

PATENT COOPERATION TREATY

From the
INTERNATIONAL SEARCHING AUTHORITY

PCT

To:

see form PCT/ISA/220

WRITTEN OPINION OF THE INTERNATIONAL SEARCHING AUTHORITY (PCT Rule 43*bis*.1)

Date of mailing
(day/month/year) see form PCT/ISA/210 (second sheet)

Applicant's or agent's file reference
see form PCT/ISA/220

FOR FURTHER ACTION
See paragraph 2 below

International application No.
PCT/EP2017/065541

International filing date (day/month/year)
23.06.2017

Priority date (day/month/year)
30.06.2016

International Patent Classification (IPC) or both national classification and IPC
INV. A01N25/28 A01N53/00

Applicant
SYNGENTA PARTICIPATIONS AG

1. This opinion contains indications relating to the following items:

- ☒ Box No. I Basis of the opinion
- ☐ Box No. II Priority
- ☐ Box No. III Non-establishment of opinion with regard to novelty, inventive step and industrial applicability
- ☐ Box No. IV Lack of unity of invention
- ☒ Box No. V Reasoned statement under Rule 43*bis*.1(a)(i) with regard to novelty, inventive step and industrial applicability; citations and explanations supporting such statement
- ☐ Box No. VI Certain documents cited
- ☒ Box No. VII Certain defects in the international application
- ☒ Box No. VIII Certain observations on the international application

2. FURTHER ACTION

If a demand for international preliminary examination is made, this opinion will usually be considered to be a written opinion of the International Preliminary Examining Authority ("IPEA") except that this does not apply where the applicant chooses an Authority other than this one to be the IPEA and the chosen IPEA has notified the International Bureau under Rule 66.1*bis*(b) that written opinions of this International Searching Authority will not be so considered.

If this opinion is, as provided above, considered to be a written opinion of the IPEA, the applicant is invited to submit to the IPEA a written reply together, where appropriate, with amendments, before the expiration of 3 months from the date of mailing of Form PCT/ISA/220 or before the expiration of 22 months from the priority date, whichever expires later.

For further options, see Form PCT/ISA/220.

Name and mailing address of the ISA:



European Patent Office
D-80298 Munich
Tel. +49 89 2399 - 0
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Date of completion of
this opinion

see form
PCT/ISA/210

Authorized Officer

Butkowskyj-Walkiw, T
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Box No. I Basis of the opinion

1. With regard to the **language**, this opinion has been established on the basis of:
 - ☒ the international application in the language in which it was filed.
 - ☐ a translation of the international application into , which is the language of a translation furnished for the purposes of international search (Rules 12.3(a) and 23.1 (b)).
2. ☐ This opinion has been established taking into account the **rectification of an obvious mistake** authorized by or notified to this Authority under Rule 91 (Rule 43bis.1(a))
3. ☐ With regard to any **nucleotide and/or amino acid sequence** disclosed in the international application, this opinion has been established on the basis of a sequence listing:
 - a. ☐ forming part of the international application as filed:
 - ☐ in the form of an Annex C/ST.25 text file.
 - ☐ on paper or in the form of an image file.
 - b. ☐ furnished together with the international application under PCT Rule 13ter.1(a) for the purposes of international search only in the form of an Annex C/ST.25 text file.
 - c. ☐ furnished subsequent to the international filing date for the purposes of international search only:
 - ☐ in the form of an Annex C/ST.25 text file (Rule 13ter.1(a)).
 - ☐ on paper or in the form of an image file (Rule 13ter.1(b) and Administrative Instructions, Section 713).
4. ☐ In addition, in the case that more than one version or copy of a sequence listing has been filed or furnished, the required statements that the information in the subsequent or additional copies is identical to that forming part of the application as filed or does not go beyond the application as filed, as appropriate, were furnished.
5. Additional comments:

Box No. V Reasoned statement under Rule 43bis.1(a)(i) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

1. Statement

Novelty (N)	Yes: Claims	<u>1-10</u>
	No: Claims	
Inventive step (IS)	Yes: Claims	
	No: Claims	<u>1-10</u>
Industrial applicability (IA)	Yes: Claims	<u>1-10</u>
	No: Claims	

2. Citations and explanations

see separate sheet

Box No. VII Certain defects in the international application

The following defects in the form or contents of the international application have been noted:

see separate sheet

Box No. VIII Certain observations on the international application

The following observations on the clarity of the claims, description, and drawings or on the question whether the claims are fully supported by the description, are made:

see separate sheet

Reference is made to the following documents:

- D1 US 2013/115261 A1 (BORZATTA VALERIO [IT] ET AL) 9 May 2013
(2013-05-09)
- D2 US 6 133 197 A (CHEN JIN-LING [US] ET AL) 17 October 2000
(2000-10-17)
- D3 US 4 285 720 A (SCHER HERBERT B) 25 August 1981 (1981-08-25)

Item V

In the light of the documents cited in the search report the subject-matter of claims 1-10 has to be considered as being novel (Art. 33(2) PCT) as none of these documents discloses a microcapsule having a polyurea wall in the claimed range in combination with the claimed polymer content and amount of pyrethroid insecticide.

However, the subject-matter of claims 1-10 is open to objection as to lack of inventive step (Art. 33(3) PCT) as the object of the present application, namely to provide microcapsules encapsulating pyrethroids having an improved toxicology profile whilst maintaining effective pest control activity and the present solution have already been suggested by D1 (paragraphs [0060] - [0068]; claims; example 10) as well as by a combination of D2 (abstract; column 4, line 59 - column 6, line 67; claims; examples) with D3 (column 3, line 51 - column 8, line 52; claims; examples).

Item VII

Contrary to the requirements of Rule 5.1(a)(ii) PCT, the relevant background art disclosed in D1, D2 and D3 is not mentioned in the description, nor are these documents identified therein.

Item VIII

Optional features like "preferably" have no limiting effect on the scope of the claim and thus the feature following any such expression is to be regarded as entirely optional.

PATENT COOPERATION TREATY

PCT

INTERNATIONAL SEARCH REPORT

(PCT Article 18 and Rules 43 and 44)

Applicant's or agent's file reference 81080-WO-ORG-P1	FOR FURTHER ACTION see Form PCT/ISA/220 as well as, where applicable, item 5 below.	
International application No. PCT/EP2017/065541	International filing date (<i>day/month/year</i>) 23 June 2017 (23-06-2017)	(Earliest) Priority Date (<i>day/month/year</i>) 30 June 2016 (30-06-2016)
Applicant SYNGENTA PARTICIPATIONS AG		

This international search report has been prepared by this International Searching Authority and is transmitted to the applicant according to Article 18. A copy is being transmitted to the International Bureau.

This international search report consists of a total of 3 sheets.

☒ It is also accompanied by a copy of each prior art document cited in this report.

1. Basis of the report

a. With regard to the **language**, the international search was carried out on the basis of:

- ☒ the international application in the language in which it was filed
☐ a translation of the international application into _____, which is the language of a translation furnished for the purposes of international search (Rules 12.3(a) and 23.1(b))

b. ☐ This international search report has been established taking into account the **rectification of an obvious mistake** authorized by or notified to this Authority under Rule 91 (Rule 43.6**bis**(a)).

c. ☐ With regard to any **nucleotide and/or amino acid sequence** disclosed in the international application, see Box No. I.

2. ☐ **Certain claims were found unsearchable** (See Box No. II)

3. ☐ **Unity of invention is lacking** (see Box No III)

4. With regard to the **title**,

- ☒ the text is approved as submitted by the applicant
☐ the text has been established by this Authority to read as follows:

5. With regard to the **abstract**,

- ☒ the text is approved as submitted by the applicant
☐ the text has been established, according to Rule 38.2, by this Authority as it appears in Box No. IV. The applicant may, within one month from the date of mailing of this international search report, submit comments to this Authority

6. With regard to the **drawings**,

- a. the figure of the **drawings** to be published with the abstract is Figure No. _____
☐ as suggested by the applicant
☐ as selected by this Authority, because the applicant failed to suggest a figure
☐ as selected by this Authority, because this figure better characterizes the invention
b. ☐ none of the figures is to be published with the abstract

INTERNATIONAL SEARCH REPORT

International application No
PCT/EP2017/065541

A. CLASSIFICATION OF SUBJECT MATTER
INV. A01N25/28 A01N53/00
ADD.

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)
A01N

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

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

EPO-Internal

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 2013/115261 A1 (BORZATTA VALERIO [IT] ET AL) 9 May 2013 (2013-05-09) paragraphs [0060] - [0068]; claims; example 10	1-10
Y	----- US 6 133 197 A (CHEN JIN-LING [US] ET AL) 17 October 2000 (2000-10-17) abstract column 4, line 59 - column 6, line 67; claims; examples	1-10
Y	----- US 4 285 720 A (SCHER HERBERT B) 25 August 1981 (1981-08-25) column 3, line 51 - column 8, line 52; claims; examples -----	1-10



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

"E" earlier application or patent but published on or after the international filing date

"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)

"O" document referring to an oral disclosure, use, exhibition or other means

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

"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

12 July 2017

Date of mailing of the international search report

19/07/2017

Name and mailing address of the ISA/

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INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No

PCT/EP2017/065541

Patent document cited in search report	Publication date	Patent family member(s)	Publication date	
US 2013115261	A1	09-05-2013	BR 102012028188 A2	20-09-2016
			CN 103081896 A	08-05-2013
			EP 2589290 A1	08-05-2013
			ES 2527187 T3	21-01-2015
			US 2013115261 A1	09-05-2013
			ZA 201208286 B	31-07-2013

US 6133197	A	17-10-2000	JO 1981 B	15-12-1997
			US 6133197 A	17-10-2000
			ZA 9704359 B	12-05-1998

US 4285720	A	25-08-1981	NONE	



(51) International Patent Classification:

A01N 25/28 (2006.01) *A01N 53/00* (2006.01)

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(81) Designated States (unless otherwise indicated, for every
kind of national protection available): AE, AG, AL, AM,
AO, AT, AU, AZ, BA, BB, BG, BH, BN, BR, BW, BY, BZ,
CA, CH, CL, CN, CO, CR, CU, CZ, DE, DJ, DK, DM, DO,
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HR, HU, ID, IL, IN, IR, IS, JO, JP, KE, KG, KH, KN, KP,
KR, KW, KZ, LA, LC, LK, LR, LS, LU, LY, MA, MD, ME,
MG, MK, MN, MW, MX, MY, MZ, NA, NG, NI, NO, NZ,
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TR), OAPI (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW,
KM, ML, MR, NE, SN, TD, TG).

(54) Title: MICROCAPSULES ENCAPSULATING LAMBDA-CYHALOTHRIN

(57) Abstract: A composition comprising a microcapsule having a polyurea wall encapsulating a pyrethroid insecticide, wherein the polyurea wall of the microcapsule is made of polyurea polymers derived from a mixture of aromatic polyisocyanate (A) and aromatic diisocyanate (B), the weight ratio of (A) to (B) is 1:1, the polymer (or wall) content of each microcapsule is from 6 to 9 % by weight, and the amount of the pyrethroid insecticide is from 15 to 60 % by weight, each based on the weight of the microcapsule; such a composition providing improved worker exposure characteristics.



MICROCAPSULES ENCAPSULATING LAMBDA-CYHALOTHRIN

The present invention relates to certain compositions comprising microcapsules encapsulating a pyrethroid insecticide, and methods of controlling damage on plants with such compositions.

5 Microencapsulation of pesticides and other agricultural chemicals has been performed for a number of years, using various microencapsulation processes or techniques, and in connection with a number of different active ingredients.

WO 97/44125, WO 96/33611, WO 95/13698 and US 4285720 each disclose microencapsulation of pesticides.

10 WO 97/44125 describes that pyrethroids, particularly lambda-cyhalothrin, when used for foliar application, should be encapsulated because of its risks to workers, but also the capsules should be such that lambda-cyhalothrin is relatively quick released for control of the target foliar pests.

The microencapsulation of different active ingredients requires different considerations, and WO 97/44125 teaches that a polyurea wall made from a certain mixture of isocyanates in a defined ratio would be preferred.

The inventors have found that microcapsules encapsulating pyrethroids should indeed have a specific defined polyurea wall, but the wall properties now found to be desirable are contradictory to the wall properties described in WO97/44125, in particular to provide improved worker exposure characteristics, such as an improved toxicology profile,, whilst still maintaining effective pest control activity.

Accordingly, in a first aspect, the present invention provides a composition comprising a microcapsule having a polyurea wall encapsulating a pyrethroid insecticide, preferably lambda-cyhalothrin, wherein the polyurea wall of the microcapsule is made of polyurea polymers derived from a mixture of aromatic polyisocyanate (A) and aromatic diisocyanate (B), the weight ratio of (A) to (B) is about 1:1, the polymer (or wall) content of each microcapsule is from about 6 to about 9, preferably about 7 to about 8, % by weight, and the amount of the pyrethroid insecticide, preferably the lambda-cyhalothrin, is from about 15 to about 60, preferably about 20 to about 55, % by weight, each based on the weight of the microcapsule.

30 In a second aspect, the present invention provides an aqueous composition comprising the composition of the first aspect, provided the composition does not contain one or more of: an active ingredient having a melting point at 25°C of less or equal to 50°C; an active ingredient in salt form, and a free or dispersed hydrophobic oil phase.

In a further aspect, the present invention provides a composition comprising an aqueous composition of the first or second aspect and one or more other active ingredients.

In a further aspect, the present invention provides a method for controlling damage to a plant by a pest comprising applying an effective amount of a composition of each of the aspects of the invention to the pest, to the plant, or to its locus thereof.

In an embodiment of each aspect, the pyrethroid insecticide is lambda-cyhalothrin. The pyrethroid insecticide is present in the core of the capsules, and in the case of lambda-cyhalothrin, it is dissolved in the organic solvent.

In an embodiment of the first aspect, the d50 median diameter of the microcapsule in the composition is from about 0.5 to about 5, preferably about 1 to about 4, microns.

In an embodiment of the first aspect, the amount of the microcapsules in the composition is from about 45 to about 55, preferably about 47 to about 53, % by weight, based on the weight of the composition.

In an embodiment of the first aspect, the pyrethroid is present in an amount of from about 20 to about 55, preferably about 40 to about 50, more preferably about 43 to about 47, % by weight, based on the weight of the microcapsule.

In a further embodiment of the first aspect, the organic phase also comprises dispersed titanium dioxide particles having a d50 median particle size diameter of about 0.1 to about 0.6, preferably about 0.15 to about 0.5, microns, and are present in an amount of from about 3 to about 7, preferably about 3.5 to about 6, % by weight, based on the weight of the microcapsule.

In an embodiment, the aromatic polyisocyanate is polymethylene polyphenylisocyanate and the aromatic diisocyanate is toluene diisocyanate, preferably an isomeric mixture of toluene diisocyanate, such as about 80% 2,4-isomer and about 20% 2,6-isomer of toluene diisocyanate.

In an embodiment of the first aspect, the composition comprises a microcapsule having a polyurea wall encapsulating lambda-cyhalothrin and dispersed titanium dioxide particles, wherein the polyurea wall of the microcapsule is made of polyurea polymers derived from a mixture of polymethylene polyphenylisocyanate (A) and an isomeric mixture of toluene diisocyanate (B), provided that the d50 median particle size diameter of the titanium dioxide particles is from about 0.15 to about 0.5 microns, the d50 median diameter of the microcapsule in the composition is from 1 to 4, microns, the weight ratio of (A) to (B) is about 1:1, the polymer (or wall) content of each microcapsule is from about 7 to about 8, % by weight, the amount of titanium dioxide is present in an amount of from about 3.5 to about 6, % by weight, and the amount of the lambda-cyhalothrin, is from about 40 to about 50, % by weight, each based on the weight of the microcapsule; and further provided the amount of the microcapsule in the composition is from about 47 to about 53, % by weight, based on the weight of the composition.

The general processes for making the microencapsulated formulations of the invention are described in WO 97/44125. However, as noted, one of the essential differences is the ratio of the isocyanate monomers used to make the polyurea microcapsule wall.

Accordingly, the process, in brief, involves encapsulation of a water immiscible material (in the present case preferably an organic phase containing a pyrethroid insecticide) within discrete capsules of polyurea. In this process, hydrolysis of an isocyanate monomer to form an amine via a carbamic acid intermediate occurs, and that in turn reacts with another isocyanate monomer to form the polyurea. In general, the process comprises two stages.

In the first stage, a physical dispersion of a water-immiscible phase in an aqueous phase is prepared. The water-immiscible phase comprises the pesticide to be encapsulated together with other material as described below. As is known in the art, the dispersion is produced by a high shear device, and this step is carried out until the desired droplet size (as discussed below) is obtained. Only mild agitation is required for the remainder of the process.

In the second stage, the dispersion is stirred under high shear and maintained at a temperature range of from about 20°C to about 90°C, during which reaction occurs involving the organic diisocyanate and organic polyisocyanate to form the polyurea at the interfaces between the droplets of the organic phase and the aqueous phase. Adjustment of the pH of the resulting mixture and the temperature range during the second stage advance this condensation reaction.

The aqueous phase is prepared from water, a protective colloid, and preferably a surfactant.

In general, the surfactant or surfactants in this phase may be anionic or nonionic surfactants with an HLB range of from about 12 to about 16. If more than one surfactant is used, individual surfactants may have HLB values lower than 12 or higher than 16 as long as the overall HLB value of the combined surfactants will be within the range of about 12 to 16. Suitable surfactants include polyethylene glycol ethers of linear alcohols, ethoxylated nonylphenols, naphthalene sulfonates, salts of long chain alkyl benzene sulfonates, block copolymers of propylene and ethylene oxides, anionic/nonionic blends, and the like. Preferably the hydrophobic portion of the surfactant has chemical characteristics similar to the water-immiscible phase. Thus, when the latter contains an aromatic solvent, one suitable surfactant would be an ethoxylated nonylphenol. Particularly preferred surfactants include block copolymers of propylene oxide and ethylene oxide, and anionic/nonionic blends.

The protective colloid present in the aqueous (or continuous) phase must absorb strongly onto the surface of the oil droplets and can be selected from a wide range of such materials including polyacrylates, methyl cellulose, polyvinyl alcohol, polyacrylamide, poly (methylvinyl ether/maleic anhydride), graft copolymers of polyvinyl alcohol and methylvinyl ether/maleic acid [hydrolyzed methyl vinyl ether/maleic anhydride (see U.S. Patent 4,448,929, which is hereby incorporated by reference herein)]; and alkali metal or alkaline earth metal lignosulfonates. Preferably, however, the protective colloid is selected from alkali metal and

alkaline earth metal lignosulfonates, most preferably sodium lignosulfonates, such as TERGITOL™ NP7, Tergitol XD, Tergitol NP40, Tergitol 15-S-20, WITCONATE™ 90.

The range of surfactant concentration in the process is from about 0.01 to about 10.0 percent by weight, based on the aqueous phase, but higher concentrations of surfactant may also be used. The protective colloid is generally present in the aqueous phase in an amount of from about 0.1 to about 5.0 percent by weight. The amount of protective colloid employed will depend on various factors, such as molecular weight, compatibility, etc., so long as enough is present to completely coat the surfaces of all the oil droplets. The protective colloid can be added to the aqueous phase prior to the addition of the organic phase, or can be added to the overall system after the addition of the organic phase or the dispersion of it. The surfactants should be chosen so as to not displace the protective colloid from the droplet surfaces.

The organic phase comprises the pyrethroid to be encapsulated, one or more solvents, an aromatic diisocyanate and an aromatic polyisocyanate. Suitable solvents include aromatic hydrocarbons such as xylenes, naphthalenes, or mixtures of aromatics; aliphatic or cycloaliphatic hydrocarbons such as hexane, heptane and cyclohexane; alkyl esters including alkyl acetates and alkyl phthalates, ketones such as cyclohexanone or acetophenone, chlorinated hydrocarbons, vegetable oils, or mixtures of two or more such solvents. Preferably the pyrethroid is lambda-cyhalothrin and is dissolved in the organic solvent.

In the event a UV protectant, such as titanium dioxide, is used, this would be encapsulated too and so titanium dioxide particles are comprised in the organic phase too, as well as dispersants used in dispersing the particles. The dispersants useful for dispersing the titanium dioxide particles are certain non-ionic surfactants which act by steric hindrance and are active only at the protectant solid/organic liquid interface and do not act as emulsifying agents. Such dispersants are suitably made up of (a) a polymeric chain having a strong affinity for the liquid and (b) a group which will absorb strongly to the solid. Examples of such dispersants are those of the HYPERMER™ and ATLOX™ lines, including Hypermer PS1, Hypermer PS2, Hypermer PS3, Atlox LPI, Atlox LP2, Atlox LP4, Atlox LP5, Atlox LP6, and Atlox 4912; and AGRIMER™ polymers such as Agrimer AL-216 and AL-220.

The present inventors have found that by carefully modifying the microcapsule properties, such as the ratio of the aromatic polyisocyanate to the aromatic diisocyanate, and the polymer wall content and amount, microcapsules are obtained providing improved work exposure characteristics (such as improved toxicology profile, e.g., at least one of better oral, dermal, and inhalation toxicity, improved skin and eye irritation, and a better skin sensitizer), but which are still relatively quickly released when applied in an agricultural environment. The walls are formed only of a mixture of aromatic diisocyanates with an aromatic polyisocyanate, in which the weight ratio of polyisocyanate to diisocyanate is about 1:1.

The diisocyanate and the polyisocyanates which may be used in this invention are those described in U.S. Patent 4,285,720. Diisocyanates usable in the process of this invention include m-phenylene diisocyanate, p-phenylene diisocyanate; 1-chloro-2,4-phenylene diisocyanate; 4,4'-methylenebis(phenyl isocyanate); 3,3'-dimethyl-4,4' -biphenylene diisocyanate; 4,4'-methylenebis (2-methylphenyl isocyanate); 3,3, dimethoxy4 ,4' -biphenylene diisocyanate; 2,4-tolulene diisocyanate; 2,6-tolulene diisocyanate, isomeric mixtures of 2,4- and 2,6-toluene diisocyanates; and 2,2' ,5 5, -tetramethyl-4,4' -biphenylene diisocyanate. Preferred are isomeric mixtures of 2,4- and 2,6-toluene diisocyanates.

Aromatic polyisocyanates usable in this invention have 3 or more isocyanate groups and include polymethylene polyphenylisocyanate, triphenylmethane triisocyanate (DESMODUR™ R) and the adduct formed between 1 mole of trimethylolpropane and 3 moles of toluene diisocyanate ("Desmodur TH").

While the prior art discloses the uses of mixtures of these two types of isocyanates, specifically toluene diisocyanate and polymethylene polyphenylisocyanate, with many active ingredients, the teaching in respect of pyrethroids suggests a higher amount of toluene diisocyanate than the polymethylene polyphenylisocyanate.

The total amount of organic isocyanates used in the process will determine the wall content of the microcapsules formed. In general, the isocyanates (and correspondingly the microcapsule walls formed from them) will comprise from about 6 to about 9 percent by weight of the microcapsule, preferably from about 7 to about 8 percent by weight.

The size of the microcapsules produced, which corresponds to the droplet size of the organic phase in the oil-in-water emulsion, is about d50 median diameter of from 0.5 to 5, preferably 1 to 4, microns. The droplet size can be adjusted by the stirring speed and time, and by the type and amount of surfactant employed, as generally known in the art.

To obtain the appropriate emulsion, the organic phase is added to the aqueous phase with stirring. A suitable dispersing means is employed to disperse the organic phase in the aqueous phase. This means may be any high shear device, operated so as to obtain the desired droplet (and corresponding microcapsule particle) size within the range of about 0.5 to about 5, preferably about 1 to about 4 microns. Once the proper droplet size is obtained, the dispersion means is discontinued and only mild agitation is required for the remainder of the process.

To form the microcapsules, the temperature of the two-phase mixture is then raised from ambient to a value of from about 20°C to about 90°C, preferably about 40°C to about 90°C. Depending on the system, as described in U.S. Patent 4,285,720, the pH value may be adjusted to an appropriate level.

The microencapsulated composition of the present invention may also contain conventional formulation aids, such as bactericides, antifoams, antifreeze agents, pH adjusters, buffers and viscosity control agents.

In a further aspect, the present invention provides a process for preparing a composition as defined in the first aspect, which process comprises the steps of (a) preparing an organic phase comprising a pyrethroid insecticide, preferably lambda-cyhalothrin, to be encapsulated, aromatic polyisocyanate (A), aromatic diisocyanate (B), wherein the weight ratio of the (A) to (B) is 1:1, and optionally titanium dioxide particles having d50 median particle size of 0.1 to about 0.6, preferably 0.15 to about 0.5, microns, dispersed in the organic phase by means of a dispersant; (b) introducing the organic phase into an aqueous phase comprising water, a protective colloid, and optionally a surfactant to form a dispersion of the organic phase in the aqueous phase; (c) mixing the dispersion under high shear to form an oil-in-water emulsion in which the oil droplets of the emulsion have a d50 median diameter of from 0.5 to 5, preferably 1 to 4, microns; (d) adjusting as necessary the temperature and/or pH of the oil-in-water emulsion such that a polymerization reaction occurs forming polyurea microcapsules containing the organic phase.

The microencapsulated formulation of the invention is an aqueous formulation containing capsules and so would have the formulation designation code CS. The microencapsulated formulations of the present invention are storage stable and have acceptable handling characteristics, so can be satisfactorily applied, for example, in a field of plants. To ensure the capsule integrity of such CS formulations, the aqueous composition comprising the capsules does not contain one or more of: an active ingredient having a melting point at 25°C of less than or equal to 50°C; an active ingredient in salt form, and a free or dispersed hydrophobic oil phase.

The microencapsulated compositions of the invention can be combined with other active ingredients.

In an embodiment, an aqueous composition of the present invention comprises the encapsulated lambda-cyhalothrin of the first aspect and one or more other insecticides and/or acaricides, preferably the one or more other insecticides and acaricides are not encapsulated with the pyrethroids.

Examples of one or more other active ingredients are from the class neonicotinoids (such as imidacloprid, acetamiprid, thiamethoxam, thiacloprid), carbamates, botanical compounds, diamides (such as chloroantraniliprole), macrocyclic lactones (such as abamectin, spinosad and emamectin), organophosphorus compounds (such as acephate), tetracarboxylic acids (such as spirotetramat), tetracarboxylic acids (such as spiromesifen, spirotetramat) and insect growth regulators (such as lufenuron).

The other active ingredient is normally suspended in the aqueous composition comprising the capsules of the invention using conventional formulation techniques; as necessary further conventional formulation aids are used to prepare a stable mixture formulation, wherein the resulting formulation would have designation ZC code.

5 In an embodiment, an aqueous composition of the present invention comprises an encapsulated lambda-cyhalothrin of the first aspect and, as a further active ingredient, suspended thiamethoxam or suspended acetamiprid.

In a further embodiment, an aqueous composition of the present invention comprises encapsulated lambda-cyhalothrin of the first aspect, and, as further active ingredients,
10 suspended thiamethoxam or suspended acetamiprid, and suspended lufenuron.

The d50, as used herein, refers to the 50th percentile rank, or median value, as measured on a volumetric basis by CIPAC method MT 187 for particle size analysis by laser diffraction. Malvern S, 2000 or 3000 instruments are the industry standard for measuring particle size by light diffraction, but similar instruments made by Cilas, Coulter-Beckman, Horiba,
15 Sympatec and others equivalents are also acceptable.

General Procedure for an encapsulated formulation of the invention

A solution of lambda-cyhalothrin in SOLVESSO 200 solvent was prepared. The dispersants were added followed by the titanium dioxide, and the resulting suspension agitated
20 with a high shear stirrer. After the titanium dioxide was well dispersed, polymethylene polyphenylisocyanate and isomeric mixture of toluene diisocyanate were added to complete the organic phase.

This phase was introduced to the aqueous phase with agitation with a high shear stirrer to form an oil-in-water emulsion. The median droplet size was about 3.0 ± 1 microns. The
25 temperature was then raised to 50°C over a 30-minute period while maintaining mild agitation, and then maintained at 50°C for 3 hours. The resulting suspension of microcapsules was allowed to cool to room temperature and other formulation aids, such as viscosity control agent(s), and bactericide were added and, the pH was adjusted to 5.0.

The compositions of the present invention are suitable for control of pests by foliar
30 application, but other applications as well, such as to soil or in or around buildings. Examples of pests are sucking insects (e.g. psyllids, thrips (e.g. *Thrips tabaci*) stink bugs (e.g. *Euschistus heros*), plant hoppers and aphids (e.g. *Aphis gossypii*, *Myzus persicae*)) and chewing insects (e.g., *Helicoverpa* and diamondback moth (DBM), caterpillars, beetles, and slugs and snails).

Suitable target plants are especially potatoes, cereals, (wheat, barley, rye, oats), rice,
35 maize, tree nuts, alfalfa, head and stem brassica (including but not limited to broccoli, cabbage, cauliflower), sugar beet, cotton, millet varieties, sorghum, tobacco, sun flowers, beans, peas, oil plants (rape, canola), soybeans, cabbages, tomatoes, eggplants (aubergines), pepper and other

vegetables, and spices as well as ornamental shrubs and flowers. The target crops also include transgenic crop plants of the foregoing varieties.

Depending on the aims to be achieved and give circumstances, the compositions of the present invention are applied at a rate per hectare, based on the active ingredient, of from 1 to 500 g/ha, such as 5 to 300g/ha, for example, 10 to 100 g/ha.

The Examples which follow serve to illustrate the invention. The invention can be distinguished from other prior art by virtue of greater efficacy at low application rates and improved toxicological profile, which can be verified by the person skilled in the art using the experimental procedures outlined in the Examples below, using lower rates if necessary.

EXAMPLES

The key components and characteristics of the polyurea microcapsule formulations are indicated in Tables 1 and 2, each formulation is a CS formulation type.

The general procedure for preparation of the microencapsulated lambda-cyhalothrin products is described in Example 3 of WO97/44125.

Tables 1 and 2 provide details of the different lambda-cyhalothrin encapsulated formulations. Each formulation in Tables 1 and 2 contained titanium dioxide in the microcapsules in an amount of about 4.6% (based on the weight of the microcapsule) and had a d50 median particle size of about 0.2 to 0.4 microns.

Some of the encapsulated lambda-cyhalothrin formulations were then combined with thiamethoxam, or both thiamethoxam and lufenuron. The 2-way (lambda-cyhalothrin & thiamethoxam) and 3-way (lambda-cyhalothrin, thiamethoxam & lufenuron) mixture formulations were of the ZC formulation type, which is a formulation containing capsules and also suspended solid particles. The thiamethoxam and lufenuron were suspended in the aqueous phase and the lambda-cyhalothrin was encapsulated in the microcapsules. The 2-way mixture and 3-way mixture formulated products were prepared using conventional formulating practices, such as incorporating suitable bactericides, surfactants, dispersants, antifreeze, and viscosity control agents to suspend the active ingredients and/or microcapsules and improve storage stability.

Details of the resulting 2-way mixture and 3-way mixture formulated products are given in table 3.

BIOLOGY

B1: release characteristics of different capsules of lambda-cyhalothrin

A sample of the CS formulation (0.30-0.35g) is weighed into a 2oz. round glass bottle. 6ml of water is then added and the sample is dispersed with gentle shaking until homogenous. Using a volumetric dispensing unit or pipette, dispense 50ml of Internal Standard (IS) (which is dicyclohexyl phthalate in hexanes at a concentration of 0.35mg/ml). The bottle is then placed on

a horizontal orbital shaker set to 200 ± 10 rpm & a timer is started. After 5 min, 1ml sample is removed from the organic phase and transferred through a $0.45 \mu\text{m}$ nylon syringe filter into a GC vial to which $25 \mu\text{l}$ of trifluoroacetic acid (TFA) is added via pipette to prevent conversion of lambda cyhalothrin to the Cis A isomer. The sample is then capped immediately and analysed by gas chromatography for lambda cyhalothrin content.

The same sampling procedure is repeated at 0.5, 2, 4, 8, 24, and 96 hour intervals and the resulting concentration of lambda Cyhalothrin is plotted as a function of time to determine the capsule release rate profile.

Results of certain formulations are given in the Tables A.

B2: Efficacy trials for *Euschistus heros* (nymphs & adults)

The efficacy of Example 3, L and M were assessed against each other in the field. Each formulation was applied on soybean about ten weeks after the planting date in an amount of 200 ml/ha in separate 96m^2 randomised block plots in Brazil. The stinkbug nymphs (of the third instar and above) and adults were counted using a standardized monitoring device three, seven and ten days after application of the formulation. A plot with no treatment was also assessed for the stinkbug nymphs and adults as the check. The each trial was carried out in four replicates.

The average of the three assessments (three, seven and ten days after application) compared to the check for each of the formulations are provided in Table B.

SAFETY and TOXICOLOGY

Certain formulations were assessed for one or more of oral toxicity, dermal toxicity, inhalation risk, skin irritation, eye irritation and skin sensitisation (LLNA) using OECD test methods indicated below in Table C.

Table D provides the results of the tests.

Table 1: Encapsulated Lambda-cyhalothrin formulations I

	Example 1	Example A	Example B	Example C
% weight of lambda-cyhalothrin	22.8	22.8	22.8	9.7
% weight of isocyanate monomers	3.8	3.8	3.8	2.8
ratio of polymethylene polyphenylisocyanate to isomeric mixture of toluene diisocyanate (80%, 2,4-isomer; 20% 2,6-isomer)	1:1	1:30	1:30	1:1
% weight of organic phase	49.7	50.6	49.7	21.4
% weight of aqueous phase	50.3	49.3	50.3	78.6
d50 median diameter of microcapsules, microns	1.5 – 3.5	1.5 – 3.5	1.5 – 3.5	10-12
% weight of polyurea polymer (or wall content) of capsule	7.7	7.5	7.7	13.1

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Table 2: Encapsulated Lambda-cyhalothrin formulations II

	Example E	Example F	Example G	Example H
% weight of lambda-cyhalothrin	22.8	22.8	22.8	22.8
% weight of isocyanate monomers	5.9	5.9	3.8	3.8
ratio of polymethylene polyphenylisocyanate to isomeric mixture of tolyene diisocyanate (80%, 2,4-isomer; 20% 2,6-isomer)	1:30	1:15	n/a	1:15
% weight of organic phase	52.4	52.4	49.7	49.7
% weight of aqueous phase	47.7	47.7	50.3	50.3
d50 median diameter of microcapsules, microns	10-12	10-12	1.5 – 3.5	1.5 – 3.5
% weight of polyurea polymer content (or wall content) of capsule	11.2	11.2	7.7	7.7

Table 3: 2 and 3-way mixture formulation of encapsulated Lambda-cyhalothrin formulations

	Example 2	Example I	Example J	Example K	Example 3	Example L
source of encapsulated lambda-cyhalothrin formulation	Example 1	Example A	Example B	Example B	Example 1	Example B
wt% of lambda-cyhalothrin	9.48	9.48	9.48	9.48	8.80	8.80
wt% of thiamethoxam	12.60	12.60	12.60	12.60	13.20	13.20
wt% of lufenuron					6.60	6.60

Comparative L contained similar components in similar amounts as Comparative K apart from a using lower amount, i.e., 0.19 wt% rather than 0.28wt%, of the anti-microbial component (PROXEL™ GXL) - a 20% aqueous dipropylene glycol solution of 1,2-benzisothiazolin-3-one.

Table A1: release profile (based on weight) of Lambda-Cyhalothrin over 96 hours*

time, hrs	Example 1	Example B	Example C	Example E	Example F	Example G	Example H
0.1	0	0.11	0.07	0.97	1.36	0	0.35
0.5	0.04	0.6	0.27	2.9	3.64	0.03	0.58
2	0.19	1.71	1.13	6.17	8.61	0.19	1.39
4	0.62	4.99	2.48	8.58	11.61	0.56	3.24
8	1.93	19.84	7.52	15.01	18.16	2.3	15.27
24	7.26	25.72	23.38	27.4	25.6	17.39	26.6
96	22.74	25.67	27.13	27.27	25.59	30.14	26.59

* all formulations had comparable storage stability.

Table A2: release profile (based on %) of Lambda-Cyhalothrin over 96 hours (from Table A1)

time, hrs	Example 1	Example B	Example C	Example E	Example F	Example G	Example H
0.1	0.0%	0.5%	0.3%	4.3%	6.0%	0.0%	1.5%
0.5	0.2%	2.6%	1.2%	12.7%	16.0%	0.1%	2.5%
2	0.8%	7.5%	5.0%	27.1%	37.8%	0.8%	6.1%
4	2.7%	21.9%	10.9%	37.6%	50.9%	2.5%	14.2%
8	8.5%	87.0%	33.0%	65.8%	79.6%	10.1%	67.0%
24	31.8%	112.8%	102.5%	120.2%	112.3%	76.3%	116.7%
96	99.7%	112.6%	119.0%	119.6%	112.2%	132.2%	116.6%

Table B: Efficacy against *Euschiustus heros* (nymphs & adults)

	% control	
	nymphs	adults
Example 3	80	74
Example L	80	78

Table C: OECD methods used in the tests in Table D, and a brief description

Test assessment	OECD test method	Brief description
Acute Oral	OECD 425	The method permits estimation of an LD50 with a confidence interval and the results allow a substance to be classified for acute toxicity according to the Globally Harmonised System of classification and labelling of chemicals. It is easiest to apply to materials that produce death within couple of days. This Test Guideline is intended for use with rodents (rat female preferably). There are a limit test and a main test. The limit test can be used efficiently to identify chemicals that are likely to have low toxicity. The test substance is administered generally in a single dose by gavage to animals fasted prior to dosing. Single animals are dosed in sequence usually at 48h intervals. The first animal is dosed a step below the best preliminary estimates of the LD50. The second animal receives a lower dose (if the first animal dies) or a higher dose (if the first animal survives). Animals are observed with a special attention given during the first 4 hours and daily thereafter, for a total of 14 days generally. Weights Animals should be determined at least weekly. All the animals should be subjected to gross necropsy. Globally the LD50 is calculated using the maximum likelihood method. Following this, it may be possible to compute interval estimates for the LD50; most narrow is the interval and better is LD50 estimation.
	OECD 223	This Test Guideline describes procedures designed to estimate the acute oral toxicity of substances to birds, and it provides three testing options: (1) limit dose test, (2) LD50-slope test, and (3) LD50-only test. The LD50-slope and LD50-only options are sequential testing procedures. The test method selected will depend on whether or not a definitive median dose (LD50) and slope of the dose-response curve are both needed. The limit dose test is the preferred test when toxicity is expected to be low and lethality is unlikely at the limit dose. The limit dose should be adequate for assessment purposes, and it is usually 2000 mg/kg-bwt. Five or ten birds are tested at the limit dose in addition to a control group. The LD50-slope test is the preferred test when regulatory or other requirements determine that the slope of the dose-response curve and/or the confidence interval is required in addition to an estimate of the LD50. This is a 3- or 4-stage test with 24 or 34 birds in addition to a control group. The LD50-only test is the preferred test when regulatory or other requirements determine that only the median lethal dose is required but neither the slope of the dose response curve or the confidence interval for the LD50

Test assessment	OECD test method	Brief description
Acute Dermal	OECD 402	is required. This may be the appropriate test to estimate a percentile of a species sensitivity distribution of LD50s and to provide information for product labelling purposes. This test has two stages, with 14 birds in addition to a control group. This method provides information on health hazard likely to arise from a short-term exposure to solid or liquid test substance by the dermal route. This Test Guideline is intended primarily for use with rodents (rat, rabbit or guinea pig may be used). For each dose at least 5 animals (of the same sex) are used. The test substance is applied to the skin (not less than 10 per cent of the body surface area) in graduated doses to several groups of experimental animals, one dose being used per group. At least three dose levels should be used, appropriately spaced to produce a dose-response curve. A limit test of at least 2000 mg/kg could be made. The observation period should be at least 14 days. During the first day the animals should be observed frequently and then the observations should be made daily. Necropsy of all animals should be carried out and all gross pathological changes should be recorded. A study of acute toxicity by the dermal route and determination of a dermal LD provides an estimate of the relative toxicity of a substance by the dermal route of exposure and they may serve as a basis for classification and labeling. It is an initial step in establishing a dosage regimen in subchronic and other studies and may provide information on dermal adsorption and the mode of toxic action of a substance by this route.
Acute Inhalation	OECD 403	This method provides information on health hazard likely to arise from short-term exposure to a test article (gas, vapour or aerosol/particulate test article) by inhalation. The revised Test Guideline describes two studies: a traditional LC50 protocol and a Concentration x Time (C x t) protocol. It can be used to estimate a median lethal concentration (LC50), non-lethal threshold concentration (LC01) and slope, and to identify possible sex susceptibility. This Test Guideline enables a test article quantitative risk assessment and classification according to the Globally Harmonized System for the Classification and Labelling of Chemicals. In the traditional LC50 protocol, animals are exposed to one limit concentration or to three concentrations, at least, for a predetermined duration, generally of 4 hours. Usually 10

Test assessment	OECD test method	Brief description
		animals should be used for each concentration. In the C x T protocol, animals are exposed to one limit concentration or a series of concentrations over multiple time durations. Usually 2 animals per C x t interval are used. Animals (the preferred species is the rat) should be observed for at least 14 days. The study includes measurements (including weighing), daily and detailed observations, as well as gross necropsy.
Skin Irritation	OECD 404	This method provides information on health hazard likely to arise from exposure to liquid or solid test substance by dermal application. This Test Guideline recommends sequential testing strategies, which include the performance of validated and accepted in vitro or ex vivo tests for corrosion/irritation. The albino rabbit is the preferable laboratory animal. The substance to be tested is applied in a single dose to a small area of skin (approximately 6cm²) of an experimental animal; untreated skin areas of the test animal serve as the control. The exposure period is 4 hours. Residual test substance should then be removed. The dose is 0.5ml (liquid) or 0.5g (solid) applied to the test site. The method consists of two tests: the initial test and the confirmatory test (used only if a corrosive effect is not observed in the initial test). All animals should be examined for signs of erythema and oedema during 14 days. The dermal irritation scores should be evaluated in conjunction with the nature and severity of lesions, and their reversibility or lack of reversibility. When responses persist to the end of the 14-day observation period, the test substance should be considered an irritant.
Eye Irritation	OECD 405	This method provides information on health hazard likely to arise from exposure to test substance (liquids, solids and aerosols) by application on the eye. This Test Guideline is intended preferably for use with albino rabbit. The test substance is applied in a single dose in the conjunctival sac of one eye of each animal. The other eye, which remains untreated, serves as a control. The initial test uses an animal; the dose level depends on the test substance nature. A confirmatory test should be made if a corrosive effect is not observed in the initial test, the irritant or negative response should be confirmed using up to two additional animals. It is recommended that it be conducted in a sequential manner in one animal at a time, rather than exposing the two additional animals simultaneously. The duration of the observation period should be sufficient to evaluate fully the magnitude and reversibility of the effects observed. The eyes should be examined at 1, 24, 48, and 72 hours after test substance application. The ocular irritation scores should be evaluated in conjunction with the

Test assessment	OECD test method	Brief description
	OECD 438	nature and severity of lesions, and their reversibility or lack of reversibility. Use of topical anesthetics and systemic analgesics to avoid or minimize pain and distress in ocular safety testing procedures is described. The Isolated Chicken Eye (ICE) test method is an <i>in vitro</i> test method that can be used to classify substances as "ocular corrosives and severe irritants". The ICE method uses eyes collected from chickens obtained from slaughterhouses where they are killed for human consumption, thus eliminating the need for laboratory animals. The eye is enucleated and mounted in an eye holder with the cornea positioned horizontally. The test substance and negative/positive controls are applied to the cornea. Toxic effects to the cornea are measured by a qualitative assessment of opacity, a qualitative assessment of damage to epithelium based on fluorescein retention, a quantitative measurement of increased thickness (swelling), and a qualitative evaluation of macroscopic morphological damage to the surface. The endpoints are evaluated separately to generate an ICE class for each endpoint, which are then combined to generate an Irritancy Classification for each test substance.
Skin Sensitisation (LLNA)	OECD 429	The basic principle underlying the Local Lymph Node Assay (LLNA) in mouse is that sensitizers induce a primary proliferation of lymphocytes in the auricular lymph nodes draining the site of chemical application. This proliferation is proportional to the dose applied and provides a measurement of sensitisation. The method described is based on the use of radioactive labelling to measure cell proliferation. A minimum of four animals is used per dose group, with a minimum of three concentrations of the test substance, plus a negative control group treated with the vehicle only, and a positive control, as appropriate. The experimental schedule of the assay is during 6 days. Thereafter, the animals are killed and a cell suspension of lymph node cells is prepared. The incorporation of 3H-methyl thymidine is measured by β-scintillation counting as disintegrations per minute (DPM). The Test Guideline includes performance standards that can be used to evaluate the validation status of new and/or modified test methods that are functionally and mechanistically similar to the LLNA. A reduced LLNA approach which could use up to 40% fewer animals is also described as an option. This study includes: measurements (weighing, DPM), and clinical daily observations. Results are expressed as

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Test assessment	OECD test method	Brief description
	OECD 406	the Stimulation Index (SI). The SI is obtained by calculation and should be ≥ 3 before classification of the test material as a skin sensitizer is warranted. This method provides information on health hazard likely to arise from exposure to test substance via intradermal injection and/or epidermal application. In this Test Guideline, the methods preferred over other are: the Guinea Pig Maximisation Test (GPMT) of Magnusson and Kligman which uses adjuvant and the non adjuvant Buehler Test. This Test Guideline is intended primarily for use with guinea pig, but recently mouse models for assessing sensitisation potential have been developed. For the GPMT at least 10 animals in the treatment group and 5 in the control group are used. For the Buehler test, a minimum of 20 animals is used in the treatment group and at least 10 animals in the control group. The test animals are initially exposed to the test substance. Following a rest period, the induction period (10-14 days), during which an immune response may develop, then the animals are exposed to a challenge dose. The GPMT is made during approximately 23-25 days, the Buehler test, during approximately 30-32 days. The concentration of test substance used for each induction exposure should be well-tolerated systemically and should be the highest to cause mild-to moderate skin irritation, for the challenge exposure the highest nonirritant dose should be used. All skin reactions and any unusual findings should be observed and recorded (other procedures may be carried out to clarify doubtful reactions).

Table D: Toxicological and safety studies

		Acute Oral, (mg/kg)	Acute Dermal, (mg/kg)	Acute Inhalation, (mg/L)	Skin Irritation	Eye Irritation	Skin Sensitisation (LLNA)
Example 1		>2000	>5000	>2.4	No	Mild	Positive
	Test method	OECD 425	OECD 402	OECD 403	OECD 404	OECD 405	OECD 429
Example A		180	>4000	nt	Mild	Moderate	Positive
	Test method	OECD 223	OECD 402	n/a	OECD 404	OECD 405	OECD 429
Example B		310	>2000	3.12	Mild	Mild	Positive
	Test method	OECD 425	OECD 402	OECD 403	OECD 404	OECD 405	OECD 406
Example 2		1596	nt	>2.0	nt	Minimal	Negative
	Test method	OECD 425	n/a	OECD 403	n/a	OECD 405	OECD 429
Comparative J		310	>2000	>2.15	Moderate	Mild	Negative
	Test method	OECD 425	OECD 402	OECD 403	OECD 404	OECD 405	OECD 406
Comparative K		310	nt	nt	nt	No	nt
	Test method	OECD 425	n/a	n/a	n/a	OECD 438	n/a
Example 3		341	>5000	2.57	Mildly irritant	Category III	Positive
	Test method	OECD 425	OECD 402	OECD 403	OECD 404	OECD 405 (EPA)	OECD 406
Comparative L		103	5000	0.54-2.08	Mildly irritant	Category IV	Negative
	Test method	OECD 425	OECD 402	OECD 403	OECD 404	OECD 405 (EPA)	OECD 406

nt = not tested; n/a = not applicable

CLAIMS

1. A composition comprising a microcapsule having a polyurea wall encapsulating a pyrethroid insecticide, wherein the polyurea wall of the microcapsule is made of polyurea polymers derived from a mixture of aromatic polyisocyanate (A) and aromatic diisocyanate (B), the weight ratio of (A) to (B) is 1:1, the polymer (or wall) content of each microcapsule is from 6 to 9 % by weight, and the amount of the pyrethroid insecticide is from 15 to 60 % by weight, each based on the weight of the microcapsule.
2. The composition according to claim 1 wherein the pyrethroid insecticide is lambda-cyhalothrin.
3. The composition according to either claim 1 or claim 2 wherein the d50 median diameter of the microcapsule in the composition is from 0.5 to 5 microns, and the amount of the microcapsules in the composition is from 45 to 55 % by weight, based on the weight of the composition.
4. The composition according to any one of claims 1 to 3 wherein the microcapsule further encapsulates titanium dioxide particles having a d50 median particle size diameter of 0.1 to 0.6 microns, in an amount of 3 to 7 % by weight, based on the weight of the microcapsule.
5. The composition according to any one of claims 1 to 4, in which the aromatic polyisocyanate is polymethylene polyphenylisocyanate.
6. The composition according to any of claims 1 to 5, in which the aromatic diisocyanate is toluene diisocyanate, preferably an isomeric mixture of toluene diisocyanate.
7. An aqueous composition comprising the composition defined in any one of claims 1 to 6, provided the composition does not contain one or more of: an active ingredient having a melting point at 25°C of less or equal to 50°C; an active ingredient in salt form, and a free or dispersed hydrophobic oil phase.
8. A composition comprising an aqueous composition defined in claim 7 and one or more other active ingredients,
9. The composition according to claim 8 wherein the one or more other active ingredient is selected from an insecticide and acaricide.

10. A method for controlling damage to plant by pests comprising applying an effective amount of a composition defined in any one of claims 1 to 9 to the pest, to the plant, or to its locus thereof.

INTERNATIONAL SEARCH REPORT

International application No
PCT/EP2017/065541

A. CLASSIFICATION OF SUBJECT MATTER
INV. A01N25/28 A01N53/00
ADD.

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)
A01N

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

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

EPO-Internal

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 2013/115261 A1 (BORZATTA VALERIO [IT] ET AL) 9 May 2013 (2013-05-09) paragraphs [0060] - [0068]; claims; example 10	1-10
Y	US 6 133 197 A (CHEN JIN-LING [US] ET AL) 17 October 2000 (2000-10-17) abstract column 4, line 59 - column 6, line 67; claims; examples	1-10
Y	US 4 285 720 A (SCHER HERBERT B) 25 August 1981 (1981-08-25) column 3, line 51 - column 8, line 52; claims; examples	1-10



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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INTERNATIONAL SEARCH REPORT

Information on patent family members

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