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(54) Title: HERBICIDAL COMPOSITION AND METHOD OF USE THEREOF

(57) Abstract: A method for preparing a formulation comprising clomazone is disclosed, the method comprising the steps of (i) combining microcapsules having a polymer shell and a core comprising clomazone, a solid carrier and a binder to form a mixture; (ii) forming granules from the resulting mixture; (iii) applying a composition comprising a binder to coat the granules; and (iv) drying the thus coated granules. A further method for preparing a formulation comprising clomazone is provided, the method comprising the steps of (i) combining microcapsules having a polymer shell and a core comprising clomazone, a solid carrier and a dispersant to form a mixture; (ii) forming granules from the resulting mixture; (iii) applying a composition comprising a dispersant to coat the granules; and (iv) drying the thus coated granules. A still further method for preparing a formulation comprising clomazone is provided, the method comprising the steps of (i) combining microcapsules having a polymer shell and a core comprising clomazone, a solid carrier, a binder and a dispersant to form a mixture; (ii) forming granules from the resulting mixture; (iii) applying a composition comprising a dispersant to coat the granules; (iv) applying a composition comprising a binder to coat the granules; and (v) drying the thus coated granules. Granules prepared by the methods and their use in controlling plant growth are also disclosed.



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HERBICIDAL COMPOSITION AND METHOD OF USE THEREOF

5 The present invention relates to a composition comprising a herbicide and to the use of the composition. In particular, the present invention concerns an extruded granular composition comprising a herbicidally active component, in particular a certain isoxazolidinone.

10 It is known that certain isoxazolidinones are active as herbicides and are useful in the control of unwanted plant growth. One particular isoxazolidinone of commercial importance is 2-(2-chlorophenyl)methyl-4,4-dimethyl-3-isoxazolidinone, known in the art and referred to hereinafter by the common name clomazone.

15 A problem with certain isoxazolidinone herbicides, in particular with clomazone, arises from the relatively high volatility of the compounds. In conventional formulations of such active ingredients, the compounds have the tendency to vapourise rapidly from the surfaces to which the formulations are applied. The vapourised active ingredient is susceptible to drift from the target area or locus, with the result that plants outside the target area may suffer adverse effects from the
20 compounds. Consequently, there is a need to avoid such secondary effects of volatile active compounds, such as clomazone.

To reduce the rapid vapourisation of the active compound, it is known to encapsulate clomazone, in particular to encapsulate a liquid formulation of the
25 compound within a polymer. Such microencapsulation of clomazone is taught, for example, in US 4,405,357. The use of microcapsules allow the active ingredient to be held in relatively large liquid droplets and reduces the rate at which the active ingredient vapourises after application. This in turn reduces the secondary effects of the active ingredient on plants outside the target area or locus.

30 Problems have been found with certain known microencapsulated forms of clomazone. In particular, it has been found that the presence of significant quantities of moisture at the target site, for example water absorbed by fertilizer present at the

time of application, can increase the volatility of the active ingredient even when microencapsulated as described above.

This problem is addressed in US 6,797,277, which proposes an extruded granular formulation for delivering active compounds with relatively high volatilities, in particular clomazone. In particular, US 6,797,277 proposes a process for making an extruded granule for delivering an agrochemical, in particular clomazone, to a crop, in which clomazone is blended with a solid carrier to form a blended composition, which is extruded through a die to form granules. The clomazone may or may not be microencapsulated. Suitable solid carriers disclosed are aluminium silicate, kaolin, montmorillinite, attapulgite, Black Charm Clay, bentonite, corn cob, sand, biodac, starch matrix, fluid earth carbon, activated carbon, or other solid diluents. Aluminium silicate is a preferred solid carrier. The granules may comprise a binder. The binder may be included in the composition comprising clomazone and the carrier before extrusion. Alternatively, the binder may be applied to the granules after extrusion, for example by way of a spray coating. The binder may be a lignosulfonate, molasses, polysaccharides, methylcellulose, acrylic polymers, bentonite, rendering fat or water soluble glues. Further, the granules may comprise a dispersant. The dispersant, if present, may be included in the composition before extrusion. Alternatively, the dispersant may be applied to the granules after extrusion, for example by spray coating. The dispersant may be a lignosulfonate, condensed naphthalene sulfonates, swelling bentonite and acid/base effervescent systems. US 6,797,277 appears to favour the use of both a lignosulfonate as the binder and the dispersant.

The sole example of an actual composition disclosed in US 6,797,277 comprises an aluminium silicate clay solid carrier, microencapsulated clomazone, a lignosulfonate binder, a lignosulfonate dispersant and water. The granular composition is described as being prepared by spraying the microencapsulated clomazone onto the clay and blending to form a uniform mixture. The resulting mixture was extruded to form granules. The resulting granules were coated with a solution of the binder and the dispersant. Finally, the granules were dried to a moisture content of from 5 to 6%.

There is a need for an improved formulation of isoxazolidinones, in particular clomazone, that addresses the problems of high volatility of the active ingredient, while avoiding the problems associated with granular formulations, such as the formation of fines or dust resulting from attrition of the granules during manufacture,
5 transport and use.

According to a first aspect of the present invention there is provided a method for preparing a formulation comprising clomazone, the method comprising the steps of:

- 10 combining microcapsules having a polymer shell and a core comprising clomazone, a solid carrier and a binder to form a mixture;
forming granules from the resulting mixture;
applying a composition comprising a binder to coat the granules; and
drying the thus coated granules.

15

The method of this aspect of the present invention provides an improved granular formulation of clomazone, that is particularly stable, more resistant to abrasion, while effective in delivering the active ingredient to the target plants once the formulation is applied at a locus, with minimal drift of the active ingredient to
20 plants outside the locus of application.

20

The method comprises forming a mixture of microencapsulated clomazone, as described in more detail hereinafter, a solid carrier and a binder. The solid carrier may be any suitable material that is susceptible to forming granules, in particular by
25 extrusion, and is acceptable for application to plants at a locus, once the finished formulation is applied. The solid carrier is generally inert, that is does not adversely affect the activity of clomazone or affect the action of other components of the finished granules. Suitable carriers include compositions comprising one or more metal salts, in particular salts of metals from Groups IA, IIA and IIIA of the Periodic
30 Table of Elements. The carrier may also comprise a transition metal salt. Preferred metal salts include those of calcium, magnesium and aluminium. Any suitable salt may be employed, in particular oxides, carbonates, sulphates, and silicates. Suitable carrier materials include clays, such as kaolinites, smectites, montmorillinites,

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bentonites, chlorites, sepiolites and atapulgites. These and other suitable solid carriers are known in the art and are generally commercially available.

5 The solid carrier may be present in any suitable amount, in particular forming from 20 to 98% by weight of the final product, preferably from 40 to 95% by weight, more preferably from 50 to 90% by weight of the finished granules.

10 The mixture further comprises one or more binders. The binder serves to adhere or bind the solid particles present in the final product and provides integrity to the granules, preventing them from crumbling and the like. The binder may be any suitable material and suitable binder materials are known in the art and are commercially available. Suitable binders include flours, starches, gums, alcohols, lipids, cellulose derivatives, alginates, and wood extracts, such as lignin and lignin derivatives, for example lignosulfonates.

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The binder is present in the mixture in sufficient amount to provide the extruded granules with integrity and will vary according to the properties of the binder being used. Typically, the binder is present in the final granular product in an amount of from 0.2 to 15% by weight, preferably from 0.5 to 10% by weight, more preferably
20 from 1 to 5% by weight.

Depending upon the composition and properties of the other components of the mixture to be formed into granules and the method of granulation, the mixture may further comprise a solvent. Suitable solvents include both organic and inorganic
25 liquids. Water is a particularly convenient solvent. Other suitable solvents include organic liquids, such as alcohols, aromatic solvents and the like. Again, suitable solvents are known in the art and are commercially available.

30 The amount of solvent present in the mixture will depend upon the properties and composition of the other components. For example, certain microencapsulated formulations of clomazone comprise the microcapsules in suspension in a solvent, in particular in aqueous suspension. Accordingly, solvent will be provided to the mixture by the clomazone formulation being used. The solvent should be present in an appropriate amount to allow the mixture to be formed into granules, for example to

be extruded, in particular from 1 to 20% by weight of the mixture, preferably from 2 to 10% by weight, more preferably from 2 to 5% by weight.

5 The mixture to be extruded may consist of microencapsulated clomazone, one or more solid carriers, and one or more binders. However, the mixture may comprise further components. In particular, the mixture to be extruded may comprise one or more dispersants. The dispersant is provided to increase the rate at which the granules disintegrate once they are delivered to the locus being treated and contacted with water, to release the active ingredient. The dispersant may comprise 10 an anionic dispersant material, a non-ionic dispersant material, or a mixture of the two. Suitable dispersants for inclusion in the granules are known in the art and are commercially available. Examples of suitable dispersants include sulphonated and unsulphonated lignin polymers, sulphonated naphthalene compounds and polyacrylate salts. Sulfonated lignin dispersants are preferred anionic dispersant 15 materials. A preferred non-ionic dispersant is naphthalene sulfonate.

If present, the mixture may contain any suitable amount of the dispersant to ensure sufficient disintegration of the granules upon application, in particular from 0.5 to 20% by weight of the finished granules, preferably from 1 to 15% by weight, more 20 preferably from 2 to 10% by weight.

The components of the mixture may be combined in any order and mixed. Suitable techniques for mixing the components and apparatus for performing the mixing are known in the art, with suitable equipment being commercially available. In 25 addition, or alternatively, the components may be mixed as a result of the action of the process for forming the mixture into granules. In one embodiment, the mixture is formed into granules by extrusion, as discussed below, in which case the components may be added directly to the inlet or feed hopper of the extruder, with the mixing taking place during the extrusion process.

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In the following step of the method of this invention, the mixture described above is formed into granules. The granules may be formed by any suitable technique. Suitable techniques are known in the art and include granulation, compaction, spray drying, fluid bed agglomeration and extrusion. Extrusion is a

particular preferred manner of forming the mixture into granules. Suitable extrusion techniques and apparatus are well known in the art, with the equipment being commercially available.

5 Once the granules are formed, they are treated to apply additional binder to the exterior of the granules. The binder may comprise the same or different materials to the binder included in the initial mixture. Suitable binder materials are as described above. The binder may be applied to the granules by any suitable means. Preferably, the binder is formed as a solution, suspension or slurry of the binder
10 material in a suitable solvent. The solvent is most preferably water. The granules may be contacted with the solution, suspension or slurry of the binder by any suitable means. Spraying is a particularly preferred means to apply the additional binder material to the outer surface of the granules.

15 The amount of binder material applied to the granules in this step will depend upon such factors as the amount of binder present in the initial mixture used to prepare the granules and the final amount of binder to be present in the finished granules. The portion of the total binder content of the granules that is applied in this step may be from 5 to 75%, preferably from 10 to 60%, more preferably from 20 to
20 50% by weight.

 As an alternative to including a dispersant in the mixture used to form the granules or in addition thereto, the granules may have additional dispersant applied to their outer surface. The additional dispersant may be applied together with the
25 binder or as a separate step. Suitable dispersant materials are as described above. The dispersant applied to the granules in this step may be the same or different to any dispersant present in the mixture used to form the granules. The dispersant may be applied to the granules by any suitable means. Preferably, the dispersant is formed as a solution, suspension or slurry of the dispersant material in a suitable
30 solvent. The solvent is most preferably water. The granules may be contacted with the solution, suspension or slurry of the dispersant by any suitable means. Spraying is a particularly preferred means to apply the additional dispersant material to the outer surface of the granules.

The amount of dispersant material applied to the granules in this step will depend upon such factors as the amount of dispersant present in the initial mixture used to prepare the granules and the final amount of dispersant to be present in the finished granules. The portion of the total dispersant content of the granules that is applied in this step may be from 0 to 100%, more particularly from 5 to 75%, preferably from 10 to 60%, more preferably from 20 to 50% by weight.

After coating with additional binder and any dispersant, if applied, the resulting granules are dried, as required, in order to remove any solvent present in the granules to a suitable level. Suitable techniques for drying the granules and drying apparatus are known in the art, with suitable drying equipment being commercially available. The granules may be dried to remove the solvent to a suitable amount, in particular to provide the finished granules with a solvent content of from 0.5 to 15% by weight, more preferably from 1 to 10% by weight.

As noted above, the granules prepared by the method of the present invention most preferably comprise a dispersant. As also noted above, the dispersant may be included in the initial mixture used to prepare the granules. However, it has been found that it is particularly advantageous if the dispersant is added both to the initial mixture and to the granules, once they have been formed.

Accordingly, in a further aspect, the present invention provides a method for preparing a formulation comprising clomazone, the method comprising the steps of: combining microcapsules having a polymer shell and a core comprising clomazone, a solid carrier and a dispersant to form a mixture; forming granules from the resulting mixture; applying a composition comprising a dispersant to coat the granules; and drying the thus coated granules.

The method of this aspect of the present invention provides an improved granular formulation of clomazone, that is particularly stable and more effective in delivering the active ingredient to the target plants once the formulation is applied at a locus, with minimal drift of the active ingredient to plants outside the locus of application.

The method comprises forming a mixture of microencapsulated clomazone, as described in more detail hereinafter, a solid carrier and a dispersant. The solid carrier may be any suitable material that is susceptible to forming granules, in particular by extrusion, as hereinbefore described.

The solid carrier may be present in any suitable amount, in particular forming from 20 to 98% by weight of the final product, preferably from 40 to 95% by weight, more preferably from 50 to 90% by weight of the finished granules.

The mixture further comprises a dispersant. The dispersant is provided to increase the rate at which the granules disintegrate once they are delivered to the locus being treated and contacted with water, to release the active ingredient. The dispersant may comprise an anionic dispersant material, a non-ionic dispersant material, or a mixture of the two. Suitable dispersants for inclusion in the granules are known in the art and are as described above.

The mixture contains any suitable amount of the dispersant to ensure sufficient disintegration of the granules upon application, in particular from 0.5 to 20% by weight of the finished granules, preferably from 1 to 15% by weight, more preferably from 2 to 10% by weight.

The mixture to be extruded may consist of microencapsulated clomazone, one or more solid carriers, and one or more dispersants. However, the mixture may comprise further components. In particular, the mixture to be extruded may comprise one or more binders. The binder serves to adhere or bind the solid particles present in the final product and provides integrity to the granules, preventing them from crumbling and the like. The binder may be any suitable material and suitable binder materials are as described above.

If present, the amount of binder in the mixture is sufficient to provide the extruded granules with integrity and will vary according to the properties of the binder being used. Typically, the binder is present in the final granular product in an amount

of from 0.2 to 15% by weight, preferably from 0.5 to 10% by weight, more preferably from 1 to 5% by weight.

5 Depending upon the composition and properties of the other components of the mixture to be formed into granules and the method of granulation, the mixture may further comprise a solvent. Suitable solvents are as described above.

10 The solvent should be present in an appropriate amount to allow the mixture to be formed into granules, for example to be extruded, in particular from 1 to 20% by weight of the mixture, preferably from 2 to 10% by weight, more preferably from 2 to 5% by weight.

The components of the mixture may be combined in any order and mixed. Suitable techniques for mixing the components and apparatus for performing the mixing are known in the art, with suitable equipment being commercially available. In addition, or alternatively, the components may be mixed as a result of the action of the process for forming the mixture into granules. In one embodiment, the mixture is formed into granules by extrusion, as discussed below, in which case the components may be added directly to the inlet or feed hopper of the extruder, with the mixing taking place during the extrusion process.

25 In the following step of the method of this aspect of the invention, the mixture described above is formed into granules. The granules may be formed by any suitable technique. Suitable techniques are known in the art and include granulation, compaction, spray drying, fluid bed agglomeration and extrusion. Extrusion is a particular preferred manner of forming the mixture into granules. Suitable extrusion techniques and apparatus are well known in the art, with the equipment being commercially available.

30 Once the granules are formed, they are treated to apply additional dispersant to the exterior of the granules. The dispersant may comprise the same or different materials to the dispersant included in the initial mixture. Suitable dispersant materials are as described above. The dispersant may be applied to the granules by any suitable means. Preferably, the dispersant is provided as a solution, suspension

or slurry of the dispersant material in a suitable solvent. The solvent is most preferably water. The granules may be contacted with the solution, suspension or slurry of the dispersant by any suitable means. Spraying is a particularly preferred means to apply the additional dispersant material to the outer surface of the granules.

5

The amount of dispersant material applied to the granules in this step will depend upon such factors as the amount of dispersant present in the initial mixture used to prepare the granules and the final amount of dispersant to be present in the finished granules. The portion of the total dispersant content of the granules that is applied in this step may be from 5 to 75%, preferably from 10 to 60%, more preferably from 20 to 50% by weight.

10

As an alternative to including a binder in the mixture used to form the granules or in addition thereto, the granules may have additional binder applied to their outer surface. The additional binder may be applied together with the dispersant or as a separate step. Suitable binder materials are as described above. The binder applied to the granules in this step may be the same or different to any binder present in the mixture used to form the granules. The binder may be applied to the granules by any suitable means. Preferably, the binder is formed as a solution, suspension or slurry of the binder material in a suitable solvent. The solvent is most preferably water. The granules may be contacted with the solution, suspension or slurry of the binder by any suitable means. Spraying is a particularly preferred means to apply the additional binder material to the outer surface of the granules.

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The amount of binder material applied to the granules in this step will depend upon such factors as the amount of binder present in the initial mixture used to prepare the granules and the final amount of binder to be present in the finished granules. The portion of the total binder content of the granules that is applied in this step may be from 0 to 100%, more particularly from 5 to 75%, preferably from 10 to 60%, more preferably from 20 to 50% by weight.

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After coating with additional dispersant, and optionally additional binder material, the resulting granules are dried, as required, in order to remove any solvent present in the granules to a suitable level. Suitable techniques for drying the

granules and drying apparatus are known in the art, with suitable drying equipment being commercially available. The granules may be dried to remove the solvent to a suitable amount, in particular to provide the finished granules with a solvent content of from 0.5 to 15% by weight, more preferably from 1 to 10% by weight.

5

As noted above, it can be advantageous to provide both binder and dispersant materials to the granules in two stages, that is first including the materials in the mixture prior to forming the granules and second applying additional material to the outer surface of the granules.

10

Accordingly, in a still further aspect, the present invention provides a method for preparing a formulation comprising clomazone, the method comprising the steps of:

- combining microcapsules having a polymer shell and a core comprising
- 15 clomazone, a solid carrier, a binder and a dispersant to form a mixture;
- forming granules from the resulting mixture;
- applying a composition comprising a dispersant to coat the granules;
- applying a composition comprising a binder to coat the granules; and
- drying the thus coated granules.

20

The method of this aspect of the present invention provides an improved granular formulation of clomazone, that is particularly stable, resistant to abrasion and formation of dust and fines, and more effective in delivering the active ingredient to the target plants once the formulation is applied at a locus, with minimal drift of the

25 active ingredient to plants outside the locus of application.

25

Details of the constituent components of the granules and the manner of preparing the granules are as hereinbefore described. As noted, the granules, once prepared are provided with both additional binder material and additional dispersant

30 material on their outer surface. The additional binder and dispersant may be applied in a single stage or in separate treatments of the granules.

30

The granules prepared by the methods of the present invention comprise clomazone as an active ingredient, in particular clomazone in a microencapsulated

form. Formulations comprising microencapsulated active agrochemical components, in particular clomazone, are known in the art and are commercially available. The microencapsulated clomazone comprises microcapsules having a polymer shell and a core, the core containing clomazone, optionally with one or more other components, such as a solvent and other adjuvants or excipients typically employed in agrochemical formulations. The polymer shell may be of any suitable polymer material and the microcapsules formed by any suitable method. Preferably, the polymer shell is formed using an interfacial polymerisation technique. Examples of suitable polymers include polyamides, polyesters, polyurethanes and polyureas. The polymer is preferably a polyurea, in particular a polyurea formed from the reaction of one or more isocyanate compounds and one or more amine compounds. Such reactions are known in the art and the reactants known and commercially available.

Examples of microencapsulation suitable for use in preparing the clomazone for inclusion in the granules of the present invention are disclosed in EP 0 719 087, EP 0 792 100, EP 0 854 675, EP 1 104 991, EP 1 164 849, EP 1 404 176, EP 1 928 593, EP 2 133 140, WO 01/24631, WO 03/099005, WO 96/22159, WO 2007/072046, WO 2007/101019, WO 2009/00545, and WO 2009/086914.

The granules prepared by the methods of the present invention may comprise further components, in addition to those discussed above. Examples of such components include those components commonly included in agrochemical formulations, in particular granular agrochemical formulations, and include one or more components selected from biocides, adjuvants, and activity modifiers, including safeners and synergising agents of the active ingredients. Such components are commercially available and their use is known in the art. The additional components may be included in the granules at any convenient stage in the method of preparation. In particular, the additional components mentioned above may be included in the initial mixture and/or applied to the granules after their formation in a single or separate treatments, as hereinbefore described.

As noted, clomazone is an active ingredient in the formulations prepared by the methods of the present invention. The finished granules may contain clomazone as the sole active component. Alternatively, the finished granules may contain one

or more additional pesticidally active components, including one or more fungicides, insecticides and/or herbicides. The additional active ingredients, if present, may be included in the initial mixture used to prepare the granules and/or applied to granules after preparation. The additional active ingredients may be present within the

5 microcapsules, together with clomazone, and/or may be present in the bulk or continuous phase of the granules.

The present invention also provides a granule composition comprising an agrochemically active component, in particular clomazone, obtainable by a method

10 as hereinbefore described.

In addition, the present invention provides the use of granules prepared by a method of the present invention in the treatment of unwanted plant growth.

15 Further, the present invention provides a method of treating plant growth at a locus, the method comprising applying to the locus a granule prepared by a method of the present invention.

The present invention will be further illustrated by way of the following

20 examples. Percentages indicated in the examples are all on the basis of the weight of the finished granules, unless otherwise stated.

EXAMPLE 1

25 Granules of Formulation A have the following composition:

	Aluminium Silicate (Carrier)	82%
	Microencapsulated clomazone (Active Ingredient)	14%
	Sulphonated lignin (Binder)	1.5%
30	Sodium naphthalene sulphonate (Dispersant)	2%
	Water	0.5%

The microencapsulated clomazone is a formulation containing microcapsules having a polyurea shell prepared by the interfacial condensation polymerisation of a

polyisocyanate with a polyamine and containing 25% by weight of clomazone, 7% by weight polymer and the remainder an inert organic solvent.

The method of preparation of the granules of Formulation A is as follows:

5

The aluminium silicate, microencapsulated clomazone and 50% of the binder material are combined to form a mixture. Sufficient water is included in the mixture to allow the mixture to be extruded. The mixture is fed to the feed hopper of a screw extruder and extruded through a plate to form granules having a nominal size of 3 mm. An aqueous suspension of the remaining binder material and the dispersant is applied to the granules by spraying, to evenly coat the outer surface of the granules. Thereafter, the resulting granules are dried in an oven to remove excess water.

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The granules have a high resistance to abrasion and low formation of dust and fines. When applied to plants at a locus, the granules disperse clomazone upon contact with water with minimal drift of clomazone out of the target locus.

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EXAMPLE 2

Granules of Formulation B have the following composition:

25	Aluminium Silicate (Carrier)	81%
	Microencapsulated clomazone (Active Ingredient)	14%
	Sulphonated lignin (Binder)	1.5%
	Sodium naphthalene sulphonate (Dispersant)	2%
	Water	1.5%

30

The microencapsulated clomazone is a formulation containing microcapsules having a polyurea shell prepared by the interfacial condensation polymerisation of a polyisocyanate with a polyamine and containing 25% by weight of clomazone, 7% by weight polymer and the remainder an inert organic solvent.

The method of preparation of the granules of Formulation B is as follows:

5 The aluminium silicate, microencapsulated clomazone and 50% of the dispersant material are combined to form a mixture. Sufficient water is included in the mixture to allow the mixture to be extruded. The mixture is fed to the feed hopper of a screw extruder and extruded through a plate to form granules having a nominal size of 3 mm. An aqueous suspension of the remaining dispersant material and the binder is applied to the granules by spraying, to evenly coat the outer surface of the granules. Thereafter, the resulting granules are dried in an oven to remove excess
10 water.

The granules have a high resistance to abrasion and low formation of dust and fines. When applied to plants at a locus, the granules disperse clomazone upon contact with water with minimal drift of clomazone out of the target locus.
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EXAMPLE 3

Granules of Formulation C have the following composition:
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Aluminium Silicate (Carrier)	79.5%
Microencapsulated clomazone (Active Ingredient)	16%
Sulphonated lignin (Binder)	2%
Sodium naphthalene sulphonate (Dispersant)	2%
25 Water	0.5%

The microencapsulated clomazone is a formulation containing microcapsules having a polyurea shell prepared by the interfacial condensation polymerisation of a polyisocyanate with a polyamine and containing 25% by weight of clomazone, 7% by
30 weight polymer and the remainder an inert organic solvent.

The method of preparation of the granules of Formulation C is as follows:

The aluminium silicate, microencapsulated clomazone, 50% of the binder material and 50% of the dispersant material are combined to form a mixture. Sufficient water is included in the mixture to allow the mixture to be extruded. The mixture is fed to the feed hopper of a screw extruder and extruded through a plate to form granules having a nominal size of 3 mm. An aqueous suspension of the remaining binder material and the remaining dispersant is applied to the granules by spraying, to evenly coat the outer surface of the granules. Thereafter, the resulting granules are dried in an oven to remove excess water.

The granules have a high resistance to abrasion and low formation of dust and fines. When applied to plants at a locus, the granules disperse clomazone upon contact with water with minimal drift of clomazone out of the target locus.

CLAIMS

1. A method for preparing a formulation comprising clomazone, the method
5 comprising the steps of:
 - (i) combining microcapsules having a polymer shell and a core comprising clomazone, a solid carrier and a binder to form a mixture;
 - (ii) forming granules from the resulting mixture;
 - (iii) applying a composition comprising a binder to coat the granules; and
10 (iv) drying the thus coated granules.
2. The method according to claim 1, wherein the carrier comprises a metal salt.
3. The method according to claim 2, wherein the metal salt is a salt of a metal
15 from Groups IA, IIA or IIIA of the Periodic Table of the Elements.
4. The method according to claim 3, wherein the metal salt is a salt of calcium, magnesium or aluminium.
- 20 5. The method according to any of claims 2 to 4, wherein the metal salt is an oxide, carbonate, sulphate, or silicate.
6. The method according to any preceding claim, wherein the carrier is a clay.
- 25 7. The method according to any preceding claim, wherein the carrier is provided in step (i) in an amount to form from 20 to 98% by weight of the finished granules.
8. The method according to any preceding claim, wherein the binder comprises a material selected from flours, starches, gums, alcohols, lipids, cellulose derivatives,
30 alginates, wood extracts, lignin and lignin derivates.
9. The method according to claim 8, wherein the binder is a liginosulfonate.

10. The method according to any preceding claim, wherein the binder is present in an amount of from 0.2 to 15% by weight of the finished granules.
11. The method according to any preceding claim, wherein the mixture formed in
5 step (i) further comprises a solvent.
12. The method according to claim 11, wherein the solvent is water.
13. The method according to any preceding claim, wherein the mixture formed in
10 step (i) further comprises a dispersant.
14. The method according to claim 13, wherein the dispersant is selected from sulphonated and unsulphonated lignin polymers, sulphonated naphthalene compounds and polyacrylate salts.
15
15. The method according to claim 14, wherein the dispersant is a sulfonated lignin or naphthalene sulphonate.
16. The method according to any preceding claim, wherein the granules are
20 formed in step (ii) by a procedure comprising granulation, compaction, spray drying, fluid bed agglomeration or extrusion.
17. The method according to claim 16, wherein the granules are formed by extrusion of the mixture.
25
18. The method according to any preceding claim, wherein the granules are treated in step (iii) with a solution, slurry or suspension of the binder material in a solvent.
- 30 19. The method according to claim 18, wherein the solvent is water.
20. The method according to any preceding claim, wherein the binder applied to the granules in step (iii) is the same as the binder included in the mixture in step (i).

21. The method according to any preceding claim, wherein from 5 to 75% of the total binder content of the finished granules is applied in step (iii).

22. The method according to any preceding claim, wherein a dispersant is applied to the granules in step (iii).

23. The method according to claim 22, wherein a dispersant is provided in the mixture of step (i) and the dispersant applied in step (iii) is the same as the dispersant provided in step (i).

24. The method according to either of claims 22 or 23, wherein from 5 to 75% by weight of the dispersant present in the finished granules is provided in step (iii).

25. The method according to any preceding claim, wherein the finished granules comprise from 0.5 to 20% by weight of dispersant.

26. The method according to any preceding claim, wherein the granules are dried in step (iv) to provide the finished granules with a solvent content of from 0.5 to 15% by weight.

27. A method for preparing a formulation comprising clomazone, the method comprising the steps of:

(i) combining microcapsules having a polymer shell and a core comprising clomazone, a solid carrier and a dispersant to form a mixture;

(ii) forming granules from the resulting mixture;

(iii) applying a composition comprising a dispersant to coat the granules; and

(iv) drying the thus coated granules.

28. The method according to claim 27, wherein the carrier comprises a metal salt.

29. The method according to claim 28, wherein the metal salt is a salt of a metal from Groups IA, IIA or IIIA of the Periodic Table of the Elements.

30. The method according to claim 29, wherein the metal salt is a salt of calcium, magnesium or aluminium.

31. The method according to any of claims 28 to 30, wherein the metal salt is an
5 oxide, carbonate, sulphate, or silicate.

32. The method according to any of claims 27 to 31, wherein the carrier is a clay.

33. The method according to any of claims 27 to 32, wherein the carrier is
10 provided in step (i) in an amount to form from 20 to 98% by weight of the finished granules.

34. The method according to any of claims 27 to 33, wherein the dispersant is
15 selected from sulphonated and unsulphonated lignin polymers, sulphonated naphthalene compounds and polyacrylate salts.

35. The method according to claim 34, wherein the dispersant is a sulfonated lignin or naphthalene sulphonate.

20 36. The method according to any of claims 27 to 35, wherein the finished granules comprise from 0.5 to 20% by weight of dispersant.

37. The method according to any of claims 27 to 36, wherein the mixture formed
25 in step (i) further comprises a solvent.

38. The method according to claim 37, wherein the solvent is water.

39. The method according to any of claims 27 to 38, wherein the mixture formed
30 in step (i) further comprises a binder.

40. The method according to claim 39, wherein the binder comprises a material selected from flours, starches, gums, alcohols, lipids, cellulose derivatives, alginates, wood extracts, lignin and lignin derivates.

41. The method according to claim 40, wherein the binder is a lignosulfonate.
42. The method according to any of claims 27 to 41, wherein the granules are formed in step (ii) by a procedure comprising granulation, compaction, spray drying,
5 fluid bed agglomeration or extrusion.
43. The method according to claim 42, wherein the granules are formed by extrusion of the mixture.
- 10 44. The method according to any of claims 27 to 43, wherein the granules are treated in step (iii) with a solution, slurry or suspension of the dispersant material in a solvent.
45. The method according to claim 44, wherein the solvent is water.
15
46. The method according to any of claims 27 to 45, wherein the dispersant applied to the granules in step (iii) is the same as the dispersant included in the mixture in step (i).
- 20 47. The method according to any of claims 27 to 46, wherein from 5 to 75% of the total dispersant content of the finished granules is applied in step (iii).
48. The method according to any of claims 27 to 48, wherein a binder is applied to the granules in step (iii).
25
49. The method according to claim 48, wherein a binder is provided in the mixture of step (i) and the binder applied in step (iii) is the same as the binder provided in step (i).
- 30 50. The method according to either of claims 48 or 49, wherein from 5 to 75% by weight of the binder present in the finished granules is provided in step (iii).
51. The method according to any of claims 27 to 50, wherein the binder is present in the finished granules in an amount of from 0.2 to 15% by weight.

52. The method according to any of claims 27 to 51, wherein the granules are dried in step (iv) to provide the finished granules with a solvent content of from 0.5 to 15% by weight.

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53. A method for preparing a formulation comprising clomazone, the method comprising the steps of:

(i) combining microcapsules having a polymer shell and a core comprising clomazone, a solid carrier, a binder and a dispersant to form a mixture;

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(ii) forming granules from the resulting mixture;

(iii) applying a composition comprising a dispersant to coat the granules;

(iv) applying a composition comprising a binder to coat the granules; and

(v) drying the thus coated granules.

15

54. The method according to claim 53, wherein the carrier comprises a metal salt.

55. The method according to claim 54, wherein the metal salt is a salt of a metal from Groups IA, IIA or IIIA of the Periodic Table of the Elements.

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56. The method according to claim 55, wherein the metal salt is a salt of calcium, magnesium or aluminium.

57. The method according to any of claims 54 to 56, wherein the metal salt is an oxide, carbonate, sulphate, or silicate.

25

58. The method according to any of claims 53 to 57, wherein the carrier is a clay.

59. The method according to any of claims 53 to 58, wherein the carrier is provided in step (i) in an amount to form from 20 to 98% by weight of the finished granules.

30

60. The method according to any of claims 53 to 59, wherein the dispersant is selected from sulphonated and unsulphonated lignin polymers, sulphonated naphthalene compounds and polyacrylate salts.

61. The method according to claim 60, wherein the dispersant is a sulfonated lignin or naphthalene sulphonate.
- 5 62. The method according to any of claims 53 to 61, wherein the finished granules comprise from 0.5 to 20% by weight of dispersant.
63. The method according to any of claims 53 to 62, wherein the binder comprises a material selected from flours, starches, gums, alcohols, lipids, cellulose
10 derivatives, alginates, wood extracts, lignin and lignin derivates.
64. The method according to claim 63, wherein the binder is a liginosulfonate.
65. The method according to any of claims 53 to 64, wherein the binder is present
15 in the finished granules in an amount of from 0.2 to 15% by weight.
66. The method according to any of claims 53 to 65, wherein the mixture formed in step (i) further comprises a solvent.
- 20 67. The method according to claim 66, wherein the solvent is water.
68. The method according to any of claims 53 to 67, wherein the granules are formed in step (ii) by a procedure comprising granulation, compaction, spray drying, fluid bed agglomeration or extrusion.
25
69. The method according to claim 68, wherein the granules are formed by extrusion of the mixture.
70. The method according to any of claims 53 to 69, wherein the granules are
30 treated in step (iii) with a solution, slurry or suspension comprising the dispersant material in a solvent.
71. The method according to claim 70, wherein the solvent is water.

72. The method according to any of claims 53 to 71, wherein the dispersant applied to the granules in step (iii) is the same as the dispersant included in the mixture in step (i).

5 73. The method according to any of claims 53 to 72, wherein from 5 to 75% of the total dispersant content of the finished granules is applied in step (iii).

74. The method according to any of claims 53 to 73, wherein the binder applied to the granules in step (iv) is the same as the binder included in the mixture in step (i).

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75. The method according to any of claims 53 to 74, wherein from 5 to 75% by weight of the binder present in the finished granules is provided in step (iv).

76. The method according to any of claims 53 to 75, wherein steps (iii) and (iv) are combined to apply both the dispersant and the binder to the granules at the same time.

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77. The method according to any of claims 53 to 76, wherein the granules are dried in step (v) to provide the finished granules with a solvent content of from 0.5 to 15% by weight.

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78. A granular formulation comprising clomazone obtainable by the method of any preceding claim.

25 79. The use of a granular formulation prepared by the method of any of claims 1 to 77 in the treatment of unwanted plant growth.

80. A method for controlling plant growth at a locus, the method comprising applying to the locus granules prepared by a method according to any of claims 1 to

30

77.

INTERNATIONAL SEARCH REPORT

International application No.

PCT/CN2011/071366

A. CLASSIFICATION OF SUBJECT MATTER

See extra sheet

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC: A01N25; A01N43; A01P13

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

WPI; EPODOC; CNPAT; CNKI

granule?, granular, microcapsul+, micro w encapsulat+, micro w capsul+, encapsulat+, clomazone, isoxazolidinone, agricultural w chemical?, herbicide?

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 6797277 B2 (WILBUR-ELLIS COMPANY), 28 Sep. 2004 (28.09.2004), see column 2, line 50-column 6, line 6, examples 1-12 and claim 7	1-80
A	WO 2009135492 A2 (CHEMINOVA AS), 12 Nov.2009 (12.11.2009), see the whole document	1-80
A	US 4936901A(MONSANTO CO), 26 Jun.1990 (26.06.1990), see the whole document	1-80
A	WO 9938611 A1(RHODIA CHIM), 05 Aug.1999(05.08.1999), see the whole document	1-80
A	CN 1343092 A (FMC CORP), 03 Apr.2002 (03.04.2002), see the whole document	1-80

☐ Further documents are listed in the continuation of Box C.

☒ See patent family annex.

* Special categories of cited documents:	“T” later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
“A” document defining the general state of the art which is not considered to be of particular relevance	“X” document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
“E” earlier application or patent but published on or after the international filing date	“Y” document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art
“L” document which may throw doubts on priority claim (S) or which is cited to establish the publication date of another citation or other special reason (as specified)	“&”document member of the same patent family
“O” document referring to an oral disclosure, use, exhibition or other means	
“P” document published prior to the international filing date but later than the priority date claimed	

Date of the actual completion of the international search 06 May 2011(06.05.2011)	Date of mailing of the international search report 26 May 2011 (26.05.2011)
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Name and mailing address of the ISA/CN The State Intellectual Property Office, the P.R.China 6 Xitucheng Rd., Jimen Bridge, Haidian District, Beijing, China 100088 Facsimile No. 86-10-62019451	Authorized officer XU, Li Telephone No. (86-10)62084406
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Information on patent family members

International application No.
PCT/CN2011/071366

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INTERNATIONAL SEARCH REPORT

International application No.

PCT/CN2011/071366

Continuation of: A. CLASSIFICATION OF SUBJECT MATTER

A01N 25/28 (2006.01) i

A01N 43/80 (2006.01) i

A01P 13/00 (2006.01) n