,4-dichloro-phenoxyacetic acid, 2-methyl-4-chlorophenoxy-acetic acid, naphthalene-1-acetic acid (as a plant

growth regulator) and methyl paraaminophenyl-sulphonylcarbamate.

The granules according to the present invention can be made by mixing and compressing the constituents (e.g. with the aid of a press or a compacter) so as to give a briquette, and then by granulating the compact material obtained.

A spraying mixture (solution or suspension) which is ready for use can be obtained from the granules according to the invention simply by placing the said granules in water, in an amount corresponding to the desired concentration of the active ingredient.

The CO₂ released during effervescence causes rapid agitation of the solution and facilitates salification of the acid; this agitation accelerates the dissolution of the ingredients in the water; furthermore, the use of an acid in the formulation makes it possible to incorporate a higher proportion of active ingredient into the composition than in the case where the salt is added separately: this is a particularly valuable advantage in the field of plant protection, where it is desirable to handle the smallest possible amount of products.

The following non-limitative examples illustrate the present invention and the manner in which it can be carried out.

EXAMPLE 1

The following are mixed:
96% pure acifluorfen (7.23 g)
anhydrous sodium carbonate (1.12 g)
aluminium stearate (0.01 g).

The mixture is ground in a mill with glass beads, the mill containing the same weight of glass beads of diameter 5 mm. The mixture is then sieved and compressed. The pellets obtained are lightly crushed in a mortar and then pounded and sieved, and the fraction of 20 to 60 mesh (U.S. standard specification for sieves, 0.25—0.84 mm) is retained.

On adding these granules to water, effervescence and dissolution take place and a solution of the sodium salt of acifluorfen ready for spraying is obtained, which is clear except that it comprises particles of aluminium stearate in suspension.

The mixture contains 83% of herbicidal acid, which is equivalent to 88% of the sodium salt.

EXAMPLE 2

The following are mixed:
acifluorfen (96% pure) (7.22 g)
sodium bicarbonate (1.85 g)
aluminium stearate (0.01 g).

The mixture is treated as in Example 1. When the granules are added to water, the results obtained are equivalent to those of Example 1.

The mixture contains 76.4% of herbicidal acid, which is equivalent to 81% of the sodium salt.

EXAMPLE 3

The following are mixed:
80% pure acifluorfen (74.9 g)

anhydrous sodium carbonate (25 g)
aluminium stearate (0.1 g).

65 The mixture is finely ground in a mechanical mill so as to give 3 to 50 micron particles.

The mixture thus obtained is compressed under a pressure of 20 bars by a pelletising machine. The pellets obtained are crushed and then sieved so that the grains having a size of 20 to 60 mesh (0.25 to 0.84 mm) are retained. These grains contain about 60% of acifluorfen, which is equivalent to 63.6% of its sodium salt.

When added to water, these granules disaggregate with the release of carbon dioxide and the formation of the sodium salt of acifluorfen, as in the previous Examples 1 and 2.

EXAMPLE 4

The following are mixed:

80 80% pure acifluorfen (63 g)
anhydrous sodium carbonate (25 g)
aluminium stearate (0.1 g).

The mixture is ground as in Example 3.

The mixture obtained is compressed under a pressure of 5 bars by a pelletising machine. The pellets obtained are crushed and then sieved so that the particles having a size of 15 to 30 mesh (0.5 to 1.2 mm) are retained. These grains contain 57.2% of acifluorfen, which is equivalent to 60% of its sodium salt; when they are placed in water, they disintegrate perfectly and give an aqueous solution of the herbicide acifluorfen sodium salt.

EXAMPLE 5

The procedure of Example 3 is followed, but the anhydrous sodium carbonate (25 g) is replaced by anhydrous sodium carbonate (18 g) and urea (7 g).

This gives granules having a similar size, which are perfectly satisfactory as regards their disintegration in water.

EXAMPLE 6

Example 3 is repeated but replacing the acifluorfen by 5-[2-chloro-4-trifluoromethyl)-phenoxy]-2-nitro-N-methanesulphonyl-benzamide, 2,4-dichlorophenoxyacetic acid, 2-methyl-4-chlorophenoxyacetic acid or naphthalene-1-acetic acid.

Satisfactory granules which disintegrate well in water are obtained in the same manner.

110 CLAIMS

1. A bioactive composition in the form of water-soluble granules, the said granules comprising 50 to 90% by weight of an acid herbicide or plant growth regulator and from 10 to 40% by weight of an alkali metal carbonate or bicarbonate, the said composition containing also 0 to 10% by weight of impurities of the acid herbicide.

2. A composition according to claim 1, wherein the active ingredient is an acid herbicide.

3. A composition according to claim 1 or 2, wherein the alkali metal carbonate or bicarbonate is sodium carbonate or bicarbonate.

4. A composition according to any one of

- claims 1 to 3, which contains 60 to 75% by weight of acid herbicide.
- 5 A composition according to any one of claims 1 to 4, wherein the molar ratio of carbonate or bicarbonate/acid herbicide or plant growth regulator is between 1 and 3, preferably between 1.3 and 2.
- 10 6. A composition according to any one of claims 1 to 5, wherein the granules are separate from one another, are capable of flowing in bulk and do not contain a water-insoluble inert filler.
- 15 7. A composition according to any one of claims 1 to 6, which contains 0.1 to 10% by weight of an additive such as a lubricant, mould-release agent, stabiliser, binder, agents promoting cohesion of the granules, agents promoting disintegration of the granules or a surface-active agent.
- 20 8. A composition according to any one of claims 1 to 7, which contains 0.05 to 1% of aluminium stearate.
9. A composition according to any one of claims 1 to 8, which contains urea.
- 25 10. A composition according to any one of claims 1 to 10, wherein the herbicide is acifluorfen.
- 30 11. A composition according to any one of claims 1 to 10 wherein the acid active ingredient is chosen from the group comprising 5-[2-chloro-4-(trifluoromethyl)-phenoxy]-2-nitro-N-methanesulphonylbenzamide, 2,4-dichlorophenoxyacetic acid, naphthalene-1-acetic acid, 2-methyl-4-chlorophenoxyacetic acid or methyl para-aminobenzenesulphonylcarbamate.