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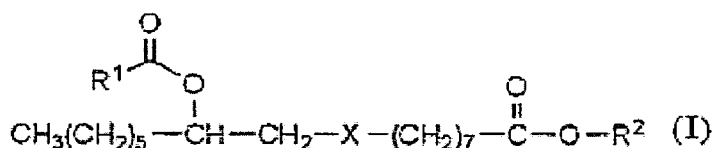
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(54) Title: METHOD FOR PRODUCING MICROCAPSULE



(57) Abstract: An object of the present invention is to provide a technique for producing a microcapsule containing a pesticidal compound in a fatty acid ester such as methyl 0- acetylrinoleate, which delays the release timing of the pesticidal compound as compared to a conventional microcapsule. Provided is a method for producing a microcapsule, which comprises: (1) keeping a mixture of a pesticidal compound, a compound represented by formula (I) : wherein X represents -CH₂-CH₂- or -CH=CH-, R¹ represents a C1-C4 alkyl group, and R² represents a C1-C4 alkyl group, and a polyisocyanate at 20 to 60°C for 3 hours or more; (2) adding the mixture to water containing a polyol or a polyamine to prepare liquid droplets in the water; and (3) forming a film of polyurethane or polyurea around the droplets.



DESCRIPTION

METHOD FOR PRODUCING MICROCAPSULE

Technical Field

5 The present invention relates to a method for producing a microcapsule and a microcapsule produced by the method.

Background Art

10 There is known a microcapsule wherein a liquid droplet, in which a pesticidal compound is suspended in a fatty acid ester such as methyl O-acetylricinoleate, is coated with a resin (see US 2009/0142406A1).

15 Summary of the Invention

Technical Problem

 A microcapsule formulation of a pesticidal compound is a formulation which is intended to control the release timing of the pesticidal compound contained therein.

20 An object of the present invention is to provide a technique for delaying the release timing of the pesticidal compound with a microcapsule containing a pesticidal compound in a fatty acid ester such as methyl O-acetylricinoleate.

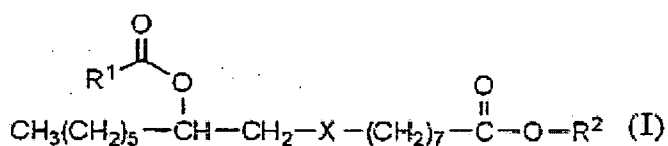
Solution to Problem

The inventors of the present invention have found that the timing of release of the pesticidal compound from the microcapsule is delayed when a microcapsule is produced by using a mixture of a pesticidal compound, a fatty acid ester such as methyl O-acetylricinoleate and a polyisocyanate being kept at 20 to 60°C for 3 hours or more. The inventors have also found that the microcapsule produced by the production method is excellent in light stability as compared to a conventional microcapsule.

The present invention provides:

[1] A method for producing a microcapsule, which comprises:

(1) keeping a mixture of a pesticidal compound, a compound represented by formula (I) and a polyisocyanate at 20 to 60°C for 3 hours or more:



wherein X represents -CH₂-CH₂- or -CH=CH-, R¹ represents a C1-C4 alkyl group, and R² represents a C1-C4 alkyl group;

(2) then adding the mixture to water containing a polyol or a polyamine, and producing liquid droplets in the water; and

(3) forming a film of polyurethane or polyurea around the droplets;

[2] The method according to [1], wherein the weight ratio of the pesticidal compound to the compound represented by formula (I) is from 10:100 to 100:100;

[3] The method according to [1] or [2], wherein the
5 compound represented by formula (I) is a C1-C4 alkyl ester of O-acetylricinoleic acid;

[4] The method according to [1] or [2], wherein the compound represented by formula (I) is methyl O-acetylricinoleate;

10 [5] The method according to any one of [1] to [4], wherein the pesticidal compound is a solid pesticidal compound;

[6] The method according to any one of [1] to [4], wherein the pesticidal compound is a neonicotinoid compound;

[7] The method according to any one of [1] to [4], wherein
15 the pesticidal compound is clothianidin; and

[8] A microcapsule produced by the method according to any one of [1] to [7].

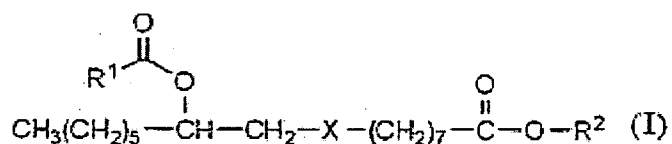
Effects of the Invention

20 According to the method for producing a microcapsule of the present invention, a microcapsule from which release of a pesticidal compound is more controlled than that from a conventional microcapsule can be obtained.

25 Description of Embodiments

The method for producing a microcapsule of the present invention comprises the following steps:

step (1) keeping a mixture of a pesticidal compound, a compound represented by formula (I) and a polyisocyanate at
 5 20 to 60°C for 3 hours or more:



wherein X represents $-\text{CH}_2-\text{CH}_2-$ or $-\text{CH}=\text{CH}-$, R^1 represents a C1-C4 alkyl group, and R^2 represents a C1-C4 alkyl group;

step (2) then adding the mixture into water containing
 10 a polyol or a polyamine, and producing liquid droplets in the water; and

step (3) forming a film of polyurethane or polyurea around the droplets.

In the present invention, the pesticidal compound is
 15 preferably a solid pesticidal compound. As used herein, the solid pesticidal compound usually means a compound having pesticidal activity and having a melting point of 15°C or higher, preferably 50°C or higher. More preferably, the solid pesticidal compound as used herein is a compound
 20 having pesticidal activity whose melting point is 15°C or higher, preferably 50°C or higher and whose solubility is 5% by weight or less in the compound represented by the above formula (I).

Examples of the pesticidal compound used in the present invention include an insecticidal compound, a fungicidal compound, a herbicidal compound, an insect growth regulating compound, a plant growth regulating compound, and an insect repelling compound.

Examples of the insecticidal compound include carbamate compounds such as propoxur, isoprocarb, xylylcarb, metolcarb, XMC, carbaryl, pirimicarb, carbofuran, methomyl, fenoxycarb, alanycarb, and metoxadiazone; organophosphorus compounds such as acephate, phenthoate, vamidothion, trichlorfon, monocrotophos, tetrachlorvinphos, dimethylvinphos, phosalone, chlorpyrifos, chlorpyrifos-methyl, pyridaphenthion, quinalphos, methidathion, methamidophos, dimethoate, formothion, azinphos-ethyl, azinphos-methyl, and salithion; neonicotinoid compounds such as imidacloprid, nitenpyram, acetamiprid, clothianidin, and thiamethoxam; 4-chloro-2-(2-chloro-2-methylpropyl)-5-(6-iodo-3-pyridylmethoxy)pyridazine-3(2H)-one, cartap, buprofezin, thiocyclam, bensultap, fenoxycarb, fenazaquin, fenpyroximate, pyridaben, hydramethylnon, thiodicarb, chlorfenapyr, fenproximate, pymetrozine, pyrimidifen, tebufenozide, tebufenpyrad, triazamate, indoxacarb, sulfluramide, milbemectin, abamectin, and paradichlorobenzene.

Examples of the fungicidal compound include

benzimidazole compounds such as benomyl, carbendazim, thiabendazole, and thiophanate-methyl; phenylcarbamate compounds such as diethofencarb; dicarboxyimide compounds such as procymidone, iprodione, and vinclozolin; azole compounds such as diniconazole, propenazole, epoxyconazole, 5 tebuconazole, difenoconazole, cyproconazole, flusilazole, and triadimefon; acylalanine compounds such as metalaxyl; carboxyamide compounds such as furametpyr, mepronil, flutolanil, and trifluzamide; organophosphorus compounds 10 such as tolclofos-methyl, fosetyl-aluminum, and pyrazophos; anilinopyrimidine compounds such as pyrimethanil, mepanipyrim, and cyprodinil; cyanopyrrole compounds such as fludioxonil, and fenpiclonil; chlorothalonil, manzeb, captan, folpet, tricyclazole, pyroquilon, probenazole, 15 fthalide, cymoxanil, dimethomorph, famoxadone, oxolinic acid and a salt thereof, fluazinam, ferimzone, diclocymet, chlobenthiazole, isovaledione, tetrachloroisophthalonitrile, thiophthalimide oxybisphenoxyarsine, and 3-iodo-2-propylbutylcarbamate.

20 Examples of the herbicidal compound include triazine compounds such as atrazine, and metribuzin; urea compounds such as fluometuron, and isoproturon; hydroxybenzonitrile compounds such as bromoxynil, and ioxynil; 2,6-dinitroaniline compounds such as pendimethalin, and 25 trifluralin; aryloxyalkanoic acid compounds and salts

thereof such as 2,4-D, dicamba, fluroxypyr, and mecoprop;
sulfonylurea compounds such as bensulfuron-methyl,
metsulfuron-methyl, nicosulfuron, primisulfuron-methyl, and
cyclosulfamuron; imidazolinone compounds and salts thereof
5 such as imazapyr, imazaquin, and imazethapyr; sulfentrazone,
paraquat, flumetsulam, triflusulfuron-methyl, fenoxaprop-p-
ethyl, cyhalofop-butyl, diflufenican, norflurazon,
isoxaflutole, glufosinate ammonium salts, glyphosate salts,
bentazone, benthocarb, mefenacet, propanil, fluthiamide,
10 flumiclorac-pentyl, and flumioxazin.

Examples of the insect growth regulating compound
include benzoylurea compounds such as diflubenzuron,
chlorfluazuron, lufenuron, hexaflumuron, flufenoxuron,
flucyclohexuron, cyromazine, diafenthiuron, hexythiazox,
15 novaluron, teflubenzuron, triflumuron, 1-(2,6-
difluorobenzoyl)-3-[2-fluoro-4-(trifluoromethyl)phenyl]urea,
1-(2,6-difluorobenzoyl)-3-[2-fluoro-4-(1,1,2,3,3,3-
hexafluoropropoxy)phenyl]urea, and 1-(2,6-difluorobenzoyl)-
3-[2-fluoro-4-(1,1,2,2-tetrafluoroethoxy)phenyl]urea; and
20 pyriproxyfen.

Examples of the plant growth regulating compound
include maleic hydrazide, chlormequat, ethephon,
gibberellin, mepichat chloride, thidiazuron, inabenfide,
paclobutrazol, and uniconazole.

25 Examples of the insect repelling compound include

1S, 3R, 4R, 6R-carane-3,4-diol, and dipropyl 2,5-pyridinedicarboxylate.

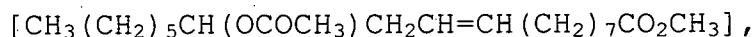
Examples of the C1-C4 alkyl groups represented by R¹ or R² in formula (I), as used herein, include a methyl
5 group, an ethyl group, a propyl group and a butyl group.

The compound represented by formula (I) can be obtained by converting the carboxyl group of ricinoleic acid or 12-hydroxystearic acid into an alkoxycarbonyl group by condensation with a lower alcohol compound, and further
10 converting the hydroxyl group of ricinoleate or 12-hydroxystearate into an acyloxy group by condensation with a lower fatty acid.

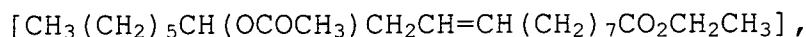
Examples of the compound represented by formula (I) include C1-C4 alkyl esters of O-acetylricinoleic acid and
15 C1-C4 alkyl esters of 12-acetoxystearic acid.

More specific examples of the compound represented by formula (I) include:

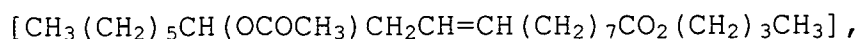
methyl O-acetylricinoleate



20 ethyl O-acetylricinoleate



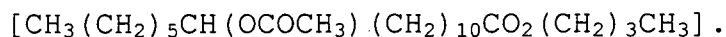
butyl O-acetylricinoleate



methyl 12-acetoxysteareate $[\text{CH}_3(\text{CH}_2)_5\text{CH}(\text{OCOCH}_3)(\text{CH}_2)_{10}\text{CO}_2\text{CH}_3],$

25 and

butyl 12-acetoxysteareate



Examples of the polyisocyanate used in the present invention include hexamethylene diisocyanate, an adduct of
5 hexamethylene diisocyanate and trimethylolpropane, a biuret condensate of 3 molecules of hexamethylene diisocyanate, an adduct of tolylene diisocyanate and trimethylolpropane, an isocyanurate condensate of tolylene diisocyanate, an isocyanurate condensate of hexamethylene diisocyanate, an
10 isocyanurate condensate of isophorone diisocyanate, an isocyanate prepolymer in which one isocyanate part of hexamethylene diisocyanate forms an isocyanurate component together with 2 molecules of tolylene diisocyanate and the other isocyanate part forms an isocyanurate component
15 together with 2 molecules of other hexamethylene diisocyanate, 4,4'-methylene bis(cyclohexylisocyanate), and trimethylhexamethylene diisocyanate.

In the present invention, the weight ratio of the pesticidal compound to the compound represented by formula
20 (I), that is, the weight ratio of the pesticidal compound to the compound represented by formula (I) is usually from 10:100 to 100:100, preferably from 20:100 to 40:100.

The amount of the polyisocyanate used in the present invention is usually determined depending on the amount of
25 the film of a microcapsule to be produced. The amount of

the film of a microcapsule to be produced is usually from 5 to 45% by weight, preferably from 10 to 30% by weight of the weight of the entire microcapsule. The amount of the polyisocyanate used in the present invention is usually
5 from 25 to 90% by weight, preferably from 40 to 70% by weight of the weight of the film of a microcapsule.

The pesticidal compound is usually dissolved or suspended in the compound represented by formula (I).

When the pesticidal compound is dissolved in the
10 compound represented by formula (I), the mixture of a pesticidal compound, a compound represented by formula (I) and a polyisocyanate used in the step (1) can be prepared by mixing the pesticidal compound, the compound represented by formula (I) and the polyisocyanate.

15 When the pesticidal compound is a solid pesticidal compound, the solid pesticidal compound suspension in the compound represented by formula (I) may be formed in accordance with the solubility of the solid pesticidal compound in the compound represented by formula (I) and the
20 weight ratio of the solid pesticidal compound to the compound represented by formula (I). When the solid pesticidal compound is suspended in the compound represented by formula (I), the mixture of a pesticidal compound, a compound represented by formula (I) and a
25 polyisocyanate used in the step (1) can be prepared, for

example, by grinding the solid pesticidal compound in the compound represented by formula (I) to obtain a suspension and then adding the polyisocyanate into the resultant suspension.

5 An example of a method for grinding the solid
pesticidal compound in the compound represented by formula
(I) is a wet grinding method which comprises adding the
solid pesticidal compound and, if necessary, beads or the
like for grinding to the compound represented by formula
10 (I) and then grinding the mixture with a grinder. Examples
of the grinder include mills such as a bead mill, a ball
mill and a rod mill, and rotor-stator homogenizers.
Specific examples of the mill include Attritor
(manufactured by MITSUI MIIKE MACHINERY CO., LTD.), Dyno
15 Mill (manufactured by WILLY A. BACHOFEN AG.
MASCHINENFABRIK), Colloid Mill (manufactured by PRIMIX
CORP.) and Pearl Mill (manufactured by ASHIZAWA FINETECH,
LTD.). Specific examples of the rotor-stator homogenizer
include Polytron Homogenizer (manufactured by KINEMATICA
20 AG).

Grinding of the solid pesticidal compound in the
compound represented by formula (I) may be attained by 2 or
more operations. For grinding the solid pesticidal
25 compound in the compound represented by formula (I), for

example, the solid pesticidal compound may be coarsely ground in the first operation and then finely ground in the second operation. An example of a method for grinding the solid pesticidal compound in the compound represented by formula (I) in 2 operations is a method which comprises
5 using a rotor-stator homogenizer for the first operation and using a mill for the second operation.

When the solid pesticidal compound is suspended in the compound represented by formula (I), the particle size of the solid pesticidal compound suspended in the compound
10 represented by formula (I) is usually 10 μm or less, preferably in the range of 1 to 5 μm in terms of volume median diameter. It is preferred that, based on the gross volume of the particles of the solid pesticidal compound suspended in the compound represented by formula (I), the
15 net volume of particles with a diameter of 10 μm or more is 10% or less.

In the present invention, the mixture of a pesticidal compound, a compound represented by formula (I) and a polyisocyanate may further contain an organic solvent.
20 Examples of the organic solvent include aliphatic hydrocarbons such as trimethylpentane, aromatic hydrocarbons such as phenylxylylethane, alkylbenzene and alkylnaphthalene, ethers such as 2-ethylhexyl ether, and
25 vegetable oils such as cotton seed oil. When the mixture

contains an organic solvent, the weight ratio of the organic solvent based on the weight of the compound represented by formula (I) is usually $1/2$ or less, preferably $3/7$ or less, more preferably $1/4$ or less.

5 In the step (1) of the microcapsule production method of the present invention, the mixture of a pesticidal compound, a compound represented by formula (I) and a polyisocyanate thus obtained is kept at 20 to 60°C for 3 hours or more, preferably at 20 to 40°C for 5 hours or more.

10 While the mixture of a pesticidal compound, a compound represented by formula (I) and a polyisocyanate is kept at 20 to 60°C, the mixture may be stirred or allowed to stand. In the step (1), the mixture is usually controlled so as to be kept at 20 to 60°C.

15 In the step (2) of the microcapsule production method of the present invention, the mixture obtained in the step (1) is added to water containing a polyol or a polyamine, and liquid droplets are produced in the water.

20 When the water contains a polyol in this step, a microcapsule with a polyurethane film is produced. When the water contains a polyamine in this step, a microcapsule with a polyurea film is produced.

The water containing a polyol can be prepared, for example, by mixing water and a polyol. The water
25 containing a polyamine can be prepared, for example, by

mixing water and a polyamine or a polyamine salt.

Examples of the polyol as used herein include ethylene glycol, propylene glycol, butylene glycol, and cyclopropylene glycol. Examples of the polyamine as used
5 herein include ethylenediamine, hexamethylenediamine, diethyltri-amine, and triethylenetetramine.

The amount of the polyol or polyamine used in the present invention is usually determined depending on the amount of the film of a microcapsule to be produced. The
10 amount of the polyol used in the present invention is usually from 5 to 80% by weight, preferably from 20 to 60% by weight of the amount of the film of a microcapsule. The amount of the polyamine used in the present invention is usually from 5 to 80% by weight, preferably from 20 to 60%
15 by weight of the amount of the film of a microcapsule.

The weight ratio of water used in the step (2) is usually in a range from 0.8 to 2 times the weight of the mixture obtained in the step (1). As water used in the step (2), deionized water is preferably used. Water used
20 in the step (2) may contain a thickening agent, if necessary.

Examples of the thickening agent include natural polysaccharides such as xanthan gum, rhamsan gum, locust bean gum, carrageenan, welan gum and gum arabic; synthetic
25 polymers such as sodium polyacrylate; semi-synthetic

polymers such as carboxymethyl cellulose; mineral powders such as aluminum magnesium silicate, smectite, bentonite, hectorite and fumed silica; and alumina sol.

An example of a method for preparing liquid droplets in water in the step (2) is a method which comprises adding the mixture obtained in the step (1) to water containing a polyol or a polyamine and then stirring the resultant mixture with a stirrer. Examples of the stirrer used in this operation include a propeller stirrer, a turbine stirrer and a high-speed shear stirrer. Specific examples of the stirrer include T.K. Homo Mixer, T.K. Homomic Line flow, T.K. Pipeline Homo Mixer, and T.K. Fill Mix (manufactured by PRIMIX CORP); Clearmix (manufactured by M TECHNIQUE CO., LTD.); Polytron Homogenizer and Megatron Homogenizer (manufactured by KINEMATICA); and Supraton (manufactured by TSUKISHIMA KIKAI CO., LTD.).

The particle size of microcapsules finally produced by the method of the present invention is almost identical to that of the liquid droplets prepared in the step (2). The liquid droplets prepared in the step (2) and the microcapsules finally produced by the method of the present invention have a particle diameter of usually in the range of 1 to 80 μm , preferably 5 to 50 μm in terms of volume median diameter.

The liquid droplets existing in water obtained in the

step (2) are made of a solution of a polyisocyanate in a compound represented by formula (I). The polyisocyanate contained in the liquid droplets is polymerized with the polyol or polyamine existing in the water at the interfaces between the liquid droplets and the water. As a result, a polyurethane or polyurea film is formed around the liquid droplets to give an aqueous suspension of microcapsules.

For the polyurethane film, the film of polyurethane resin is formed around the liquid droplets, for example, by heating a water dispersion of the liquid droplets prepared in the step (2) to 40 to 80°C and then keeping the dispersion at the same temperature for about 0.5 to 48 hours while stirring. For the polyurea film, the film of polyurea resin is formed around the liquid droplets, for example, by adjusting a water dispersion of the liquid droplets prepared in the step (2) to a neutral to weak alkaline pH and then keeping the dispersion at 0 to 50°C for about 0.5 to 48 hours.

According to the method as described above, a microcapsule is produced in the form of a water suspension.

The water suspension of the microcapsule thus produced can be subjected to centrifugation, filtration or spray drying to obtain a powder formulation of the microcapsule.

To the water suspension of the microcapsule produced by the method as described above, a thickening agent, an

antifreezing agent, a preservative, a density regulator, a pH regulator or water can be further added. In this case, the microcapsule thus produced can be used, for example, as a water suspended pesticidal composition containing 0.1 to 5 30% by weight of a pesticidal compound.

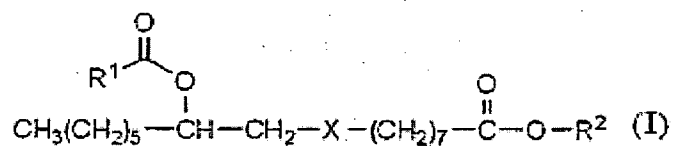
Examples of the thickening agent include those described above. Examples of the antifreezing agent include propylene glycol. Examples of the preservative include p-hydroxybenzoic acid esters; isothiazoline 10 derivatives such as 5-chloro-2-methyl-4-isothiazolin-3-one and 2-methyl-4-isothiazolin-3-one; 2-bromo-2-nitropropane-1,3-diol; and salicylic acid derivatives. Specific examples of the preservative include Biohope L (manufactured by K.I. CHEMICAL INDUSTRY CO., LTD.) and 15 Proxel GXL (manufactured by AVECIA K.K.). Examples of the density regulator include water-soluble salts such as sodium sulfate and water-soluble compounds such as urea. Examples of the pH regulator include disodium hydrogen phosphate, dipotassium hydrogen phosphate and sodium 20 hydroxide.

When the pesticidal compound is an insecticidal compound, a pesticidal composition containing the microcapsule of the present invention is sprayed on pests or habitats of pests in an amount of about 0.1 to 1,000 25 g/1,000 m², preferably about 1 to 100 g/1,000 m² of the

pesticidal compound.

Examples of a microcapsule produced by the method of the present invention are shown below.

A microcapsule produced by a method for producing a
 5 microcapsule which comprises keeping a mixture of a
 pesticidal compound, a compound represented by formula (I)
 and a polyisocyanate at 20 to 60°C for 3 hours or more:



wherein X represents $-\text{CH}_2-\text{CH}_2-$ or $-\text{CH}=\text{CH}-$, R^1 represents a
 10 C1-C4 alkyl group, and R^2 represents a C1-C4 alkyl group;
 then adding the mixture to water containing a polyol or a
 polyamine, and producing liquid droplets in the water; and
 then forming a film of polyurethane or polyurea around the
 droplets (hereinafter, referred to as the present
 15 microcapsule).

The present microcapsule, wherein the net volume of
 microcapsule particles with a particle diameter of 5 μm or
 less is less than 20% based on the gross volume of
 microcapsule particles and the net volume of microcapsule
 20 particles with a particle diameter of 50 μm or more is less
 than 20% based on the gross volume of microcapsule
 particles.

The present microcapsule, wherein the net volume of

microcapsule particles with a particle diameter of 5 μm or less is less than 20% based on the gross volume of microcapsule particles, the net volume of microcapsule particles with a particle diameter of 50 μm or more is less than 20% based on the gross volume of microcapsule particles, and the ratio of volume median diameter/film thickness is from 25 to 150.

The present microcapsule, wherein the net volume of microcapsule particles with a particle diameter of 5 μm or less is less than 20% based on the gross volume of microcapsule particles, the net volume of microcapsule particles with a particle diameter of 50 μm or more is less than 20% based on the gross volume of microcapsule particles, and the film is made of polyurethane.

The present microcapsule, wherein the net volume of microcapsule particles with a particle diameter of 5 μm or less is less than 20% based on the gross volume of microcapsule particles, the net volume of microcapsule particles with a particle diameter of 50 μm or more is less than 20% based on the gross volume of microcapsule particles, the ratio of volume median diameter/film thickness is from 25 to 150, and the film is made of polyurethane.

The present microcapsule, wherein the weight ratio of the pesticidal compound to the compound represented by

formula (I) is from 20:100 to 40:100, the net volume of microcapsule particles with a particle diameter of 5 μm or less is less than 20% based on the gross volume of microcapsule particles, and the net volume of microcapsule particles with a particle diameter of 50 μm or more is less than 20% based on the gross volume of microcapsule particles.

The present microcapsule, wherein the weight ratio of the pesticidal compound to the compound represented by formula (I) is from 20:100 to 40:100, the net volume of microcapsule particles with a particle diameter of 5 μm or less is less than 20% based on the gross volume of microcapsule particles, the net volume of microcapsule particles with a particle diameter of 50 μm or more is less than 20% based on the gross volume of microcapsule particles, and the ratio of volume median diameter/film thickness is from 25 to 150.

The present microcapsule, wherein the weight ratio of the pesticidal compound to the compound represented by formula (I) is from 20:100 to 40:100, the net volume of microcapsule particles with a particle diameter of 5 μm or less is less than 20% based on the gross volume of microcapsule particles, the net volume of microcapsule particles with a particle diameter of 50 μm or more is less than 20% based on the gross volume of microcapsule

particles, and the film is made of polyurethane.

The present microcapsule, wherein the weight ratio of the pesticidal compound to the compound represented by formula (I) is from 20:100 to 40:100, the net volume of
5 microcapsule particles with a particle diameter of 5 μm or less is less than 20% based on the gross volume of microcapsule particles, the net volume of microcapsule particles with a particle diameter of 50 μm or more is less than 20% based on the gross volume of microcapsule
10 particles, the ratio of volume median diameter/film thickness is from 25 to 150, and the film is made of polyurethane.

The present microcapsule, which contains an isothiazoline derivative as a preservative, and wherein the
15 net volume of microcapsule particles with a particle diameter of 5 μm or less is less than 20% based on the gross volume of microcapsule particles and the net volume of microcapsule particles with a particle diameter of 50 μm or more is less than 20% based on the gross volume of
20 microcapsule particles.

The present microcapsule, which contains an isothiazoline derivative as a preservative, and wherein the net volume of microcapsule particles with a particle diameter of 5 μm or less is less than 20% based on the
25 gross volume of microcapsule particles, the net volume of

microcapsule particles with a particle diameter of 50 μm or more is less than 20% based on the gross volume of microcapsule particles, and the ratio of volume median diameter/film thickness is from 25 to 150.

5 The present microcapsule, which contains an isothiazoline derivative as a preservative, and wherein the net volume of microcapsule particles with a particle diameter of 5 μm or less is less than 20% based on the gross volume of microcapsule particles, the net volume of
10 microcapsule particles with a particle diameter of 50 μm or more is less than 20% based on the gross volume of microcapsule particles, and the film is made of polyurethane.

 The present microcapsule, which contains an
15 isothiazoline derivative as a preservative, and wherein the net volume of microcapsule particles with a particle diameter of 5 μm or less is less than 20% based on the gross volume of microcapsule particles, the net volume of microcapsule particles with a particle diameter of 50 μm or
20 more is less than 20% based on the gross volume of microcapsule particles, the ratio of volume median diameter/film thickness is from 25 to 150, and the film is made of polyurethane.

 The present microcapsule, which contains an
25 isothiazoline derivative as a preservative, and wherein the

weight ratio of the pesticidal compound to the compound represented by formula (I) is from 20:100 to 40:100, the net volume of microcapsule particles with a particle diameter of 5 μm or less is less than 20% based on the gross volume of microcapsule particles, and the net volume of microcapsule particles with a particle diameter of 50 μm or more is less than 20% based on the gross volume of microcapsule particles.

The present microcapsule, which contains an isothiazoline derivative as a preservative, and wherein the weight ratio of the pesticidal compound to the compound represented by formula (I) is from 20:100 to 40:100, the net volume of microcapsule particles with a particle diameter of 5 μm or less is less than 20% based on the gross volume of microcapsule particles, the net volume of microcapsule particles with a particle diameter of 50 μm or more is less than 20% based on the gross volume of microcapsule particles, and the ratio of volume median diameter/film thickness is from 25 to 150.

The present microcapsule, which contains an isothiazoline derivative as a preservative, and wherein the weight ratio of the pesticidal compound to the compound represented by formula (I) is from 20:100 to 40:100, the net volume of microcapsule particles with a particle diameter of 5 μm or less is less than 20% based on the

gross volume of microcapsule particles, the net volume of microcapsule particles with a particle diameter of 50 μm or more is less than 20% based on the gross volume of microcapsule particles, and the film is made of polyurethane.

The present microcapsule, which contains an isothiazoline derivative as a preservative, and wherein the weight ratio of the pesticidal compound to the compound represented by formula (I) is from 20:100 to 40:100, the net volume of microcapsule particles with a particle diameter of 5 μm or less is less than 20% based on the gross volume of microcapsule particles, the net volume of microcapsule particles with a particle diameter of 50 μm or more is less than 20% based on the gross volume of microcapsule particles, the ratio of volume median diameter/film thickness is from 25 to 150, and the film is made of polyurethane.

The present microcapsule, which contains a pH regulator and an isothiazoline derivative as a preservative, and wherein the net volume of microcapsule particles with a particle diameter of 5 μm or less is less than 20% based on the gross volume of microcapsule particles and the net volume of microcapsule particles with a particle diameter of 50 μm or more is less than 20% based on the gross volume of microcapsule particles.

The present microcapsule, which contains a pH regulator and an isothiazoline derivative as a preservative, and wherein the net volume of microcapsule particles with a particle diameter of 5 μm or less is less than 20% based on the gross volume of microcapsule particles, the net volume of microcapsule particles with a particle diameter of 50 μm or more is less than 20% based on the gross volume of microcapsule particles, and the ratio of volume median diameter/film thickness is from 25 to 150.

The present microcapsule, which contains a pH regulator and an isothiazoline derivative as a preservative, and wherein the net volume of microcapsule particles with a particle diameter of 5 μm or less is less than 20% based on the gross volume of microcapsule particles, the net volume of microcapsule particles with a particle diameter of 50 μm or more is less than 20% based on the gross volume of microcapsule particles, and the film is made of polyurethane.

The present microcapsule, which contains a pH regulator and an isothiazoline derivative as a preservative, and wherein the net volume of microcapsule particles with a particle diameter of 5 μm or less is less than 20% based on the gross volume of microcapsule particles, the net volume of microcapsule particles with a particle diameter of 50 μm or more is less than 20% based on the gross volume of

microcapsule particles, the ratio of volume median diameter/film thickness is from 25 to 150, and the film is made of polyurethane.

The present microcapsule, which contains a pH
5 regulator and an isothiazoline derivative as a preservative,
and wherein the weight ratio of the pesticidal compound to
the compound represented by formula (I) is from 20:100 to
40:100, the net volume of microcapsule particles with a
particle diameter of 5 μm or less is less than 20% based on
10 the gross volume of microcapsule particles, and the net
volume of microcapsule particles with a particle diameter
of 50 μm or more is less than 20% based on the gross volume
of microcapsule particles.

The present microcapsule, which contains a pH
15 regulator and an isothiazoline derivative as a preservative,
and wherein the weight ratio of the pesticidal compound to
the compound represented by formula (I) is from 20:100 to
40:100, the net volume of microcapsule particles with a
particle diameter of 5 μm or less is less than 20% based on
20 the gross volume of microcapsule particles, the net volume
of microcapsule particles with a particle diameter of 50 μm
or more is less than 20% based on the gross volume of
microcapsule particles, and the ratio of volume median
diameter/film thickness is from 25 to 150.

25 The present microcapsule, which contains a pH

regulator and an isothiazoline derivative as a preservative,
and wherein the weight ratio of the pesticidal compound to
the compound represented by formula (I) is from 20:100 to
40:100, the net volume of microcapsule particles with a
particle diameter of 5 μm or less is less than 20% based on
the gross volume of microcapsule particles, the net volume
of microcapsule particles with a particle diameter of 50 μm
or more is less than 20% based on the gross volume of
microcapsule particles, and the film is made of
polyurethane.

The present microcapsule, which contains a pH
regulator and an isothiazoline derivative as a preservative,
and wherein the weight ratio of the pesticidal compound to
the compound represented by formula (I) is from 20:100 to
40:100, the net volume of microcapsule particles with a
particle diameter of 5 μm or less is less than 20% based on
the gross volume of microcapsule particles, the net volume
of microcapsule particles with a particle diameter of 50 μm
or more is less than 20% based on the gross volume of
microcapsule particles, the ratio of volume median
diameter/film thickness is from 25 to 150, and the film is
made of polyurethane.

Examples

Hereinafter, the present invention will be described

in more detail by way of production examples and test examples, which the present invention is not limited to.

Production Example 1

5 250 g of clothianidin and 750 g of methyl O-acetylricinoleate (Ricsizer C-101, manufactured by Itoh Oil Chemicals Co., Ltd., content: 95.5%) were mixed (the resultant mixture is referred to as the mixture 1-1). The mixture 1-1 was stirred with a rotor-stator homogenizer
10 (Polytron Homogenizer, manufactured by KINEMATICA AG) to grind clothianidin contained in the mixture 1-1 for about 10 minutes (the resultant mixture is referred to as the mixture 1-2). The clothianidin particles in the mixture 1-2 had a volume median diameter of 0.5 mm.

15 The mixture 1-2 was fed into Dyno Mill (manufactured by WILLY A. BACHOFEN AG. MASCHINENFABRIK, vessel size: 600 mL, filled with 1,150 g of spherical glass of 1 mm diameter, revolution speed of impeller: 12 m/sec as a peripheral speed) at a rate of 3 L/hr to further grind the
20 clothianidin particles (the resultant mixture is referred to as the mixture 1-3). The clothianidin particles in the mixture 1-3 had a volume median diameter of 2.4 μ m, wherein the net volume of the particles with a diameter of 10 μ m or more was 1.6% based on the gross volume of microcapsule
25 particles.

To 100 g of the mixture 1-3 was added 21.6 g of a polyisocyanate (Sumidur L-75, adduct of trimethylolpropane and toluenediisocyanate, manufactured by SUMIKA BAYER URETHANE CO., LTD.) at 20°C (the resultant mixture is referred to as the mixture 1-4). The mixture 1-4 was kept at 20°C for 24 hours (the resultant mixture is referred to as the mixture 1-5).

To 108.8 g of water (deionized water) were added 12.4 g of ethylene glycol and 12.6 g of gum arabic (Arabic Cole SS manufactured by SAN-EI YAKUHHIN BOEKI CO., LTD.) to prepare an aqueous phase. All of the aqueous phase and all of the mixture 1-5 were mixed (the resultant mixture is referred to as the mixture 1-6).

The mixture 1-6 was stirred with a homogenizer (T.K. Autohomomixer, manufactured by PRIMIX CORP., revolution speed: 9,000 rpm) at room temperature to form liquid droplets in water (the resultant mixture is referred to as the mixture 1-7).

The mixture 1-7 was stirred at 60°C for 24 hours to give an aqueous suspension composition of clothianidin microcapsules (hereinafter, referred to as the aqueous suspension composition 1).

The resultant microcapsules had a volume median diameter of 20.9 μm , wherein the net volume of the microcapsules with a particle diameter of 5 μm or less was

8.9% based on the gross volume of microcapsule particles, and the net volume of the microcapsules with a particle diameter of 50 μm or more was 0% based on the gross volume of microcapsule particles.

5

Production Example 2

250 g of clothianidin and 750 g of methyl O-acetylricinoleate (Ricsizer C-101, manufactured by Itoh Oil Chemicals Co., Ltd., content: 95.5%) were mixed (the resultant mixture is referred to as the mixture 2-1). The mixture 2-1 was stirred with a rotor-stator homogenizer (Polytron Homogenizer, manufactured by KINEMATICA AG) to grind clothianidin contained in the mixture 2-1 for about 10 minutes (the resultant mixture is referred to as the mixture 2-2). The clothianidin particles in the mixture 2-2 had a volume median diameter of 0.5 mm.

The mixture 2-2 was fed into Dyno Mill (manufactured by WILLY A. BACHOFEN AG. MASCHINENFABRIK, vessel size: 600 mL, filled with 1,150 g of spherical glass of 1 mm diameter, revolution speed of impeller: 12 m/sec as a peripheral speed) at a rate of 3 L/hr to further grind the clothianidin particles (the resultant mixture is referred to as the mixture 2-3). The clothianidin particles in the mixture 2-3 had a volume median diameter of 2.7 μm , wherein the net volume of the particles with a diameter of 10 μm or

more was 1.9% based on the gross volume of microcapsule particles.

To 100 g of the mixture 2-3 was added 21.6 g of a polyisocyanate (Sumidur L-75, adduct of trimethylolpropane and toluenediisocyanate, manufactured by SUMIKA BAYER URETHANE CO., LTD.) at 40°C (the resultant mixture is referred to as the mixture 2-4). The mixture 2-4 was kept at 40°C for 5 hours (the resultant mixture is referred to as the mixture 2-5).

To 108.8 g of water (deionized water) were added 12.4 g of ethylene glycol and 12.6 g of gum arabic (Arabic Cole SS manufactured by SAN-EI YAKUHIH BOEKI CO., LTD.) to prepare an aqueous phase. All of the aqueous phase and all of the mixture 2-5 were mixed (the resultant mixture is referred to as the mixture 2-6).

The mixture 2-6 was stirred with a homogenizer (T.K. Autohomomixer, manufactured by PRIMIX CORP., revolution speed: 9,000 rpm) at room temperature to form liquid droplets in water (the resultant mixture is referred to as the mixture 2-7).

The mixture 2-7 was stirred at 60°C for 24 hours to give an aqueous suspension composition of clothianidin microcapsules (hereinafter, referred to as the aqueous suspension composition 2).

The resultant microcapsules had a volume median

diameter of 17.7 μm , wherein the net volume of the microcapsules with a particle diameter of 5 μm or less was 11.6% based on the gross volume of microcapsule particles, and the net volume of the microcapsules with a particle diameter of 50 μm or more was 0% based on the gross volume of microcapsule particles.

Production Example 3

250 g of clothianidin and 750 g of methyl O-acetylricinoleate (Ricsizer C-101, manufactured by Itoh Oil Chemicals Co., Ltd., content: 95.5%) were mixed (the resultant mixture is referred to as the mixture 3-1). The mixture 3-1 was stirred with a rotor-stator homogenizer (Polytron Homogenizer, manufactured by KINEMATICA AG) to grind clothianidin contained in the mixture 3-1 for about 10 minutes (the resultant mixture is referred to as the mixture 3-2). The clothianidin particles in the mixture 3-2 had a volume median diameter of 0.5 mm.

The mixture 3-2 was fed into Dyno Mill (manufactured by WILLY A. BACHOFEN AG. MASCHINENFABRIK, vessel size: 600 mL, filled with 1,150 g of spherical glass of 1 mm diameter, revolution speed of impeller: 12 m/sec as a peripheral speed) at a rate of 3 L/hr to further grind the clothianidin particles (the resultant mixture is referred to as the mixture 3-3). The clothianidin particles in the

mixture 3-3 had a volume median diameter of 2.5 μm , wherein the net volume of the particles with a diameter of 10 μm or more was 1.8% based on the gross volume of microcapsule particles.

5 To 100 g of the mixture 3-3 was added 21.6 g of a polyisocyanate (Sumidur L-75, adduct of trimethylolpropane and toluenediisocyanate, manufactured by SUMIKA BAYER URETHANE CO., LTD.) at 30°C (the resultant mixture is referred to as the mixture 3-4). The mixture 3-4 was kept
10 at 30°C for 20 hours (the resultant mixture is referred to as the mixture 3-5).

 To 108.8 g of water (deionized water) were added 12.4 g of ethylene glycol and 12.6 g of gum arabic (Arabic Cole SS manufactured by SAN-EI YAKUHIH BOEKI CO., LTD.) to
15 prepare an aqueous phase. All of the aqueous phase and all of the mixture 3-5 were mixed (the resultant mixture is referred to as the mixture 3-6).

 The mixture 3-6 was stirred with a homogenizer (T.K. Autohomomixer, manufactured by PRIMIX CORP., revolution
20 speed: 9,000 rpm) at room temperature to form liquid droplets in water (the resultant mixture is referred to as the mixture 3-7).

 The mixture 3-7 was stirred at 60°C for 24 hours to give an aqueous suspension composition of clothianidin
25 microcapsules (hereinafter, referred to as the aqueous

suspension composition 3).

The resultant microcapsules had a volume median diameter of 22.5 μm , wherein the net volume of the microcapsules with a diameter of 5 μm or less was 7.9% based on the gross volume of microcapsule particles, and the net volume of the microcapsules with a diameter of 50 μm or more was 0.4% based on the gross volume of microcapsule particles.

10 Production Example 4

250 g of clothianidin and 750 g of methyl O-acetylricinoleate (Ricsizer C-101, manufactured by Itoh Oil Chemicals Co., Ltd., content: 95.5%) were mixed (the resultant mixture is referred to as the mixture 4-1). The mixture 4-1 was stirred with a rotor-stator homogenizer (Polytron Homogenizer, manufactured by KINEMATICA AG) to grind clothianidin contained in the mixture 4-1 for about 10 minutes (the resultant mixture is referred to as the mixture 4-2). The clothianidin particles in the mixture 4-2 had a volume median diameter of 0.5 mm.

The mixture 4-2 was fed into Dyno Mill (manufactured by WILLY A. BACHOFEN AG. MASCHINENFABRIK, vessel size: 600 mL, filled with 1,150 g of spherical glass of 1 mm diameter, revolution speed of impeller: 12 m/sec as a peripheral speed) at a rate of 3 L/hr to further grind the

clothianidin particles (the resultant mixture is referred to as the mixture 4-3). The clothianidin particles in the mixture 4-3 had a volume median diameter of 2.5 μm , wherein the net volume of the particles with a diameter of 10 μm or more was 1.8% based on the gross volume of microcapsule particles.

To 100 g of the mixture 4-3 was added 9.8 g of a polyisocyanate (Sumidur L-75, adduct of trimethylolpropane and toluenediisocyanate, manufactured by SUMIKA BAYER URETHANE CO., LTD.) was added at 40°C (the resultant mixture is referred to as the mixture 4-4). The mixture 4-4 was kept at 40°C for 20 hours (the resultant mixture is referred to as the mixture 4-5).

To 108.8 g of water (deionized water) were added 5.6 g of ethylene glycol and 12.6 g of gum arabic (Arabic Cole SS manufactured by SAN-EI YAKUHIN BOEKI CO., LTD.) to prepare an aqueous phase. All of the aqueous phase and all of the mixture 4-5 were mixed (the resultant mixture is referred to as the mixture 4-6).

The mixture 4-6 was stirred with a homogenizer (T.K. Autohomomixer, manufactured by PRIMIX CORP., revolution speed: 9,000 rpm) at room temperature to form liquid droplets in water (the resultant mixture is referred to as the mixture 4-7).

The mixture 4-7 was stirred at 60°C for 24 hours to

give an aqueous suspension composition of clothianidin microcapsules (hereinafter, referred to as the aqueous suspension composition 4).

The resultant microcapsules had a volume median
5 diameter of 19.8 μm , wherein the net volume of the microcapsules with a diameter of 5 μm or less was 9.4% based on the gross volume of microcapsule particles, and the net volume of the microcapsules with a diameter of 50 μm or more was 0.4% based on the gross volume of
10 microcapsule particles.

Production Example 5

250 g of clothianidin and 750 g of methyl O-acetylricinoleate (Ricsizer C-101, manufactured by Itoh Oil
15 Chemicals Co., Ltd., content: 95.5%) were mixed (the resultant mixture is referred to as the mixture 5-1). The mixture 5-1 was stirred with a rotor-stator homogenizer (Polytron Homogenizer, manufactured by KINEMATICA AG) to grind clothianidin contained in the mixture 5-1 for about
20 10 minutes (the resultant mixture is referred to as the mixture 5-2). The clothianidin particles in the mixture 5-2 had a volume median diameter of 0.5 μm .

The mixture 5-2 was fed into Dyno Mill (manufactured by WILLY A. BACHOFEN AG. MASCHINENFABRIK, vessel size: 600
25 mL, filled with 1,150 g of spherical glass of 1 mm diameter,

revolution speed of impeller: 12 m/sec as a peripheral speed) at a rate of 3 L/hr to further grind the clothianidin particles (the resultant mixture is referred to as the mixture 5-3). The clothianidin particles in the mixture 5-3 had a volume median diameter of 2.9 μm , wherein the net volume of the particles with a diameter of 10 μm or more was 3.4% based on the gross volume of microcapsule particles.

To 100 g of the mixture 5-3 was added 21.6 g of a polyisocyanate (Sumidur L-75, adduct of trimethylolpropane and toluenediisocyanate, manufactured by SUMIKA BAYER URETHANE CO., LTD.) at 40°C (the resultant mixture is referred to as the mixture 5-4). The mixture 5-4 was kept at 40°C for 10 hours (the resultant mixture is referred to as the mixture 5-5).

To 108.8 g of water (deionized water) were added 12.4 g of ethylene glycol and 12.6 g of gum arabic (Arabic Cole SS manufactured by SAN-EI YAKUHHIN BOEKI CO., LTD.) to prepare an aqueous phase. All of the aqueous phase and all of the mixture 5-5 were mixed (the resultant mixture is referred to as the mixture 5-6).

The mixture 5-6 was stirred with a homogenizer (T.K. Autohomomixer, manufactured by PRIMIX CORP., revolution speed: 9,000 rpm) at room temperature to form liquid droplets in water (the resultant mixture is referred to as

the mixture 5-7).

The mixture 5-7 was stirred at 60°C for 24 hours to give an aqueous suspension composition of clothianidin microcapsules (hereinafter, referred to as the aqueous suspension composition 5).

The resultant microcapsules had a volume median diameter of 19.6 μm , wherein the net volume of the microcapsules with a diameter of 5 μm or less was 9.1% based on the gross volume of microcapsule particles, and the net volume of the microcapsules with a diameter of 50 μm or more was 0.3% based on the gross volume of microcapsule particles.

Production Example 6

250 g of clothianidin and 750 g of methyl O-acetylricinoleate (Ricsizer C-101, manufactured by Itoh Oil Chemicals Co., Ltd., content: 95.5%) were mixed (the resultant mixture is referred to as the mixture 6-1). The mixture 6-1 was stirred with a rotor-stator homogenizer (Polytron Homogenizer, manufactured by KINEMATICA AG) to grind clothianidin contained in the mixture 6-1 for about 10 minutes (the resultant mixture is referred to as the mixture 6-2). The clothianidin particles in the mixture 6-2 had a volume median diameter of 0.5 mm.

The mixture 6-2 was fed into Dyno Mill (manufactured

by WILLY A. BACHOFEN AG. MASCHINENFABRIK, vessel size: 600 mL, filled with 1,150 g of spherical glass of 1 mm diameter, revolution speed of impeller: 12 m/sec as a peripheral speed) at a rate of 3 L/hr to further grind the

5 clothianidin particles (the resultant mixture is referred to as the mixture 6-3). The clothianidin particles in the mixture 6-3 had a volume median diameter of 2.9 μm , wherein the net volume of the particles with a diameter of 10 μm or more was 3.4% based on the gross volume of microcapsule
10 particles.

To 100 g of the mixture 6-3 was added 21.6 g of a polyisocyanate (Sumidur L-75, adduct of trimethylolpropane and toluenediisocyanate, manufactured by SUMIKA BAYER URETHANE CO., LTD.) at 60°C (the resultant mixture is
15 referred to as the mixture 6-4). The mixture 6-4 was kept at 60°C for 3 hours (the resultant mixture is referred to as the mixture 6-5).

To 108.8 g of water (deionized water) were added 12.4 g of ethylene glycol and 12.6 g of gum arabic (Arabic Cole
20 SS manufactured by SAN-EI YAKUHI BOEKI CO., LTD.) to prepare an aqueous phase. All of the aqueous phase and all of the mixture 6-5 were mixed (the resultant mixture is referred to as the mixture 6-6).

The mixture 6-6 was stirred with a homogenizer (T.K.
25 Autohomomixer, manufactured by PRIMIX CORP., revolution

speed: 9,000 rpm) at room temperature to form liquid droplets in water (the resultant mixture is referred to as the mixture 6-7).

The mixture 6-7 was stirred at 60°C for 24 hours to
5 give an aqueous suspension composition of clothianidin microcapsules (hereinafter, referred to as the aqueous suspension composition 6).

The resultant microcapsules had a volume median diameter of 21.9 μm , wherein the net volume of the
10 microcapsules with a diameter of 5 μm or less was 8.6% based on the gross volume of microcapsule particles, and the net volume of the microcapsules with a diameter of 50 μm or more was 0.1% based on the gross volume of microcapsule particles.

15

Production Example 7

250 g of clothianidin and 750 g of methyl O-acetylricinoleate (Ricsizer C-101, manufactured by Itoh Oil Chemicals Co., Ltd., content: 95.5%) were mixed (the
20 resultant mixture is referred to as the mixture 7-1). The mixture 7-1 was stirred with a rotor-stator homogenizer (Polytron Homogenizer, manufactured by KINEMATICA AG) to grind clothianidin contained in the mixture 7-1 for about 10 minutes (the resultant mixture is referred to as the
25 mixture 7-2). The clothianidin particles in the mixture 7-

2 had a volume median diameter of 0.4 mm.

The mixture 7-2 was fed into Dyno Mill (manufactured by WILLY A. BACHOFEN AG. MASCHINENFABRIK, vessel size: 600 mL, filled with 1,150 g of spherical glass of 1 mm diameter, revolution speed of impeller: 12 m/sec as a peripheral speed) at a rate of 3 L/hr to further grind the clothianidin particles (the resultant mixture is referred to as the mixture 7-3). The clothianidin particles in the mixture 7-3 had a volume median diameter of 2.5 μ m, wherein the net volume of the particles with a diameter of 10 μ m or more was 1.8% based on the gross volume of microcapsule particles.

To 100 g of the mixture 7-3 was added 21.6 g of a polyisocyanate (Sumidur L-75, adduct of trimethylolpropane and toluenediisocyanate, manufactured by SUMIKA BAYER URETHANE CO., LTD.) at 25°C (the resultant mixture is referred to as the mixture 7-4). The mixture 7-4 was kept at 25°C for 20 hours (the resultant mixture is referred to as the mixture 7-5).

To 108.8 g of water (deionized water) were added 12.4 g of ethylene glycol and 12.6 g of gum arabic (Arabic Cole SS manufactured by SAN-EI YAKUHIH BOEKI CO., LTD.) to prepare an aqueous phase. All of the aqueous phase and all of the mixture 7-5 were mixed (the resultant mixture is referred to as the mixture 7-6).

The mixture 7-6 was stirred with a homogenizer (T.K. Autohomomixer, manufactured by PRIMIX CORP., revolution speed: 11,000 rpm) at room temperature to form liquid droplets in water (the resultant mixture is referred to as the mixture 7-7).

The mixture 7-7 was stirred at 60°C for 24 hours to give an aqueous suspension composition of clothianidin microcapsules (hereinafter, referred to as the aqueous suspension composition 7).

The resultant microcapsules had a volume median diameter of 13.6 μm , wherein the net volume of the microcapsules with a diameter of 5 μm or less was 15.9% based on the gross volume of microcapsule particles, and the net volume of the microcapsules with a diameter of 50 μm or more was 0% based on the gross volume of microcapsule particles.

Production Example 8

250 g of clothianidin and 750 g of methyl O-acetylricinoleate (Ricsizer C-101, manufactured by Itoh Oil Chemicals Co., Ltd., content: 95.5%) were mixed (the resultant mixture is referred to as the mixture 8-1). The mixture 8-1 was stirred with a rotor-stator homogenizer (Polytron Homogenizer, manufactured by KINEMATICA AG) to grind clothianidin contained in the mixture 8-1 for about

10 minutes (the resultant mixture is referred to as the mixture 8-2). The clothianidin particles in the mixture 8-2 had a volume median diameter of 0.4 mm.

The mixture 8-2 was fed into Dyno Mill (manufactured by WILLY A. BACHOFEN AG. MASCHINENFABRIK, vessel size: 600 mL, filled with 1,150 g of spherical glass of 1 mm diameter, revolution speed of impeller: 12 m/sec as a peripheral speed) at a rate of 3 L/hr to further grind the clothianidin particles (the resultant mixture is referred to as the mixture 8-3). The clothianidin particles in the mixture 8-3 had a volume median diameter of 2.5 μ m, wherein the net volume of the particles with a diameter of 10 μ m or more was 1.8% based on the gross volume of microcapsule particles.

To 100 g of the mixture 8-3 was added 21.6 g of a polyisocyanate (Sumidur L-75, adduct of trimethylolpropane and toluenediisocyanate, manufactured by SUMIKA BAYER URETHANE CO., LTD.) at 25°C (the resultant mixture is referred to as the mixture 8-4). The mixture 8-4 was kept at 25°C for 20 hours (the resultant mixture is referred to as the mixture 8-5).

To 108.8 g of water (deionized water) were added 12.4 g of ethylene glycol and 12.6 g of gum arabic (Arabic Cole SS manufactured by SAN-EI YAKUHIH BOEKI CO., LTD.) to prepare an aqueous phase. All of the aqueous phase and all

of the mixture 8-5 were mixed (the resultant mixture is referred to as the mixture 8-6).

The mixture 8-6 was stirred with a homogenizer (T.K. Autohomomixer, manufactured by PRIMIX CORP., revolution
5 speed: 7,000 rpm) at room temperature to form liquid droplets in water (the resultant mixture is referred to as the mixture 8-7).

The mixture 8-7 was stirred at 60°C for 24 hours to give an aqueous suspension composition of clothianidin
10 microcapsules (hereinafter, referred to as the aqueous suspension composition 8).

The resultant microcapsules had a volume median diameter of 34.9 μm , wherein the net volume of the microcapsules with a diameter of 5 μm or less was 0% based
15 on the gross volume of microcapsule particles, and the net volume of the microcapsules with a particle diameter of 50 μm or more was 16.9% based on the gross volume of microcapsule particles.

20 Production Example 9

375 g of clothianidin and 750 g of methyl O-acetylricinoleate (Ricsizer C-101, manufactured by Itoh Oil Chemicals Co., Ltd., content: 95.5%) were mixed (the
resultant mixture is referred to as the mixture 9-1). The
25 mixture 9-1 was stirred with a rotor-stator homogenizer

(Polytron Homogenizer, manufactured by KINEMATICA AG) to grind clothianidin contained in the mixture 9-1 for about 10 minutes (the resultant mixture is referred to as the mixture 9-2). The clothianidin particles in the mixture 9-2 had a volume median diameter of 0.6 mm.

The mixture 9-2 was fed into Dyno Mill (manufactured by WILLY A. BACHOFEN AG. MASCHINENFABRIK, vessel size: 600 mL, filled with 1,150 g of spherical glass of 1 mm diameter, revolution speed of impeller: 12 m/sec as a peripheral speed) at a rate of 3 L/hr to further grind the clothianidin particles (the resultant mixture is referred to as the mixture 9-3). The clothianidin particles in the mixture 9-3 had a volume median diameter of 2.0 μm , wherein the net volume of the particles with a diameter of 10 μm or more was 0% based on the gross volume of microcapsule particles.

To 100 g of the mixture 9-3 was added 21.6 g of a polyisocyanate (Sumidur L-75, adduct of trimethylolpropane and toluenediisocyanate, manufactured by SUMIKA BAYER URETHANE CO., LTD.) at 25°C (the resultant mixture is referred to as the mixture 9-4). The mixture 9-4 was kept at 25°C for 20 hours (the resultant mixture is referred to as the mixture 9-5).

To 108.8 g of water (deionized water) were added 12.4 g of ethylene glycol and 12.6 g of gum arabic (Arabic Cole

SS manufactured by SAN-EI YAKUHIN BOEKI CO., LTD.) to prepare an aqueous phase. All of the aqueous phase and all of the mixture 9-5 were mixed (the resultant mixture is referred to as the mixture 9-6).

5 The mixture 9-6 was stirred with a homogenizer (T.K. Autohomomixer, manufactured by PRIMIX CORP., revolution speed: 9,000 rpm) at room temperature to form liquid droplets in water (the resultant mixture is referred to as the mixture 9-7).

10 The mixture 9-7 was stirred at 60°C for 24 hours to give an aqueous suspension composition of clothianidin microcapsules (hereinafter, referred to as the an aqueous suspension composition 9).

15 The resultant microcapsules had a volume median diameter of 18.4 μm , wherein the net volume of the microcapsules with a particle diameter of 5 μm or less was 11.2% based on the gross volume of microcapsule particles, and the net volume of the microcapsules with a particle diameter of 50 μm or more was 0% based on the gross volume
20 of microcapsule particles.

Production Example 10

150 g of clothianidin and 750 g of methyl O-acetylricinoleate (Ricsizer C-101, manufactured by Itoh Oil
25 Chemicals Co., Ltd., content: 95.5%) were mixed (the

resultant mixture is referred to as the mixture 10-1). The mixture 10-1 was stirred with a rotor-stator homogenizer (Polytron Homogenizer, manufactured by KINEMATICA AG) to grind clothianidin contained in the mixture 10-1 for about 10 minutes (the resultant mixture is referred to as the mixture 10-2). The clothianidin particles in the mixture 10-2 had a volume median diameter of 0.4 mm.

The mixture 10-2 was fed into Dyno Mill (manufactured by WILLY A. BACHOFEN AG. MASCHINENFABRIK, vessel size: 600 mL, filled with 1,150 g of spherical glass of 1 mm diameter, revolution speed of impeller: 12 m/sec as a peripheral speed) at a rate of 3 L/hr to further grind the clothianidin particles (the resultant mixture is referred to as the mixture 10-3). The clothianidin particles in the mixture 10-3 had a volume median diameter of 2.9 μ m, wherein the net volume of the particles with a diameter of 10 μ m or more was 3.3% based on the gross volume of microcapsule particles.

To 100 g of the mixture 10-3 was added 21.6 g of a polyisocyanate (Sumidur L-75, adduct of trimethylolpropane and toluenediisocyanate, manufactured by SUMIKA BAYER URETHANE CO., LTD.) at 25°C (the resultant mixture is referred to as the mixture 10-4). The mixture 10-4 was kept at 25°C for 20 hours (the resultant mixture is referred to as the mixture 10-5).

To 108.8 g of water (deionized water) was added 12.4 g of ethylene glycol and 12.6 g of gum arabic (Arabic Cole SS manufactured by SAN-EI YAKUHIH BOEKI CO., LTD.) to prepare an aqueous phase. All of the aqueous phase and all of the mixture 10-5 were mixed (the resultant mixture is referred to as the mixture 10-6).

The mixture 10-6 was stirred with a homogenizer (T.K. Autohomomixer, manufactured by PRIMIX CORP., revolution speed: 9,000 rpm) at room temperature to form liquid droplets in water (the resultant mixture is referred to as the mixture 10-7).

The mixture 10-7 was stirred at 60°C for 24 hours to give an aqueous suspension composition of clothianidin microcapsules (hereinafter, referred to as the aqueous suspension composition 10).

The resultant microcapsules had a volume median diameter of 19.9 μm , wherein the net volume of the microcapsules with a particle diameter of 5 μm or less was 9.4% based on the gross volume of microcapsule particles, and the net volume of the microcapsules with a particle diameter of 50 μm or more was 0% based on the gross volume of microcapsule particles.

Production Example 11

25 250 g of clothianidin and 750 g of methyl O-

acetylricinoleate (Ricsizer C-101, manufactured by Itoh Oil Chemicals Co., Ltd., content: 95.5%) were mixed (the resultant mixture is referred to as the mixture 11-1). The mixture 11-1 was stirred with a rotor-stator homogenizer (Polytron Homogenizer, manufactured by KINEMATICA AG) to grind clothianidin contained in the mixture 11-1 for about 10 minutes (the resultant mixture is referred to as the mixture 11-2). The clothianidin particles in the mixture 11-2 had a volume median diameter of 0.5 mm.

The mixture 11-2 was fed into Dyno Mill (manufactured by WILLY A. BACHOFEN AG. MASCHINENFABRIK, vessel size: 600 mL, filled with 1,150 g of spherical glass of 1 mm diameter, revolution speed of impeller: 12 m/sec as a peripheral speed) at a rate of 3 L/hr to further grind the clothianidin particles (the resultant mixture is referred to as the mixture 11-3). The clothianidin particles in the mixture 11-3 had a volume median diameter of 2.9 μ m, wherein the net volume of the particles with a diameter of 10 μ m or more was 3.3% based on the gross volume of microcapsule particles.

To 100 g of the mixture 11-3 was added 21.6 g of a polyisocyanate (Sumidur L-75, adduct of trimethylolpropane and toluenediisocyanate, manufactured by SUMIKA BAYER URETHANE CO., LTD.) at 25°C (the resultant mixture is referred to as the mixture 11-4). The mixture 11-4 was

kept at 25°C for 20 hours (the resultant mixture is referred to as the mixture 11-5).

To 108.8 g of water (deionized water) were added 12.4 g of ethylene glycol and 12.6 g of gum arabic (Arabic Cole
5 SS manufactured by SAN-EI YAKUHIH BOEKI CO., LTD.) to prepare an aqueous phase. All of the aqueous phase and all of the mixture 11-5 were mixed (the resultant mixture is referred to as the mixture 11-6).

The mixture 11-6 was stirred with a homogenizer (T.K.
10 Autohomomixer, manufactured by PRIMIX CORP., revolution speed: 9,000 rpm) at room temperature to form liquid droplets in water (the resultant mixture is referred to as the mixture 11-7).

The mixture 11-7 was stirred at 60°C for 24 hours to
15 give an aqueous suspension composition of clothianidin microcapsules (hereinafter, referred to as the aqueous suspension composition 11).

The resultant microcapsules had a volume median diameter of 19.9 μm , wherein the net volume of the
20 microcapsules with a particle diameter of 5 μm or less was 9.4% based on the gross volume of microcapsule particles, and the net volume of the microcapsules with a particle diameter of 50 μm or more was 0% based on the gross volume of microcapsule particles.

25 The aqueous suspension composition 11 was mixed with

an aqueous solution prepared by mixing 68.05 g of water,
0.05 g of xanthan gum, 0.1 g of aluminum magnesium silicate,
0.5 g of disodium hydrogen phosphate and 0.1 g of Biohope L
(preservative, manufactured by K.I. CHEMICAL INDUSTRY CO.,
5 LTD.) to give an aqueous suspension formulation of
microcapsules. The aqueous suspension formulation had pH
7.9.

Production Example 12

10 250 g of clothianidin and 750 g of methyl O-
acetylricinoleate (Ricsizer C-101, manufactured by Itoh Oil
Chemicals Co., Ltd., content: 95.5%) were mixed (the
resultant mixture is referred to as the mixture 12-1). The
mixture 12-1 was stirred with a rotor-stator homogenizer
15 (Polytron Homogenizer, manufactured by KINEMATICA AG) to
grind clothianidin contained in the mixture 12-1 for about
10 minutes (the resultant mixture is referred to as the
mixture 12-2). The clothianidin particles in the mixture
12-2 had a volume median diameter of 0.5 mm.

20 The mixture 12-2 was fed into Dyno Mill (manufactured
by WILLY A. BACHOFEN AG. MASCHINENFABRIK, vessel size: 600
mL, filled with 1,150 g of spherical glass of 1 mm diameter,
revolution speed of impeller: 12 m/sec as a peripheral
speed) at a rate of 3 L/hr to further grind the
25 clothianidin particles (the resultant mixture is referred

to as the mixture 12-3). The clothianidin particles in the mixture 12-3 had a volume median diameter of 2.9 μm , wherein the net volume of the particles with a diameter of 10 μm or more was 3.3% based on the gross volume of microcapsule particles.

To 100 g of the mixture 12-3 was added 21.6 g of a polyisocyanate (Sumidur L-75, adduct of trimethylolpropane and toluenediisocyanate, manufactured by SUMIKA BAYER URETHANE CO., LTD.) at 25°C (the resultant mixture is referred to as the mixture 12-4). The mixture 12-4 was kept at 25°C for 20 hours (the resultant mixture is referred to as the mixture 12-5).

To 108.8 g of water (deionized water) were added 12.4 g of ethylene glycol and 12.6 g of gum arabic (Arabic Cole SS manufactured by SAN-EI YAKUHIH BOEKI CO., LTD.) to prepare an aqueous phase. All of the aqueous phase and all of the mixture 12-5 were mixed (the resultant mixture is referred to as the mixture 12-6).

The mixture 12-6 was stirred with a homogenizer (T.K. Autohomomixer, manufactured by PRIMIX CORP., revolution speed: 9,000 rpm) at room temperature to form liquid droplets in water (the resultant mixture is referred to as the mixture 12-7).

The mixture 12-7 was stirred at 60°C for 24 hours to give an aqueous suspension composition of clothianidin

microcapsules (hereinafter, referred to as the aqueous suspension composition 12).

The resultant microcapsules had a volume median diameter of 19.9 μm , wherein the net volume of the microcapsules with a particle diameter of 5 μm or less was 9.4% based on the gross volume of microcapsule particles, and the net volume of the microcapsules with a particle diameter of 50 μm or more was 0% based on the gross volume of microcapsule particles.

The aqueous suspension composition 12 was mixed with an aqueous solution prepared by mixing 68.55 g of water, 0.05 g of xanthan gum, 0.1 g of aluminum magnesium silicate, 0.1 g of sodium hydroxide and 0.1 g of Biohope L (preservative, manufactured by K.I. CHEMICAL INDUSTRY CO., LTD.) to give an aqueous suspension formulation of microcapsules. The aqueous suspension formulation had pH 7.9.

Production Example 13

250 g of clothianidin and 750 g of methyl O-acetylricinoleate (Ricsizer C-101, manufactured by Itoh Oil Chemicals Co., Ltd., content: 95.5%) were mixed (the resultant mixture is referred to as the mixture 13-1). The mixture 13-1 was stirred with a rotor-stator homogenizer (Polytron Homogenizer, manufactured by KINEMATICA AG) to

grind clothianidin contained in the mixture 13-1 for about 10 minutes (the resultant mixture is referred to as the mixture 13-2). The clothianidin particles in the mixture 13-2 had a volume median diameter of 0.5 mm.

5 The mixture 13-2 was fed into Dyno Mill (manufactured by WILLY A. BACHOFEN AG. MASCHINENFABRIK, vessel size: 600 mL, filled with 1,150 g of spherical glass of 1 mm diameter, revolution speed of impeller: 12 m/sec as a peripheral speed) at a rate of 3 L/hr to further grind the
10 clothianidin particles (the resultant mixture is referred to as the mixture 13-3). The clothianidin particles in the mixture 13-3 had a volume median diameter of 2.9 μ m, wherein the net volume of the particles with a diameter of 10 μ m or more was 3.3% based on the gross volume of
15 microcapsule particles.

To 100 g of the mixture 13-3 was added 21.6 g of a polyisocyanate (Sumidur L-75, adduct of trimethylolpropane and toluenediisocyanate, manufactured by SUMIKA BAYER URETHANE CO., LTD.) at 25°C (the resultant mixture is
20 referred to as the mixture 13-4). The mixture 13-4 was kept at 25°C for 20 hours (the resultant mixture is referred to as the mixture 13-5).

To 108.8 g of water (deionized water) were added 12.4 g of ethylene glycol and 12.6 g of gum arabic (Arabic Cole
25 SS manufactured by SAN-EI YAKUHIN BOEKI CO., LTD.) to

prepare an aqueous phase. All of the aqueous phase and all of the mixture 13-5 were mixed (the resultant mixture is referred to as the mixture 13-6).

5 The mixture 13-6 was stirred with a homogenizer (T.K. Autohomomixer, manufactured by PRIMIX CORP., revolution speed: 9,000 rpm) at room temperature to form liquid droplets in water (the resultant mixture is referred to as the mixture 13-7).

10 The mixture 13-7 was stirred at 60°C for 24 hours to give an aqueous suspension composition of clothianidin microcapsules (hereinafter, referred to as the aqueous suspension composition 13).

The resultant microcapsules had a volume median diameter of 19.9 μm , wherein the net volume of the microcapsules with a particle diameter of 5 μm or less was 9.4% based on the gross volume of microcapsule particles, and the net volume of the microcapsules with a particle diameter of 50 μm or more was 0% based on the gross volume of microcapsule particles.

20 The aqueous suspension composition 13 was mixed with an aqueous solution prepared by mixing 68.05 g of water, 0.05 g of xanthan gum, 0.1 g of aluminum magnesium silicate, 0.5 g of dipotassium hydrogen phosphate and 0.1 g of Biohope L (preservative, manufactured by K.I. CHEMICAL
25 INDUSTRY CO., LTD.) to give an aqueous suspension

formulation of microcapsules. The aqueous suspension formulation had pH 7.2.

Production Example 14

5 250 g of clothianidin and 750 g of methyl O-acetylricinoleate (Ricsizer C-101, manufactured by Itoh Oil Chemicals Co., Ltd., content: 95.5%) were mixed (the resultant mixture is referred to as the mixture 14-1). The mixture 14-1 was stirred with a rotor-stator homogenizer
10 (Polytron Homogenizer, manufactured by KINEMATICA AG) to grind clothianidin contained in the mixture 14-1 for about 10 minutes (the resultant mixture is referred to as the mixture 14-2). The clothianidin particles in the mixture 14-2 had a volume median diameter of 0.5 mm.

15 The mixture 14-2 was fed into Dyno Mill (manufactured by WILLY A. BACHOFEN AG. MASCHINENFABRIK, vessel size: 600 mL, filled with 1,150 g of spherical glass of 1 mm diameter, revolution speed of impeller: 12 m/sec as a peripheral speed) at a rate of 3 L/hr to further grind the
20 clothianidin particles (the resultant mixture is referred to as the mixture 14-3). The clothianidin particles in the mixture 14-3 had a volume median diameter of 2.9 μm , wherein the net volume of the particles with a diameter of 10 μm or more was 3.3% based on the gross volume of
25 microcapsule particles.

To 100 g of the mixture 14-3 was added 21.6 g of a polyisocyanate (Sumidur L-75, adduct of trimethylolpropane and toluenediisocyanate, manufactured by SUMIKA BAYER URETHANE CO., LTD.) at 25°C (the resultant mixture is referred to as the mixture 14-4). The mixture 14-4 was kept at 25°C for 20 hours (the resultant mixture is referred to as the mixture 14-5).

To 108.8 g of water (deionized water) were added 12.4 g of ethylene glycol and 12.6 g of gum arabic (Arabic Cole SS manufactured by SAN-EI YAKUHI BOEKI CO., LTD.) to prepare an aqueous phase. All of the aqueous phase and all of the mixture 14-5 were mixed (the resultant mixture is referred to as the mixture 14-6).

The mixture 14-6 was stirred with a homogenizer (T.K. Autohomomixer, manufactured by PRIMIX CORP., revolution speed: 9,000 rpm) at room temperature to form liquid droplets in water (the resultant mixture is referred to as the mixture 14-7).

The mixture 14-7 was stirred at 60°C for 24 hours to give an aqueous suspension composition of clothianidin microcapsules (hereinafter, referred to as the aqueous suspension composition 14).

The resultant microcapsules has a volume median diameter of 19.9 μm , wherein the net volume of the microcapsules with a particle diameter of 5 μm or less was

9.4% based on the gross volume of microcapsule particles, and the net volume of the microcapsules with a particle diameter of 50 μm or more was 0% based on the gross volume of microcapsule particles.

5 The aqueous suspension composition 14 was mixed with an aqueous solution prepared by mixing 68.25 g of water, 0.05 g of xanthan gum, 0.1 g of aluminum magnesium silicate, 0.3 g of disodium hydrogen phosphate and 0.1 g of Biohope L (preservative, manufactured by K.I. CHEMICAL INDUSTRY CO., LTD.) to give an aqueous suspension formulation of
10 microcapsules. The aqueous suspension formulation had pH 6.2.

Production Example 15

15 250 g of clothianidin and 750 g of methyl O-acetylricinoleate (Ricsizer C-101, manufactured by Itoh Oil Chemicals Co., Ltd., content: 95.5%) were mixed (the resultant mixture is referred to as the mixture 15-1). The mixture 15-1 was stirred with a rotor-stator homogenizer
20 (Polytron Homogenizer, manufactured by KINEMATICA AG) to grind clothianidin contained in the mixture 15-1 for about 10 minutes (the resultant mixture is referred to as the mixture 15-2). The clothianidin particles in the mixture 15-2 had a volume median diameter of 0.5 μm .

25 The mixture 15-2 was fed into Dyno Mill (manufactured

by WILLY A. BACHOFEN AG. MASCHINENFABRIK, vessel size: 600 mL, filled with 1,150 g of spherical glass of 1 mm diameter, revolution speed of impeller: 12 m/sec as a peripheral speed) at a rate of 3 L/hr to further grind the clothianidin particles (the resultant mixture is referred to as the mixture 15-3). The clothianidin particles in the mixture 15-3 had a volume median diameter of 2.9 μ m, wherein the net volume of the particles with a diameter of 10 μ m or more was 3.3% based on the gross volume of microcapsule particles.

To 100 g of the mixture 15-3 was added 21.6 g of a polyisocyanate (Sumidur L-75, adduct of trimethylolpropane and toluenediisocyanate, manufactured by SUMIKA BAYER URETHANE CO., LTD.) at 25°C (the resultant mixture is referred to as the mixture 15-4). The mixture 15-4 was kept at 25°C for 20 hours (the resultant mixture is referred to as the mixture 15-5).

To 108.8 g of water (deionized water) were added 12.4 g of ethylene glycol and 12.6 g of gum arabic (Arabic Cole SS manufactured by SAN-EI YAKUHIN BOEKI CO., LTD.) to prepare an aqueous phase. All of the aqueous phase and all of the mixture 15-5 were mixed (the resultant mixture is referred to as the mixture 15-6).

The mixture 15-6 was stirred with a homogenizer (T.K. Autohomomixer, manufactured by PRIMIX CORP., revolution

speed: 9,000 rpm) at room temperature to form liquid droplets in water (the resultant mixture is referred to as the mixture 15-7).

The mixture 15-7 was stirred at 60°C for 24 hours to give an aqueous suspension composition of clothianidin microcapsules (hereinafter, referred to as the aqueous suspension composition 15).

The resultant microcapsules had a volume median diameter of 19.9 μm , wherein the net volume of the microcapsules with a particle diameter of 5 μm or less was 9.4% based on the gross volume of microcapsule particles, and the net volume of the microcapsules with a particle diameter of 50 μm or more was 0% based on the gross volume of microcapsule particles.

The aqueous suspension composition 15 was mixed with an aqueous solution prepared by mixing 68.35 g of water, 0.05 g of xanthan gum, 0.1 g of aluminum magnesium silicate and 0.2 g of Proxel GXL (preservative, manufactured by Avecia K.K.) to give an aqueous suspension formulation of microcapsules. The aqueous suspension formulation had pH 7.4.

Comparative Production Example 1

An aqueous suspension composition (hereinafter, referred to as the comparative aqueous suspension

composition 1) was produced in the same manner as in Production Example 1 except that the mixture 1-4 was kept at 20°C for 1 hour.

5 Comparative Production Example 2

An aqueous suspension composition (hereinafter, referred to as the comparative aqueous suspension composition 2) was produced in the same manner as in Production Example 5 except that the mixture 5-4 was kept
10 at 40°C for 1 hour.

Comparative Production Example 3

An aqueous suspension composition (hereinafter, referred to as the comparative aqueous suspension
15 composition 3) was produced in the same manner as in Production Example 1 except that the mixture 1-4 was kept at 50°C for 1 hour.

Comparative Production Example 4

20 An aqueous suspension composition (hereinafter, referred to as the comparative aqueous suspension composition 4) was produced in the same manner as in Production Example 1 except that the mixture 1-4 was kept at 60°C for 0.5 hour.

Test Example 1

A mixture of 0.5 g of each of the aqueous suspension compositions shown in the following Table 1 with 249.5 g of water was allowed to stand at room temperature for 2 hours.

5 Then, the mixture was centrifuged at 3,000 rpm for 15 minutes. About 1 mL of the supernatant was separated, and 10 μ L out of the 1 mL was subjected to high performance liquid chromatography to analyze the amount of clothianidin. Based on the analysis value thus obtained, the amount of
10 clothianidin contained in the supernatant and the amount of clothianidin contained in a microcapsule were calculated. The residual rate of clothianidin contained in microcapsule based on the amount of clothianidin contained initially in the microcapsule is shown in Table 1.

15 [Table 1]

	Residual rate of clothianidin contained in microcapsule (%)
Aqueous suspension composition 1	79
Aqueous suspension composition 2	78
Aqueous suspension composition 3	79
Aqueous suspension composition 4	91
Aqueous suspension composition 5	82
Aqueous suspension composition 6	76
Comparative aqueous suspension composition 1	39
Comparative aqueous suspension composition 2	41
Comparative aqueous suspension composition 3	45
Comparative aqueous suspension composition 4	38

Test Example 2

Each of the aqueous suspension compositions shown in the following Table 2 was diluted 200 times with water.

Then, 0.4 mL of the dilution was spread over a glass petri dish of 6 cm diameter. Then, the dilution was air dried at room temperature. The petri dish was exposed to sunlight for 250 hours (cumulative illuminance: 1810 Lx).

To the petri dish was added 10 mL of acetonitrile and then stirred. Then, 10 μ L of the mixture was subjected to high performance liquid chromatography to analyze the amount of clothianidin. Based on the analysis value thus obtained, the amount of clothianidin remaining in the petri dish was calculated. The residual rate of clothianidin based on the amount of clothianidin contained in the dilution initially spread over the petri dish is shown in Table 2.

[Table 2]

	Residual rate of clothianidin (%)
Aqueous suspension composition 1	79
Aqueous suspension composition 2	81
Aqueous suspension composition 3	86
Aqueous suspension composition 4	89
Aqueous suspension composition 5	82
Comparative aqueous suspension composition 1	54
Comparative aqueous suspension composition 3	61

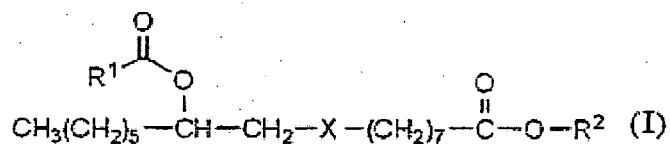
Industrial Applicability

According to the method for producing a microcapsule of the present invention, it is possible to produce a microcapsule with the delayed release timing of a pesticidal compound contained therein as compared to a conventional microcapsule.

CLAIMS

1. A method for producing a microcapsule, which comprises:

- 5 (1) keeping a mixture of a pesticidal compound, a compound represented by formula (I) and a polyisocyanate at 20 to 60°C for 3 hours or more:



wherein X represents $-\text{CH}_2-\text{CH}_2-$ or $-\text{CH}=\text{CH}-$, R^1 represents a
10 C1-C4 alkyl group, and R^2 represents a C1-C4 alkyl group;

(2) then adding the mixture to water containing a polyol or a polyamine, and producing prepare liquid droplets in the water; and

(3) forming a film of polyurethane or polyurea around
15 the droplets.

2. The method according to claim 1, wherein the weight ratio of the pesticidal compound to the compound represented by formula (I) is from 10:100 to 100:100.

20

3. The method according to claim 1, wherein the compound represented by formula (I) is a C1-C4 alkyl ester of O-acetylricinoleic acid.

4. The method according to claim 1, wherein the compound represented by formula (I) is methyl O-acetylricinoleate.

5

5. The method according to claim 1, wherein the pesticidal compound is a solid pesticidal compound.

6. The method according to claim 1, wherein the
10 pesticidal compound is a neonicotinoid compound.

7. The method according to claim 1, wherein the pesticidal compound is clothianidin.

15 8. A microcapsule produced by the method according to claim 1.

INTERNATIONAL SEARCH REPORT

International application No.

PCT/JP2010/059489

A. CLASSIFICATION OF SUBJECT MATTER

Int.Cl. A01N25/28 (2006.01) i, A01N51/00 (2006.01) i, A01P7/04 (2006.01) i,
B01J13/16 (2006.01) i

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)

Int.Cl. A01N25/28, A01N51/00, A01P7/04, B01J13/16

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

Published examined utility model applications of Japan 1922-1996
Published unexamined utility model applications of Japan 1971-2010
Registered utility model specifications of Japan 1996-2010
Published registered utility model applications of Japan 1994-2010

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

CAplus (STN), REGISTRY (STN)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	WO 2007/069461 A1 (Sumitomo Chemical Co., Ltd.) 2007.06.21, Claims, Examples & JP 2007-186497 A & US 2009/0142406 A1 & EP 1961303 A1	1-8
A	JP 10-7505 A (Aritsune Yakuhin Kogyo Kabushiki Kaisha) 1998.01.13, Claim 5 (Family: none)	1-8
A	JP 57-24303 A (Mitsui Toatsu Chemical, Inc.) 1982.02.08, Table 1, No.5 (Family: none)	1-8



Further documents are listed in the continuation of Box C.



See patent family annex.

* Special categories of cited documents:

"A" document defining the general state of the art which is not considered to be of particular relevance

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"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)

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"P" document published prior to the international filing date but later than the priority date claimed

"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

"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

"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

"&" document member of the same patent family

Date of the actual completion of the international search

21.06.2010

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INTERNATIONAL SEARCH REPORT

International application No.

PCT/JP2010/059489

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT		
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	JP 50-116638 A (Fumakilla Co., Ltd.) 1975.09.12, Page 2, lower left column (3) (Family: none)	1-8
A	JP 2009-62292 A (Sumitomo Chemical Co., Ltd.) 2009.03.26, Claims, Examples & US 2009/0060966 A1	1-8
A	JP 6-256116 A (Sumitomo Chemical Co., Ltd.) 1994.09.13, Full text & BR 9400630 A	1-8