(54) METHOD FOR PRODUCING AGROCHEMICAL GRANULE

VERFAHREN ZUR HERSTELLUNG EINES AGROCHEMISCHEN GRANULATS

PROCÉDÉ DE PRODUCTION DE GRANULÉS AGROCHIMIQUES

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(56) References cited:
• DATABASE WPI Week 200406 Thomson
  Scientific, London, GB; AN 2004-055884
The present invention relates to a method for producing agrochemical granules. More specifically, the present invention relates to a method for producing agrochemical granules having stable quality and which contain an agrochemical active ingredient, one or more kinds of hydrophobic substances, and a substance capable of absorbing oil.


As methods for producing agrochemical granules, the following methods are known. For example, PTL 1 discloses a production method in which a sustained-release agrochemical composition containing an agrochemical active component, a hydrophobic substance having a boiling point of 50°C or higher, a substance capable of absorbing oil, and a hydrophilic substance having a boiling point of 50°C or higher are melted, and the resultant is granulated by extrusion under a condition of heating performed at a temperature equal to or higher than the melting point. Specifically, PTL 1 discloses a method in which 20 g of acetamiprid as an active substance, 620 g of precipitated calcium carbonate as a carrier, 20 g of polyvinyl alcohol as a release control agent, and 40 g of white carbon capable of absorbing oil are uniformly mixed together, 300 g of molten paraffin wax (melting point of about 70°C) is added thereto, the mixture is kneaded with a kneader while being kept at a product temperature of 85°C, the kneaded material is granulated by being extruded through a screen, which is heated at 85°C and has 1 mm openings, then ground with a disintegrator, and then the resultant is sieved and classified into granules of 0.8 mm to 2 mm in size.

PTL 2 discloses a method for producing agrochemical granules composed of an agrochemical active ingredient, a thermoplastic material, and an inorganic diluting carrier, through (i) a mixing step, (ii) a kneading step, and (iii) an extruding step. In this method, extruding granulation is performed at a temperature that is equal to or higher than a freezing point and lower than a melting point of a hydrophobic substance having a high melting point. Specifically, PTL 2 discloses a method in which 2% by weight of acetamiprid as an agrochemical active ingredient, 15% by weight of carnauba wax (melting point of 83°C and a freezing point of 73°C to 74°C) and 5% by weight of paraffin wax (melting point of 70°C) as hydrophobic substances, 5% by weight of white carbon as a substance capable of absorbing oil, 2% by weight of polyvinyl alcohol as a water-soluble substance, and 10% by weight of talc and 61% by weight of calcium carbonate as a carrier are loaded into a Henschel mixer and mixed together, the mixture is discharged as a granular material at 80°C, the granular material is loaded into a screw extrusion granulator (EXR-130) manufactured by Fuji Paudal Co., Ltd., kneaded at 80°C, and granulated at the same temperature by being extruded through a die having 0.8 mm openings, and the granules are further ground with a disintegrator, thereby obtaining agrochemical granules.

Patent Literature

- PTL 1: PCT International Publication No. WO95/09532

JP 2003 171207 A discloses a method to produce agrochemical granules by mix-heating active ingredients, thermoplastic material and carrier, cooling, and granulating under predefined conditions.

JP 8 092007 A concerns a method for producing an agricultural chemical raw powder composition by heating and mixing raw materials.

Problems to be Solved by the Invention

However, in view of stably producing granules having excellent quality, the above production methods
The present invention aims to provide a method for producing agrochemical granules containing an agrochemical active ingredient, one or more kinds of hydrophobic substances, and a substance capable of absorbing oil, with stable quality. Means for Solving the Problems

[0011] The present inventors conducted thorough research regarding the reason why the quality is not stable in the above methods. As a result, they found that it is more important to regulate the temperature in the kneading step than to regulate the temperature in the extruding granulation step, and granules having excellent quality can be more stably produced if a temperature profile of the kneading step is controlled within a certain range. The present invention has been completed based on this knowledge.

[0012] That is, the present invention includes the following.

[1] A method for producing agrochemical granules, comprising: (i) a step of obtaining a kneaded material by loading an agrochemical active ingredient, one or more kinds of hydrophobic substances, and a substance capable of absorbing oil into a kneading device, kneading them at a heating temperature of 8°C higher than the highest melting point of the hydrophobic substances and equal to or lower than 130°C, and then causing the material to be discharged from the kneading device at a temperature of 4°C or lower than the highest melting point of the hydrophobic substances and equal to or higher than 60°C; and (iii) a step of granulating the obtained kneaded material by an extrusion molding method, wherein a heating temperature in a section close to the opening for discharging kneaded material of the kneading device is 30°C to 70°C; and steps (ii) and (iii) are performed consecutively.

[2] A method for producing agrochemical granules, comprising: (i) a step of obtaining a mixture by mixing an agrochemical active ingredient, one or more kinds of hydrophobic substances, and a substance capable of absorbing oil together, (ii) a step of obtaining a kneaded material by loading the obtained mixture into a kneading device, kneading the mixture at a heating temperature which is 8°C higher than the highest melting point of the hydrophobic substances and equal to or lower than 130°C, and then causing the kneaded material to be discharged from the kneading device at a temperature of 4°C or more lower than the highest melting point of the hydrophobic substances and equal to or higher than 60°C; and (iii) a step of granulating the obtained kneaded material by an extrusion molding method, wherein a heating temperature in a section close to the opening for discharging kneaded material of the kneading device is 30°C to 70°C and steps (ii) and (iii) are performed consecutively.

Effects of the Invention

According to the method for producing agrochemical granules of the present invention, it is possible to stably produce agrochemical granules containing an agrochemical active ingredient, one or more kinds of hydrophobic substances, and a substance capable of absorbing oil, with high quality.
BEST MODE FOR CARRYING OUT THE INVENTION

[0014] The method for producing agrochemical granules of the present invention includes (i) a step of obtaining a kneaded material by loading an agrochemical active ingredient, one or more kinds of hydrophobic substances, and a substance capable of absorbing oil into a kneading device, kneading them at a heating temperature of 8°C higher than the highest melting point of the hydrophobic substances and equal to or lower than 130°C, and then causing the kneaded material to be discharged from the kneading device at a temperature of 4°C or more lower than the highest melting point of the hydrophobic substances and equal to or higher than 60°C, and (ii) a step of granulating the obtained kneaded material by an extrusion molding method.

[0015] In the present invention, the term "melting point that is the highest of the hydrophobic substances" refers to a melting point of a hydrophobic substance having the highest melting point among one or more kinds of the hydrophobic substances used in the method for producing agrochemical granules of the present invention.

[0016] The agrochemical active ingredient used in the present invention is not particularly limited as long as it can be formulated into granules by kneading and extruding granulation. In addition, the agrochemical active ingredient is preferably a substance having solubility in water of 60 ppm or greater. If an agrochemical active ingredient showing high solubility in water is used, the agrochemical active ingredient is rapidly released from the granules.

[0017] Specific examples of the agrochemical active ingredient include the following germicides, herbicides, insecticides and miticides, plant growth regulators, and the like.

<Germicides>


<Herbicides>

[0019] 2,4-D, MCPA, clometho, chlorotoluron, diuron, linuron, isouuron, fenuron, neburon, simazine, atrazine, simetryn, prometryn, hexazinone, propazine, desmetryne, terbuturon, propanil, bromoxynil, ioxynil, pyridate, chloridazon, bentazon, chlormethoxyfen, bifenox, sodium acifluorfen, flumioxazin, thiaziazine, oxadiazon, sulfentrazone, pentozaxone, pyraclonil, pyrazolinate, pyrazoxyfen, benzofenap, mesotione, isoxaflutole, isoachloroxide, amitrole, acolinfen, difufenaf, benzobicyclon, diclofop-methyl, fluazifop-butyl, alloxynid sodium, clethodim, sethoxydim, tralkoxydim, tepraloxydim, bensulfonyl-methyl, pyrazosulfuron-ethyl, rimsulfuron, imazosulfuron, prosulfuron, fumetsulum, diclosulam, metosulfam, imazaquin, pyrithiobac-sodium, bispyribac-sodium, pyriminoxybenz-methyl, fucarbazone, propoxycarbazone, glyphosate, a glyphosate ammonium salt, glufosinate, trifluralin, pendimethalin, benfluralin, prodiame, propan, dithiopyr, alachlor, metolachlor, petoxiamid, acetochlor, propachlor, dimethenamid, diphenamid, napropamide, mfenacet, fentrizima, molinate, dimepiperate, cycloate, esprocarb, thiobencarb, thiocarbazil, bensulide, dalapon, asulam, DNOC, dinoseb, fluopoxam, triazifam, quinclorac, cinmethylin, daizomet, dymron, etobenzamide, oxaziclocomeone, pyributicarb, and the like.

<Insecticides and miticides>

[0020] Insecticides based on organic organophosphorus or carbamate: fenothion, fenitrothion, diazinon, chlorpyrifos, ESP, amidothion, phenothio, dimethioth, formothion, malathion, trichlorfon, thionetom, phosmet, dichlorvos, acephate, EPBP, methyl parathion, oxydemeton-methyl, ethion, salithion, cyanophos, isoxathion, pyridafenthion, phosalone, methidathion, sulprofos, chlorfenvinphos, tetrachlorvinphos, dimethylvinphos, propaphos, isofenphos, ethylthiometon, profenophos, pyraclonil, monocrotophos, azinphos-methyl, aldicarb, mesomil, thiocarb, carbofuran, carbosulfan, fenuron, carbaryl, fenthiocarbazol, benzthiaz, bensulide, dalapon, asulam, DNOC, dinoseb, fluopoxam, triazifam, quinclorac, cinmethylin, daizomet, dymron, etobenzamide, oxaziclocomeone, pyributicarb, and the like.

[0021] Pyrethroid-based insecticides: permethrin, cypermethrin, deltamethrin, fenvalerate, fenpropithrin, pyrethrin, allethrin, tetramethrin, resmethrin, dimethrin, propargitin, phenothrin, prothrin, fluvinate, cyfluthrin, cyhalothrin, flucytrinate, ethofenprox, cycloprothrin, phylloquinone, tecloflalam, phthalide, phenezine oxide, thiabendazole, tricyclazole, vinclozolin, cyroxilan, cyclobutanol, guazatine, propamocarb hydrochloride, oxolinic acid, cylufenamid, iminoctadine, kresoxim-methyl, triazine, fenhexamid, cyazofamid, cyprodinil, prothiocapazole, fenbuconazole, trifloxystrobin, azoxystrobin, hexaconazole, imibenconazole, tebuconazole, difenoconazole, carpropamid, and the like.
tralometrin, silicafluoron, acrinathrin, and the like

[0022] Benzoylurea-based insecticides and others: di-
flubenzuron, chlorfluanuron, hexafluuron, triflumuron,
flufenoxuron, flucycoxuron, buprofezin, pyriproxyfen,
methoprene, benzoezin, diafenthiuron, imiacloprid,
fipronil, nicotine sulfate, rotenone, metaldehyde, aceta-
miprid, chlorfenapyr, nitenpyram, thiacloprid, clothiani-
din, thiamethoxam, dinotefuron, indoxacarb, pymetro-
azine, spinosad, emamectin, pyridalyl, tebufenozide,
chromafenozide, methoxyfenozide, tolfenpyrad, and the like

Nematocides: fenamiphos, fosthiazate, cadusafos, and the like

[0023] Miticides: Chlorobenzilate, phenisobromolate,
dicofol, amitraz, BPPS, benzomate, hexathiazox, fenb-
uttar oxide, polynactin, renomethionate, CPCBS, tet-
radifen, avermectin, milbemectin, clofentezine, cyhexa-
tin, pyridaben, fenpyroximate, tebufenpyrad, pyrilmidfen,
fenothiocarb, dienochlor, fluacrypyrim, acequinocyl,
bifenazate, ethoxazol, spirodiclofen, fenazaquin, and the like

<Plant growth regulators>

[0024] Gibberellins (for example, gibberelin A3, gib-
berelin A4, and gibberelin A7), IAA, NAA, and the like

[0025] Among these, the agrochemical active ingredi-
ent is preferably at least one kind selected from the group
consisting of nitenpyram, imiacloprid, acetamiprid, thi-
amethoxam, clothianidin, thiacloprid, and dinotefuran.

[0026] The content of the agrochemical active ingredient
in the agrochemical granules is preferably 0.01 to
50% by weight and more preferably 0.01 to 20% by weight.

[0027] The hydrophobic substance preferably used in the present
invention is not particularly limited as long as it can be
formulated into granules by kneading and extruding gran-
ulation. The melting point of the hydrophobic substance
is preferably 50°C to 120°C and more preferably 50°C to
100°C. Examples of the hydrophobic substance include carnauba wax, shellac, bees wax, Japan wax, rice wax,
candelilla wax, fatty acids or hydrogenated products
thereof that are obtained by decomposing vegetable fat-
and-oil or animal fat-and-oil, stearic acid, behenic acid,
hydrogenated fatty acids of rapeseed, hydrogenated
palm fatty acids, hydrogenated fatty acids of beef tallow,
hydrogenated castor oil, paraffin wax, microcrystalline
wax, and montanolic acid ester wax, and the like. These
can be used alone, or two or more kinds of these can be
used in combination.

[0028] The content of the hydrophobic substance in
the agrochemical granules is preferably 15 to 80% by
weight and more preferably 18 to 70% by weight. More-
over, if a hydrophobic substance showing a high degree
of crystallinity is used, the agrochemical active ingredient
is slowly released from the granules.

[0029] The substance capable of absorbing oil that is
used in the present invention is a substance that can
absorb a molten hydrophobic substance and can be
turned into powder outwardly. Specific examples thereof
include starch, starch derivatives, celluloses, amorphous
silicon dioxide, and the like. One kind of these can be
used alone, or two or more kinds of these may be used
in combination.

[0030] The amorphous silicon dioxide can be produced
by a wet method and is called white carbon in general.
Examples of commercially available products of the
amorphous silicon dioxide include Carplex #67, Carplex
#80, Carplex CS-5, Carplex CS-7, (all manufactured by
Shionogi & Co., Ltd.) and the like.

[0031] Among the agrochemical granules, the content
of the substance capable of absorbing oil is preferably
0.05 to 30% by weight and more preferably 0.5 to 20%
by weight.

[0032] In the present invention, in addition to the agro-
chemical active ingredient, hydrophobic substance, and
substance capable of absorbing oil, additives that can be
contained in agrochemical granules may be used. Ex-
amples of the additives include a release control agent,
a binder, a bulking agent, a surfactant, a carrier, a color-
ant, and the like.

[0033] Examples of the release control agent include
water-soluble polymers, water-soluble inorganic sub-
stances, water-soluble organic substances, and the like.
Examples of the water-soluble polymers include alginic
acid, sodium alginate, xanthan gum, carrageenan, kar-
ya gum, hydroxypropyl cellulose, hydroxypropyl methyl-
cellulose, hydroxypropyl starch; polyvinyl alcohol, car-
boxyvinyl polymers, sodium polyacrylate, and the like.
Examples of the water-soluble inorganic or organic sub-
stances include sodium sulfate, sodium chloride, citric
acid or a salt thereof, adipic acid or a salt thereof, urea,
pregelatinized starch, lactose, and the like. The content
of the release control agent in the agrochemical granules
is preferably 0 to 50% by weight and more preferably
0.01 to 40% by weight.

[0034] Examples of the carrier or bulking agent include
clay, talc, calcium carbonate, and the like. The carrier or
bulking agent is used in an amount such that the total
amount of the agrochemical active ingredient, hydropho-
bic substance, substance capable of absorbing oil, and
release control agent plus the carrier or bulking agent
becomes 100% by weight.

(i) Mixing step

[0035] First, in the method for producing agrochemical
granules of the present invention, the agrochemical ac-
tive ingredient, one or more kinds of hydrophobic sub-
stances, the substance capable of absorbing oil, and oth-
er optional additives can be mixed with together. The
order of mixing the respective components, the mixing
method, and the like are not particularly limited. The ob-
tained mixture is preferably in a powder or granule state,
since the mixture is easy to handle and transferred easily to the next step in this state. In addition, the mixing may be performed in the kneading step described below.

(ii) Kneading step

[0036] In the kneading step, first, the agrochemical active ingredient, one or more kinds of hydrophobic substances, the substance capable of absorbing oil, and other optional additives are loaded in a kneading device. The order of loading the respective components into the kneading device is not particularly limited. The agrochemical active ingredient, one or more kinds of hydrophobic substances, the substance capable of absorbing oil, and other additives which are optionally added may be loaded into the kneading device after being made into a mixture by the above mixing step.

[0037] The temperature of the agrochemical active ingredient, one or more kinds of hydrophobic substances, the substance capable of absorbing oil, and other optional additives are heated and kneaded in the kneading device. In the present invention, the heating temperature in the kneading device needs to reach a temperature equal to or higher than the highest melting point of the hydrophobic substances, at least once. The lower limit of the highest heating temperature is preferably 4°C higher than the highest melting point of the hydrophobic substances loaded into the kneading device, more preferably 8°C higher than the melting point, and even more preferably 10°C higher than the melting point. The upper limit of the heating temperature is not particularly limited, as long as the agrochemical active ingredient, one or more kinds of hydrophobic substances, the substance capable of absorbing oil, and other optional additives are not thermally decomposed. However, the upper limit is 130°C, preferably 120°C, and more preferably 115°C.

[0038] Heating can be performed by, for example, causing a medium such as steam or hot water to flow in a jacket mounted on the kneading device, or by applying electricity to an electric heater mounted on the kneading device. In view of ease of regulating temperature, a high thermal conduction efficiency, and the like, heating by a jacket is preferable.

[0040] The kneading device is not particularly limited as long as it can perform kneading by applying heat, and examples thereof include single-screw kneading devices, double-screw kneading devices, roll kneaders, and the like. Among these, a continuous single-screw kneading device or a continuous double-screw kneading device is preferable, and a double-screw kneading device is more preferable in the respect that a temperature profile is easily controlled, and the kneading state is easily regulated. In a continuous kneading device, the portion from the opening for injecting raw material components to the opening for discharging the kneaded material can be divided into plural sections. Moreover, the respective sections can be controlled to have different temperatures and to be in different kneading states.

[0041] The kneading state can be designed by the combination of a paddle and a screw installed in the kneading device. Examples of the paddle include a paddle for kneading (flat paddle), a paddle having a function of feeding in addition to a function of kneading (helical paddle), a paddle having a function of backward feeding in addition to a function of kneading (backward helical paddle), and the like. When the paddle rotates, the kneaded material undergoes volumetric change by compression or stretching and is influenced by a shearing action between a trough and a paddle or between paddles.

[0042] Finally, the kneading material is discharged from the kneading device. The upper limit of the temperature (product temperature) at the time of discharging is 60°C, preferably 85°C, and more preferably 70°C.

[0043] By the heat applied from the outside or the heat caused by friction or the like, the product temperature of the kneaded material in the kneading device can slowly increase immediately after the material is loaded into the kneading device and reach a temperature equal to or higher than the highest melting point of the hydrophobic substances. In order to control the temperature (product temperature) at the time of discharging to fall within the above temperature range, in the section close to the opening for discharging kneaded material of the kneading device, it is preferable to regulate the product temperature of the kneaded material by causing hot water at low temperature or the like to flow in the jacket. The heating temperature in the section close to the opening for discharging kneaded material of the kneading device is 30°C to 70°C and more preferably 35°C to 60°C.

[0044] Moreover, if a kneading device equipped with a vent is used, unnecessary volatile components can be removed by evaporation in the kneading step.

(ii) Extruding granulation step

[0045] Subsequently, the obtained kneaded material is granulated by an extrusion molding method. The method according to the invention may be performed with an extrusion molding device having a kneading function, since the above kneading step and the extruding granulation step can be performed consecutively. The conditions of extrusion molding are not particularly limited. The upper limit of the temperature at the time of extruding the kneaded material is a temperature of 4°C or more lower
The obtained granulated material is cooled. Sometimes the cooled granulated material contains materials fused with or adhering to each other. In this case, a disintegrating step can be performed. The granulated material can be disintegrated using a known disintegrating device. Moreover, the granulated material can optionally be dried or classified. In the manner described above, granules fused with or adhering to each other practically were not observed.

The obtained granulated material is cooled. The obtained kneaded material (a product temperature of 75°C) was loaded into an extrusion granulator (Fine Riu User EXRC JS-100, manufactured by Dalton Co., Ltd.) and granulated by extrusion at an extrusion temperature of 73°C, and as a result, a granulated material having a uniform cylindrical shape was obtained. In the granulated material, granules fused with or adhering to each other practically were not observed.

Example 2

20 parts by weight of acetamiprid (Mospiran raw material [purity of 99.0% or higher], manufactured by NIPPON SODA CO., LTD.) as an agrochemical active ingredient, 605 parts by weight of sedimentary calcium carbonate, 100 parts by weight of talc (supplier: Neolight Co., Ltd.), 50 parts by weight of silicon dioxide (Carplex #80, manufactured by Shionogi & Co., Ltd.), 15 parts by weight of polyvinyl alcohol (Gohsenol GL-05S, manufactured by The Nippon Synthetic Chemical Industry Co., Ltd.), 57 parts by weight of ground paraffin wax (WAX 150, melting point of 65.5°C to 68.2°C, manufactured by NIPPON SEIRO CO., LTD.), and 153 parts by weight of ground carnauba wax (melting point of 83 ± 3°C) were loaded into a ribbon mixer and mixed together.

The obtained mixture (a product temperature of about 33°C) was loaded into a continuous double-screw kneading device (KRC kneader, manufactured by KURI-MOTO, LTD.) so as to be kneaded under heating, and discharged from the continuous double-screw kneading device at a product temperature of 75°C. The continuous double-screw kneading device was divided into two sections. The heating temperature of the section (first section) close to the opening for injecting mixture was set to 89°C by causing steam to flow in the jacket. The heating temperature of the section (second section) close to the opening for discharging kneaded material was set to 43°C by causing hot water to flow in the jacket.

Example 2

20 parts by weight of acetamiprid (Mospiran raw material [purity of 99.0% or higher], manufactured by NIPPON SODA CO., LTD.) as an agrochemical active ingredient, 605 parts by weight of sedimentary calcium carbonate, 100 parts by weight of talc (supplier: Neolight Co., Ltd.), 50 parts by weight of silicon dioxide (Carplex #80, manufactured by Shionogi & Co., Ltd.), 15 parts by weight of polyvinyl alcohol (Gohsenol GL-05S, manufactured by The Nippon Synthetic Chemical Industry Co., Ltd.), 57 parts by weight of ground paraffin wax (WAX 150, melting point of 65.5°C to 68.2°C, manufactured by NIPPON SEIRO CO., LTD.), and 153 parts by weight of ground carnauba wax (melting point of 83 ± 3°C) were loaded into a ribbon mixer and mixed together.

The obtained mixture (a product temperature of about 33°C) was loaded into a continuous double-screw kneading device (KRC kneader, manufactured by KURI-MOTO, LTD.) so as to be kneaded under heating, and discharged from the continuous double-screw kneading device at a product temperature of 75°C. The continuous double-screw kneading device was divided into two sections. The heating temperature of the section (first section) close to the opening for injecting mixture was set to 89°C by causing steam to flow in the jacket. The heating temperature of the section (second section) close to the opening for discharging kneaded material was set to 43°C by causing hot water to flow in the jacket.

The obtained mixture (a product temperature of about 33°C) was loaded into a continuous double-screw kneading device (KRC kneader, manufactured by KURI-MOTO, LTD.) so as to be kneaded under heating, and discharged from the continuous double-screw kneading device at a product temperature of 75°C. The continuous double-screw kneading device was divided into two sections. The heating temperature of the section (first section) close to the opening for injecting mixture was set to 89°C by causing steam to flow in the jacket. The heating temperature of the section (second section) close to the opening for discharging kneaded material was set to 43°C by causing hot water to flow in the jacket.

The obtained mixture (a product temperature of about 33°C) was loaded into a continuous double-screw kneading device (KRC kneader, manufactured by KURI-MOTO, LTD.) so as to be kneaded under heating, and discharged from the continuous double-screw kneading device at a product temperature of 75°C. The continuous double-screw kneading device was divided into two sections. The heating temperature of the section (first section) close to the opening for injecting mixture was set to 89°C by causing steam to flow in the jacket. The heating temperature of the section (second section) close to the opening for discharging kneaded material was set to 43°C by causing hot water to flow in the jacket.

The obtained mixture (a product temperature of about 33°C) was loaded into a continuous double-screw kneading device (KRC kneader, manufactured by KURI-MOTO, LTD.) so as to be kneaded under heating, and discharged from the continuous double-screw kneading device at a product temperature of 75°C. The continuous double-screw kneading device was divided into two sections. The heating temperature of the section (first section) close to the opening for injecting mixture was set to 89°C by causing steam to flow in the jacket. The heating temperature of the section (second section) close to the opening for discharging kneaded material was set to 43°C by causing hot water to flow in the jacket.
In the granulated material, granules fused with or adhering to each other were practically not observed.

[0055] The granulated material was cooled to room temperature in a dryer (vibro-fluidized bed dryer, manufactured by TOKUJU CORPORATION). Thereafter, the granulated material was disintegrated with a disintegrator and classified to a size of 2000 μm to 710 μm by using a sieve (a round vibration sifter, manufactured by TOKUJU CORPORATION), thereby obtaining granules. The yield of granules from the kneaded material was 91%.

Comparative example 1

[0056] Granules were produced in the same manner as in Examples 1, except that in the continuous double-screw kneading device, the heating temperature of the section (first section) close to the opening for injecting kneaded material was changed to 113°C, the heating temperature of the section (second section) close to the opening for discharging kneaded material was changed to 103°C, and the product temperature at the time when the kneaded material was discharged from the continuous double-screw kneading device was changed to 84°C. As a result, a slightly soft kneaded material was obtained. The granulated material contained a large amount of granules that adhered to or were fused with each other. The yield of granules from the kneaded material was 81%.

Comparative example 2

[0057] Granules were obtained in the same manner as in Example 1, except that in the continuous double-screw kneading device, the heating temperature of the section (first section) close to the opening for injecting kneaded material was changed to 90°C, the heating temperature of the section (second section) close to the opening for discharging kneaded material was changed to 22°C, and the product temperature at the time when the kneaded material was discharged from the continuous double-screw kneading device was changed to 76°C. As a result, due to insufficient kneading, a kneaded material having hard surfaces was obtained. The granulated material contained an extremely large amount of powdery materials or finely split materials. The yield of granules from the kneaded material was 72%.

Comparative example 3

[0058] An attempt at producing granules was made in the same manner as in Example 1, except that in the continuous double-screw kneading device, the heating temperature of the section (first section) close to the opening for injecting kneaded material was changed to 85°C, the heating temperature of the section (second section) close to the opening for discharging kneaded material was changed to 55°C, and the product temperature at the time when the kneaded material was dis- charged from the continuous double-screw kneading device was changed to 79°C. As a result, due to insufficient kneading, a powdery kneaded material was obtained, and extruding granulation could not be performed.

Comparative example 4

[0059] An attempt at producing granules was made in the same manner as in Example 1, except that in the continuous double-screw kneading device, the heating temperature of the section (first section) close to the opening for injecting kneaded material was changed to 95°C, the heating temperature of the section (second section) close to the opening for discharging kneaded material was changed to 95°C, and the product temperature at the time when the kneaded material was discharged from the continuous double-screw kneading device was changed to 98°C. As a result, an extremely soft kneaded material was obtained. The materials extruded from the granulating machine adhered to or were fused with each other, and formed a shape of candies or dumplings. Accordingly, they could not be granulated.

INDUSTRIAL APPLICABILITY

[0060] According to the method for producing agrochemical granules of the present invention, it is possible to stably produce agrochemical granules containing an agrochemical active ingredient, one or more kinds of hydrophobic substances, and a substance capable of absorbing oil, with high quality. Accordingly, the present invention is extremely useful in the industrial field.

Claims

1. A method for producing agrochemical granules, comprising:

(ii) a step of obtaining a kneaded material by loading an agrochemical active ingredient, one or more kinds of hydrophobic substances, and a substance capable of absorbing oil into a kneading device, and kneading them at a heating temperature of 8°C higher than the highest melting point of the hydrophobic substances and equal to or lower than 130°C, and then causing the material to be discharged from the kneading device at a temperature of 4°C or more lower than the highest melting point of the hydrophobic substances and equal to or higher than 60°C; and

(iii) a step of granulating the obtained kneaded material by an extrusion molding method, wherein

a heating temperature in a section close to the opening for discharging kneaded material of the kneading...
device is 30°C to 70°C; and
steps (ii) and (iii) are performed consecutively.

2. A method for producing agrochemical granules, comprising:

(i) a step of obtaining a mixture by mixing an agrochemical active ingredient, one or more kinds of hydrophobic substances, and a substance capable of absorbing oil together;
(ii) a step of obtaining a kneaded material by loading the obtained mixture into a kneading device, kneading the mixture at a heating temperature which is 8°C higher than the highest melting point of the hydrophobic substances and equal to or lower than 130°C, and then causing the kneaded material to be discharged from the kneading device at a temperature of 4°C or more lower than the highest melting point of the hydrophobic substances and equal to or higher than 60°C; and
(iii) a step of granulation of the obtained kneaded material by an extrusion molding method, wherein

a heating temperature in a section close to the opening for discharging kneaded material of the kneading device is 30°C to 70°C and

steps (ii) and (iii) are performed consecutively.

3. The method for producing agrochemical granules according to claim 1 or 2, further comprising:

a step of disintegrating the granulated material after step (iii).

4. The method for producing agrochemical granules according to any one of Claims 1 to 3, wherein water solubility of the agrochemical active ingredient is 60 ppm or greater.

5. The method for producing agrochemical granules according to any one of Claims 1 to 4, wherein the agrochemical active ingredient is at least one kind selected from the group consisting of nitenpyram, imidacloprid, acetamiprid, thiamethoxam, clothianidin, thiacloprid, and dinotefuran.

6. The method for producing agrochemical granules according to any one of Claims 1 to 5, wherein the hydrophobic substance is at least one kind selected from the group consisting of candelilla wax, fatty acids or hydrogenated products thereof that are obtained by decomposing vegetable fat-and-oil or animal fat-and-oil, stearic acid, behenic acid, hydrogenated fatty acids of rapeseed, hydrogenated palm fatty acids, hydrogenated fatty acids of beef tallow, hydrogenated castor oil, paraffin wax, microcrystalline wax, and montanolic acid ester wax.

7. The method for producing agrochemical granules according to any one of Claims 1 to 6, wherein the substance capable of absorbing oil is at least one kind selected from the group consisting of amorphous silicon dioxide, starch, starch derivatives, and celluloses.

8. The method for producing agrochemical granules according to any one of Claims 1 to 7, further containing: a release control agent.

9. The method for producing agrochemical granules according to any one of Claims 1 to 8, wherein the content of the agrochemical active ingredient is 0.01 to 50% by weight, the content of the hydrophobic substance is 15 to 80% by weight, and the content of the substance capable of absorbing oil is 0.05 to 30% by weight.

10. The method for producing agrochemical granules according to any one of Claims 1 to 9, wherein the kneading device is a continuous single-screw kneading device or a continuous double-screw kneading device.

Patentansprüche

1. Verfahren zur Herstellung agrochemischer Granula umfassend:

(ii) einen Schritt des Erhaltens eines gekneteten Materials durch Beladen einer Knetvorrichtung mit einem agrochemischen Aktivbestandteil, einer oder mehrerer Arten von hydrophoben Substanzen und einer Substanz, die befähigt ist, Öl zu absorbieren, und Kneten derselben bei einer Heiztemperatur von 8°C größer als der höchste Schmelzpunkt der hydrophoben Substanzen und gleich oder weniger als 130°C; und Veranlassen, dass das Material aus der Knetvorrichtung bei einer Temperatur von 4°C oder mehr geringer als der höchste Schmelzpunkt der hydrophoben Substanzen und gleich oder größer als 60°C entladen wird; und

(iii) einen Schritt der Granulierung des erhaltenen gekneteten Materials mittels eines Extrusionsformgebungsverfahrens, worin

eine Heiztemperatur in dem der Öffnung zum Entladen des gekneteten Materials aus der Knetvorrichtung benachbarten Bereich von 30°C bis 70°C ist; und die Schritte (ii) und (iii) werden aufeinanderfolgend durchgeführt.
2. Verfahren zur Herstellung agrochemischer Granula umfassend:

(i) einen Schritt des Erhaltens einer Mischung durch gemeinsames Mischen eines agrochemischen Aktivbestandteils, einer oder mehrerer Arten von hydrophoben Substanzen und einer Substanz, die befähigt ist, Öl zu absorbieren;
(ii) einen Schritt des Erhaltens eines gekneteten Materials durch Beladen einer Knetvorrichtung der erhaltenen Mischung, Kneten derselben bei einer Heiztemperatur von 8°C größer als der höchste Schmelzpunkt der hydrophoben Substanzen und gleich oder weniger als 130°C und dann Veranlassen, dass das Material aus der Knetvorrichtung bei einer Temperatur von 4°C oder mehr geringer als der höchste Schmelzpunkt der hydrophoben Substanzen und gleich oder größer als 60°C entladen wird; und
(iii) einen Schritt der Granulierung des erhaltenen gekneteten Materials mittels eines Extrusionsformgebungsverfahrens, worin

   eine Heiztemperatur in dem der Öffnung zum Entladen des gekneteten Materials aus der Knetvorrichtung benachbarten Bereich 30°C bis 70°C ist; und die Schritte (ii) und (iii) werden aufeinanderfolgend durchgeführt.

3. Verfahren zur Herstellung agrochemischer Granula gemäß Anspruch 1 oder 2, weiterhin umfassend:

   einen Schritt der Desintegrierung des nach Schritt (iii) erhaltenen granulierten Materials.

4. Verfahren zur Herstellung agrochemischer Granula nach irgendeinem der Ansprüche 1 bis 3, worin die Wasserlöslichkeit des agrochemischen Aktivbestandteils 60 ppm oder größer ist.

5. Verfahren zur Herstellung agrochemischer Granula nach irgendeinem der Ansprüche 1 bis 4, worin der Gehalt des agrochemischen Aktivbestandteils 0,01 bis 50 Gew.-%, der Gehalt der hydrophoben Substanz 15 bis 80 Gew.-% und der Gehalt der Substanz, die befähigt ist, Öl zu absorbieren, 0,05 bis 30 Gew.-% ist.

6. Verfahren zur Herstellung agrochemischer Granula nach irgendeinem der Ansprüche 1 bis 5, worin die Knetvorrichtung eine kontinuierlich arbeitende Einschrauben-Knetvorrichtung oder eine kontinuierlich arbeitende Doppelschrauben-Knetvorrichtung ist.

7. Verfahren zur Herstellung agrochemischer Granula nach irgendeinem der Ansprüche 1 bis 6, worin die Substanz, die befähigt ist Öl, zu absorbieren, mindestens eine Art ausgewählt aus der Gruppe bestehend aus amorphem Siliziumdioxid, Stärke, Stärkederivaten und Cellulosen.

8. Verfahren zur Herstellung agrochemischer Granula nach irgendeinem der Ansprüche 1 bis 7, weiterhin umfassend:

   ein die Freisetzung steuendes Mittel.

9. Verfahren zur Herstellung agrochemischer Granula nach irgendeinem der Ansprüche 1 bis 8, worin der Gehalt des agrochemischen Aktivbestandteils 0,01 bis 50 Gew.-%, der Gehalt der hydrophoben Substanz 15 bis 80 Gew.-% und der Gehalt der Substanz, die befähigt ist, Öl zu absorbieren, 0,05 bis 30 Gew.-% ist.

10. Verfahren zur Herstellung agrochemischer Granula nach irgendeinem der Ansprüche 1 bis 9, worin die Knetvorrichtung eine kontinuierlich arbeitende Einschrauben-Knetvorrichtung oder eine kontinuierlich arbeitende Doppelschrauben-Knetvorrichtung ist.

#### Revendications

1. Procédé de production de granulés agrochimiques, comprenant :

   (ii) une étape d’obtention d’un matériau malaxé par chargement d’un principe actif agrochimique, d’un ou de plusieurs types de substances hydrophobes et d’une substance apte à absorber de l’huile dans un dispositif de malaxage, et leur malaxage à une température de chauffage de 8 °C supérieure au point de fusion le plus élevé des substances hydrophobes et inférieure ou égale à 130 °C, puis en amenant le matériau à être déchargé du dispositif de malaxage à une température d’au moins 4 °C inférieure au point de fusion le plus élevé des substances hydrophobes et supérieure ou égale à 60 °C ; et

   (iii) une étape de granulation du matériau malaxé obtenu par un procédé de moulage par extrusion, dans laquelle

   une température de chauffage dans une section proche de l’ouverture pour décharger le matériau malaxé du dispositif de malaxage est de 30 °C à 70 °C ;
et les étapes (ii) et (iii) sont exécutées consécutivement.

2. Procédé de production de granulés agrochimiques, comprenant :

(i) une étape d’obtention d’un mélange par mélange d’un principe actif agrochimique, d’un ou de plusieurs types de substances hydrophobes et d’une substance apte à absorber de l’huile ensemble ;
(ii) une étape d’obtention d’un matériau malaxé par chargement du mélange obtenu dans un dispositif de malaxage, malaxage du mélange à une température de chauffage qui est de 8 °C supérieure au point de fusion le plus élevé des substances hydrophobes et inférieure ou égale à 130 °C, puis en amenant le matériau malaxé à être déchargé du dispositif de malaxage à une température au moins 4 °C inférieure au point de fusion le plus élevé des substances hydrophobes et supérieure ou égale à 60 °C ; et
(iii) une étape de granulation du matériau malaxé obtenu par un procédé de moulage par extrusion, dans laquelle une température de chauffage dans une section proche de l’ouverture pour décharger le matériau malaxé du dispositif de malaxage est de 30 °C à 70 °C ; et les étapes (ii) et (iii) sont exécutées consécutivement.

3. Procédé de production de granulés agrochimiques selon la revendication 1 ou 2, comprenant en outre :

une étape de désintégration du matériau granulé après l’étape (iii).

4. Procédé de production de granulés agrochimiques selon l’une quelconque des revendications 1 à 3, dans lequel la solubilité dans l’eau du principe actif agrochimique est de 60 ppm ou plus.

5. Procédé de production de granulés agrochimiques selon l’une quelconque des revendications 1 à 4, dans lequel le principe actif agrochimique est au moins un type choisi dans le groupe constitué par le nitrobenzyl, l’imidaclopride, l’acétamipride, le thiaméthoxame, la clothianidine, le thiaclopride et le diéthylfurane.

6. Procédé de production de granulés agrochimiques selon l’une quelconque des revendications 1 à 5, dans lequel la substance hydrophobe est au moins un type choisi dans le groupe constitué par la cire japonaise, la cire de riz, la cire de candelilla, les acides gras ou les produits hydrogénés de ceux-ci qui sont obtenus par décomposition de graisse et huile végétale ou de graisse et huile animale, l’acide stéarique, l’acide béhénique, les acides gras de colla hydrogénés, les acides gras de palme hydrogénés, les acides gras de suif de boeuf hydrogénés, l’huile de ricin hydrogénée, la paraffine, une cire microcrystalline et la cire d’ester d’acide montanoïde.

7. Procédé de production de granulés agrochimiques selon l’une quelconque des revendications 1 à 6, dans lequel la substance apte à absorber de l’huile est au moins un type choisi dans le groupe constitué par le dioxyde de silicium amorphe, l’amidon, les dérivés d’amidon et les celluloses.

8. Procédé de production de granulés agrochimiques selon l’une quelconque des revendications 1 à 7, contenant en outre : un agent de contrôle de libération.

9. Procédé de production de granulés agrochimiques selon l’une quelconque des revendications 1 à 8, dans lequel la teneur du principe actif agrochimique est de 0,01 à 50 % en poids, la teneur de la substance hydrophobe est de 15 à 80 % en poids, et la teneur de la substance apte à absorber de l’huile est de 0,05 à 30 % en poids.

10. Procédé de production de granulés agrochimiques selon l’une quelconque des revendications 1 à 9, dans lequel le dispositif de malaxage est un dispositif de malaxage continu à une seule vis ou un dispositif de malaxage continu à deux vis.
REFERENCES CITED IN THE DESCRIPTION

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Patent documents cited in the description

• JP 2011042222 A [0002]
• WO 9509532 A [0006]
• JP 2003252702 A [0006]
• JP 2004043370 A [0006]
• JP 2003171207 A [0007]
• JP 8092007 A [0008]