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(54) Title: SYNERGISTIC INSECTICIDAL COMPOSITION

(57) Abstract: The present invention relates to a novel synergistic Insecticidal composition comprising Clothianidin, chlorantraniliprole and Kojic acid as the active ingredients, useful in control of stem borer in agricultural crops, particularly in Rice (Oryza sativa).



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## SYNERGISTIC INSECTICIDAL COMPOSITION

### FIELD OF INVENTION

The present invention belongs to the field of pesticide containing heterocyclic compounds. In particular, the invention provides synergistic insecticidal combination comprising Clothianidin, Chlorantraniliprole and a UV Protectant that enables a reduction in percent degradation of insecticide after exposure to the sunlight or ultra violet radiation without affecting the synergistic activity of the insecticidal combination.

### BACKGROUND OF THE INVENTION

The protection of crops and its produce from insect pest damage is essential in agriculture produce enhancement. Chemical control by the use of various chemicals and formulations is an important tool in agriculture for the prevention and control of pests. Insecticides of many types and groups are reported in the literature and a large number are in use, commercially, for control of pests in agriculture.

Clothianidin (Clothianidin), chemical name: E)-1-(the chloro-1,3-thiazoles-5-of 2-ylmethyl)-3-methyl-2-nitroguanidine; Chemical molecular formula:  $C_6H_8ClN_5O_2S$ . It is a neonicotinoid. Neonicotinoids are a class of insecticides that are chemically similar to nicotine, which has been used as a pesticide since the late 1700s. Clothianidin and other neonicotinoids act on the central nervous system of insects as an agonist of acetylcholine, the neurotransmitter that stimulates nAChR, targeting the same receptor site (AChR) and activating post-synaptic acetylcholine receptors but not inhibiting AChE.

chlorantraniliprole, the bromo-N-[4-chloro-2-methyl-6-[(of chemical name: 3-methyl-carbamoyl) benzene]-1-(3-chloropyridine-2-yl)-1-hydrogen-pyridine-5-formamide. Chlorantraniliprole was developed world-wide by DuPont belonging to a new class of selective insecticides featuring a novel mode of action to control a range of pests belonging to the order Lepidoptera and some other Coleoptera, Diptera and Isoptera species. Chlorantraniliprole opens muscular calcium channels (in particular the ryanodine receptor), rapidly causing paralysis and ultimately death of sensitive species. The differential selectivity Chlorantraniliprole had towards insect ryanodine receptors explained the outstanding profile of low mammalian toxicity. Chlorantraniliprole is active on chewing pest insects primarily by ingestion and secondarily by contact.

Kojic acid is a fungal metabolic product produced by a few species of *Aspergillus*, especially by *A. oryzae*, which has the Japanese common name koji. This acid is a by-product in the fermentation process of malting rice, for use in the manufacturing of sake, the Japanese rice wine.

Kojic acid was first isolated in 1907 by Saito from mycelia of *A. oryzae* grown on steamed rice. In 1912 Yabuta gave it the name kojic acid, and only in 1924 he deciphered the correct structure of the molecule of this acid. The major applications of Kojic acid (KA) and its derivatives in medicine are based on their biocompatibility, antimicrobial and antiviral, antitumor, antidiabetic, anticancer, anti-speck, anti-parasitic, and pesticidal and insecticidal properties. In addition, KA and its derivatives are used as anti-oxidant, anti-proliferative, anti-inflammatory, radio protective and skin-lightening agent in skin creams, lotions, soaps, and dental care products. KA has the ability to act as a UV protector in plants and human beings.

Many pesticides are sensitive to sunlight and decompose upon exposure to sunlight. The decomposition might already have taken place during storage or even more after application to the environment. As a result, the pesticidal activity decreases, higher amounts of pesticides have to be applied, the pesticides have to be applied in shorter intervals, or eventually toxic degradation products are produced.

There is therefore a need in the art for combinations of insecticidal compounds with UV arrestor compounds that help improve stability of insecticides and further enhance the spectrum of control. With crop tolerances decreasing, lower use rates being imposed and increasing resistance, there is a need for a combination of actives that allows for broader disease control spectrum that combines curative and preventive actives and has a lower dosage.

## OBJECT OF THE INVENTION

It is an object of the present invention to provide an environmentally friendly pest control composition comprising at least two active ingredients and a UV photo degradation arrester derived from biological sources like microorganisms and plants.

Another object of the present invention is to provide combination of insecticides that possess an enhanced efficacy over the individual active compounds used in isolation.

Another object of the present invention is to provide an insecticide combination and a UV arrestor that causes an enhanced greening of the crops to which it is administered.

Yet another object of the present invention is to provide a combination that results into reduced pest pressure in the crops to which it is applied.

Yet another object of the present invention is to provide a combination that achieves increased yield in the crops to which it is applied.

5 Yet another object of the present invention is to provide a combination which enhances the protection to plants from attack or infestation by insects.

### **SUMMARY OF THE INVENTION**

10 Accordingly, in one aspect, the present invention provides a novel, stable, synergistic, environmentally friendly, broad spectrum, economic and safer combination of at least two insecticidal active ingredients and a UV photo degradation and process for preparation thereof.

15 In yet another aspect, the present invention provides an insecticidal combination comprising a first active ingredient being Clothianidin, second active ingredient being Chlorantraniliprole, and a photo degradation arrester, wherein said UV photo degradation arrester is selected from the group consisting of kojic acid, TEPA(Tetra ethyl pentaamine)and DABCO (1,4-Diazabicyclo [2.2.2] octane) which arrests the photo degradation of the formulation and increases the efficacy of the formulation for a duration of up to 8 to120 hours.

It is yet another object of the present invention to provide a pesticidal composition that is effective in controlling target insects in Rice crop.

### **DETAILED DESCRIPTION OF THE INVENTION**

20 Those skilled in the art will be aware that the invention described herein is subject to variations and modifications other than those specifically described. It is to be understood that the invention described herein includes all such variations and modifications. The invention also includes all such steps, features, compositions and compounds referred to or indicated in this specification, individually or collectively, and all combinations of any two or more of said steps  
25 or features.

Unless otherwise specified, all terms used in disclosing the invention, including technical and scientific terms, have the meaning as commonly understood by one of the ordinary skill in the art to which the invention belongs. For further guidance, term definitions may be included to better appreciate the teaching of the present invention.

As used herein, the term “plant” refers to any plant or part thereof including aerial and subterranean parts of the plant. It is contemplated that the parts of the plant may be, for example, flowers, fruits or vegetables, shoots, leaves, needles, stalks, stems, fruiting bodies, seeds also roots and that parts of the plants may or may not be attached to the remainder of the plant.

The term ‘disease control’ as used herein denotes control and prevention of a disease. Controlling effects include all deviation from natural development, for example: killing, retardation, decrease of the pests. The term ‘plants’ refers to all physical parts of a plant, including seeds, seedlings, saplings, roots, tubers, stems, stalks, foliage and fruits.

The term “locus” of a plant as used herein is intended to embrace the place on which the plants are growing, where the plant propagation materials of the plants are sown or where the plant propagation materials of the plants will be placed into the soil. The term “plant propagation material” is understood to denote generative parts of a plant, such as seeds, vegetative material such as cuttings or tubers, roots, fruits, tubers, bulbs, rhizomes and parts of plants, germinated plants and young plants which are to be transplanted after germination or after emergence from the soil. These young plants may be protected before transplantation by a total or partial treatment by immersion.

It is to be understood that the singular forms “a,” “an,” and “the” include plural referents unless the context clearly dictates otherwise.

Features that are described and/or illustrated with respect to one embodiment may be used in the same way or in a similar way in one or more other embodiments and/or in combination with or instead of the features of the other embodiments.

It should be emphasized that the term “comprises/comprising” when used in this specification is taken to specify the presence of stated features, steps or components but does not preclude the presence or addition of one or more other features, steps, components or groups thereof.

The term “insects” as used herein, includes all organisms in the class “Insecta.”

The term “insecticidal” as used herein, refers to the ability of a pesticide to increase mortality or inhibit growth rate of insects.

The terms “weight percent”, “wt-%”, “percent by weight”, “% by weight” and variations thereof, as used herein, refer to the concentration of a substance as the weight of that substance divided

by the total weight of the composition and multiplied by 100. It is understood that, as used here, "percent", "%", and the like are intended to be synonymous with "weight percent", "wt. %", etc.

The present invention provides an insecticidal composition comprising a) Clothianidin as the first active ingredient b) Chlorantraniliprole as the second active ingredient and c) Kojic acid as a UV photo degradation arrester, wherein Clothianidin is present in an amount ranging from 5-20% w/w, Chlorantraniliprole is present in an amount ranging from 4-12% w/w and Kojic acid is present in an amount ranging from 0.1 to 4% the insecticidal composition. In a preferred embodiment, Clothianidin is present in an amount of 10% w/w, Chlorantraniliprole is present in an amount of 06% w/w and Kojic acid is present in an amount of 02% w/w of the insecticidal composition.

In an embodiment, the insecticidal composition of the present invention comprising the said actives exhibits synergistic effect in control of insects in agricultural crops, particularly in Rice crop.

In an embodiment, the insecticidal composition of the present invention further comprises at least one agriculturally acceptable excipient. In an embodiment, the at least one excipient is selected from the group consisting of at least a wetting agent, at least a dispersing agent, at least a defoaming agent, at least a binder, at least a suitable carrier, and combinations thereof. In a preferred embodiment, the insecticidal composition is in the form of a wettable granule formulation further comprising at least a wetting agent, at least a dispersing agent, at least a defoaming agent, at least a binder, and at least a suitable carrier.

In an embodiment, the at least a dispersing agent is selected from the group comprising sodium lignosulphonates, sodium naphthalene sulphonate- formaldehyde condensates, aliphatic alcohol ethoxylates, tristyrylphenol ethoxylates and esters, ethylene oxide/propylene oxide block copolymers. In a preferred embodiment, the at least a dispersing agent is sodium salt of naphthalene sulfonate condensate. The at least a dispersing agent weight concentration in said insecticidal composition is in the range of 3-8%w/w

In an embodiment, the at least a wetting agent is selected from the group consisting of blend of alkyl naphthalene sulfonate, sodium salt, sodium laurel sulphate, and combinations thereof. In a preferred embodiment, the at least a wetting agent is sodium laurel sulphate. The at least a wetting agent weight concentration in said insecticidal composition is in the range of 1-5% w/w.

In an embodiment, the at least an anti-foaming is selected from the group consisting of silicone emulsions, long-chain alcohols, fatty acids, organic fluorine compounds, and combinations thereof. The at least an anti-foaming agent weight concentration in said insecticidal composition is in the range of 0.2-1%w/w.

5 In an embodiment, the at least a carrier is selected from the group consisting of dextrose, lactose, soluble starch, galactose, amyloextrin, ammonium sulfate, maltose, mannitol, sucrose, sorbitol, china clay, and combinations thereof.

10 In an embodiment, the insecticidal composition is formulated in a form selected from the group comprising water-soluble concentrates (SL), emulsifiable concentrates (EC), emulsions (EW), micro-emulsions (ME), Suspension concentrates (SC), oil-based suspension concentrates (OD), flowable suspensions (FS), water-dispersible granules (WG), water-soluble granules (SG), wettable powders (WP), water soluble powders (SP), granules (GR), encapsulated granules (CG), fine granules (FG), macrogranules (GG), dry flowables (DF), aqueous Suspo-emulsions (SE), capsule suspensions (CS) and microgranules (MG). In a preferred embodiment, the insecticidal  
15 composition is in the form of suspension concentrates (SC), water-dispersible granule (WDG) and wettable powder (WP).

In a more preferred embodiment, the insecticidal composition of the present invention is formulated in the form of water dispersible granule (WDG) comprising a wetting agent, a dispersing agent, a defoaming agent, a binder, and a suitable carrier.

20 The present invention also provides a process of preparing a Insecticidal composition comprising a) Clothianidin b) chlorantraniliprole and c) kojic acid wherein Clothianidin propargyl is present in an amount ranging from 5-20% w/w Chlorantraniliprole is present in an amount ranging from 4-12% w/w and Kojic acid is present in an amount ranging from .1-4%% w/w of the Insecticidal composition; as a water-dispersible formulation, said process comprising: (1) preparing a blend  
25 of clothianidin, chlorantraniliprole, Kojic acid , and suitable agriculturally acceptable excipients to obtain a first pre-mix; (2) grinding the first pre-mix by jet-milling to obtain a second pre-mix having mean particle size of less than 10 microns; (3) preparing a dough from the second pre-mix; (4) subjecting the second pre-mix to an extruder to obtain granules; and (5) drying the granules to obtain the water-dispersible formulation.

30 **EXAMPLES**

The examples below are given solely for the purpose of illustration and are not to be construed as limitations of the present invention, as many variations thereof are possible without departing from the spirit and scope of the invention.

**Example 1: Preparation of insecticidal composition as Emulsion concentrates (EC)**

5 Table 1 below provides the insecticidal composition as described in the present specification, formulated as a Emulsion concentrates (EC)

Table 1(a) Composition of the insecticidal composition (CI 6102EC)

Ingredient	Percentage w/w
Chlorantraniliprole technical (Basis of 100%)	06.00%
Clothianidin technical (Basis of 100%)	10.00%
Kojic acid (Basis of 100%)	02.00% w/w
PPG (Poly Propylene glycol)	04.00% w/w
(A) Non-ionic/Anionic surfactant emulsifier (A: Blend of sulphonated and ethoxylated hydrocarbons) (B: Blend of sulphonated and ethoxylated hydrocarbons)	12.00 %
NMP	25.00%
DMSO	15.00%
C-9	Q.S to make
<b>Total</b>	100.00 w/w

**Preparation method of EC:**



The desired quantity of the chlorantraniliprole were weighed and dissolved in DMSO and NMP (full forms?). Clothianidin and kojic acid were added in the mixture slowly followed by adding C-9 solvent into it. The mixture was then subjected to mixing vessel and mixed it for 30 min at the rate 1000 rpm. Then the mixture was then checked for quality parameters.

- 5 The EC formulation as described above in Table 1(a) was tested for the below mentioned quality parameters as listed.

Table 1(c): Quality parameters of EC formulation

Parameter	Desired quality
Description/ physical appearance	The material is emulsion form.
A.I. Content-	Chlorantraniliprole -6.0% (+ 10 & -05) Clothianidin -17% (+5 & -05) Kojic acid -0.53% (+10 & -05)
PH range	6.0 to 8.0
Emulsion stability	Emulsion remains stable after 60 min
Persistent foam	60 ml after 1 min
Acidity as H <sub>2</sub> SO <sub>4</sub>	0.5% max

**Example 2: Preparation of insecticidal composition as Wettable dispersible granules (CI 6012 WDG)**

- 10 Table 2(a) below provides a insecticidal composition as described in the present specification, formulated as a Wettable Dispersible granules (WDG)

Table 2(a) Composition of the insecticidal composition (CI 6102 WDG)

Ingredient	Tentative percentage w/w
Chlorantraniliprole Technical (basis of 100%)	06.00 m/m

Ingredient	Tentative percentage w/w
Clothianidin technical (basis of 100%)	10% m/m
Kojic acid (100% basis)	02.00% m/m
Sodium bi-carbonate	01.00% m/m
Citric acid	00.50% m/m
Dispersing agent (Sodium salt of naphthalene sulfonate condensate )	05.00% m/m
Wetting agent (Sodium laurel sulphate)	02.00% m/m
Antifoam	00.05% m/m
PPT Silica	05.00% m/m
China clay	QS to make % m/m
Total Quantity	100.00% m/m

#### Preparation method of WDG

The desired quantity of the active ingredients and excipients were weighed and mixed in a blender. The mixture was then subjected to grinding through a jet mill and grinding was carried out until a mean particle size of below 10 micron was obtained. After grinding, the homogenous mix was again put in a blender and a dough was prepared by dough mixer. After this step, granules were prepared by carrying the dough through an extruder. The granules thus prepared were then dried by using hot air or and oven. The dried granules were then checked for quality parameters.

The WDG formulation as described above in Table 2(a) was tested for the below mentioned quality parameters as listed.

Table 2(b): Quality parameters of WDG formulation

Parameter	Desired quality
Description/ physical appearance	The material should consist of dry, free flowing granules, which should wet on mixing with water, thereby resulting into a solution suitable for spray.  The material shall be free from visible extraneous matter
A.I. Content-	Chlorantraniliprole -6 % m/m ((±5%)  Clothianidin -10%% m/m (+5, -3%)  Kojic acid -2% m/m(+5, -3%)
Wettability	Max. for 120 secs
Suspensibility	Min 60%
Acidity as H <sub>2</sub> SO <sub>4</sub>	0.5% max

### Example 3: Field evaluation of the bio efficacy of the present insecticidal composition

The presently disclosed insecticidal trimix (coded as CI-6102 WG) was tested for its bioefficacy against Insects on Rice crop and variety Pusa -44. The plants were aligned in a plot size of 50 sq. meter, with the spacing of 10 cm between individual crop plants and a spacing of 10 cm between the rows. The trial was laid out in a random block design consisting of a total of 9 treatments in three replications. The trial was conducted at a temp. of 35°C under 70% relative humidity, no winds and under optimum soil moisture conditions. The application of the insecticidal trimix was carried out after 25<sup>th</sup> and 50<sup>th</sup> day of transplanting. For conducting the application 1<sup>st</sup> brood of the stem borer was required.

Measured quantity of the chemical was added to required volume of water @ 375 lit. /ha for spray. The spray tank was filled with ½ the quantity of clean required volume of water and then the measured chemical (according to the dose) was added followed by the rest half quantity of

water. The solution was stirred well before application. Knapsack sprayer fitted with boom along with flood jet nozzle was used to apply the insecticidal solution.

Table 3(a) below provides the treatment details of the Rice crop in the trial experiment conducted. For purposes of convenience, the present composition has been represented by the code "CI-6102 WG".

Treat	Treatment details	Dose a.i./hectare	Dose/hectare
T-1	Untreated	-	-
T-2	CI-6102 WG	50 + 30 + 10	500
T-3	CI-6102 WG	62.5 + 37.5 + 12.5	625
T-4	CI-6102 WG	75 + 45 + 15	750
T-5	CI-6102 WG	50 + 30	500
T-6	CI-6102 WG	62.5 + 37.5	625
T-7	CI-6102 WG	75 + 45	750
T-8	Clothianidin 50 WDG	125	250
T-9	Chlorantraniliprole 18.50 SC	69.37	375

**Note:** CI-6102 WG (Clothianidin 10% + Chlorantraniliprole 06% + Kojic acid 2% WG

CI-6102 WG (Clothianidin 10% + Chlorantraniliprole 06% WG

Table 3(b) below provides the treatment details of the Rice crop in the trial experiment conducted. For purposes of convenience, the present composition has been represented by the code "CI-0741WG".

Treat	Treatment details	Dose a.i./hectare	Dose/hectare
T-1	Untreated	-	-
T-2	CI--0741 WG	35 + 20 + 5	500

Treat	Treatment details	Dose a.i./hectare	Dose/hectare
T-3	CI--0741 WG	43.75 + 25 + 6.25	625
T-4	CI--0741 WG	52.5 + 30 + 7.5	750
T-5	CI--0704 WG	35 + 20	500
T-6	CI--0704 WG	43.75 + 25	625
T-7	CI--0704 WG	52.5 + 30	750
T-8	Clothianidin 50 WDG	125	250
T-9	Chlorantraniliprole 18.50 SC	69.37	375

**Note**

CI--0741 WG (Clothianidin 07 % + Chlorantraniliprole 04% + Kojic acid 1% WG)

CI--0704 WG (Clothianidin 07% + Chlorantraniliprole 04% WG)

Table 3(c) below provides the treatment details of the Rice crop in the trial experiment conducted. For purposes of convenience, the present composition has been represented by the code "CI-1483WG".

Treat	Treatment details	Dose a.i./hectare	Dose/hectare
T-1	Untreated	-	-
T-2	CI-1483 WG	70 + 40 + 15	500
T-3	CI-1483 WG	87.5 + 50 + 18.75	625
T-4	CI-1483 WG	105 + 60 + 22.5	750
T-5	CI-1483 WG	70 + 40	500
T-6	CI-1483 WG	87.5 + 50	625

<b>T-7</b>	CI-1483 WG	105 + 60	750
<b>T-8</b>	Clothianidin 50 WDG	125	250
<b>T-9</b>	Chlorantraniliprole 18.50 SC	69.37	375

**Note**

Sample-1 CI-1483 WG (Clothianidin 14 % + Chlorantraniliprole 08% + Kojic acid 3% WG

Sample-2 CI-1408 WG (Clothianidin 14% + Chlorantraniliprole 08% WG

**EVALUATION OF BIO-EFFICACY IN RICE CROP**

- 5 **Stem borer count:** The number of counts were conducted on dead hearts from whole plot of each treatment at 20 days after 1<sup>st</sup> and 2<sup>nd</sup> application

Stem borer control: The percent stem borer control was determined based on no. of live dead hearts at 20 days after 1<sup>st</sup> and 20 days after second application as per following:

Percent Insect control = IC – IT

$$10 \quad \frac{\text{.....} \times 100}{IC}$$

Where IC = No. of Dead hearts in control plot

WT = No. of Dead hearts in treated plot

Table 4(a) Effect of insecticidal treatment on stem borer at 20 Days after 1<sup>st</sup> and 2<sup>nd</sup> application

Treatments	Dose (a.i./ha)	Dead hearts and Percent control over Untreated (Average 10 Hills/Treatments)			
		20 DA1A	% Control	20 DA2A	% Control
Untreated	-	55.5	-	70.0	-
CI-6102 WG (Treat -1)	50 + 30 + 10	7.5	74.77	19.0	72.85

Treatments	Dose (a.i./ha)	Dead hearts and Percent control over Untreated (Average 10 Hills/Treatments)			
		20 DA1A	% Control	20 DA2A	% Control
CI-6102 WG (Treat -2)	<b>62.5 + 37.5 + 12.5</b>	<b>4.5</b>	<b>78.37</b>	<b>18.0</b>	<b>74.28</b>
CI-6102 WG (Treat -3)	75 + 45 + 15	4.0	81.98	16.0	77.14
CI-6102 WG (Treat-4)	50 + 30	16.0	71.17	17.0	75.71
CI-6102 WG (Treat-5)	62.5 + 37.5	12.0	78.37	13.0	81.42
CI-6102WG (Treat-6)	75 + 45	9.0	83.78	11.0	84.28
Clothianidin 50 WDG	125	15.5	72.07	18.0	70.0
Chlorantraniliprole 18.50 SC	69.37	8.5	84.68	10.5	85.0

Table 4(a) shows the graphical representation of the percent control achieved at 21 days after various treatments T1-T-7 for the Dead hearts. T2 (62.5 + 37.5 + 12.5) gm a.i per hectare showing maximum percent control of Rice stem borer.

No plant phytotoxicity was observed in case of CI-6102 WG. Data is shown in below tables 4(b):

5 This dose represents the recommended dosages of each of Clothianidin, chlorantraniliprole, and Kojic acid respectively. It is to be understood that under regulatory conditions, the dosage amount of each of Clothianidin, chlorantraniliprole, and Kojic acid is not to be exceeded in real-world application.

#### **Example 4: Evaluation of phytotoxicity of the present insecticidal composition**

Visual observations were recorded at 3, 7 and 10 days after application (DAA) of testing products. The parameters observed were leaf injury on tip/surface, necrosis, vein clearing, epinasty, hyponasty and wilting. The score scale (1-10) followed for leaf injury on tips/surface is given below in Table ...

5 **Phytotoxicity symptoms scoring and rating for leaf injury on tip/surface**

Leaf injury on tips/surface	Rating
0-10%	1
11-20%	2
21-30%	3
31-40%	4
41-50%	5
51-60%	6
61-70%	7
71-80%	8
81-90%	9
91-100%	10

20 **Phytotoxicity studies:**

Table-4(b): Study conducted to assess the phytotoxic effect after various treatments on soyabean crop at 3 DAA

Treatments	Treatment details	Dose/hectare	3 DAA					
			L	N	V	E	H	W
T-1	Untreated	-	-	-	-	-	-	-



<b>T-2</b>	CI-6102 WG	50 + 30 + 10	0	0	0	0	0	0
<b>T-3</b>	CI-6102 WG	62.5 + 37.5 + 12.5	0	0	0	0	0	0
<b>T-4</b>	CI-6102 WG	75 + 45 + 15	0	0	0	0	0	0
<b>T-5</b>	CI-6102 WG	50 + 30	0	0	0	0	0	0
<b>T-6</b>	CI-6102 WG	62.5 + 37.5	0	0	0	0	0	0
<b>T-7</b>	CI-6102 WG	75 + 45	0	0	0	0	0	0
<b>T-8</b>	Clothianidin 50 WDG	125	0	0	0	0	0	0
<b>T-9</b>	Chlorantraniliprole 18.50 SC	69.37	3	0	0	0	0	0

DAA – Days after application,

L- Leaf injury on tips/surface, N-Necrosis, V- Vein clearing, E- Epinasty, H- Hyponasty, W-wilting

Table-4 (c): Phytotoxic effect of various treatments on Rice crop after 7 DAA

<b>Treatments</b>	<b>Treatment details</b>	<b>Dose/hectare</b>	<b>7 DAA</b>					
			<b>L</b>	<b>N</b>	<b>V</b>	<b>E</b>	<b>H</b>	<b>W</b>
<b>T-1</b>	Untreated	-	-	-	-	-	-	-
<b>T-2</b>	CI-6102 WG	50 + 30 + 10	0	0	0	0	0	0
<b>T-3</b>	CI-6102 WG	62.5 + 37.5 + 12.5	0	0	0	0	0	0
<b>T-4</b>	CI-6102 WG	75 + 45 + 15	0	0	0	0	0	0
<b>T-5</b>	CI-6102 WG	50 + 30	0	0	0	0	0	0
<b>T-6</b>	CI-6102 WG	62.5 + 37.5	0	0	0	0	0	0

<b>T-7</b>	CI-6102 WG	75 + 45	0	0	0	0	0	0
<b>T-8</b>	Clothianidin 50 WDG	125	0	0	0	0	0	0
<b>T-9</b>	Chlorantraniliprole 18.50 SC	69.37	3	0	0	0	0	0

DAA – Days after application,

L- Leaf injury on tips/surface, N-Necrosis, V- Vein clearing, E- Epinasty, H- Hyponasty, W-wilting

Table-4(d) Phytotoxic effect of various treatments on Rice crop after 10 DAA

Treatments	Treatment details	Dose/hectare	10 DAA					
			L	N	V	E	H	W
<b>T-1</b>	Untreated	-	-	-	-	-	-	-
<b>T-2</b>	CI-6102 WG	50 + 30 + 10	0	0	0	0	0	0
<b>T-3</b>	CI-6102 WG	62.5 + 37.5 + 12.5	0	0	0	0	0	0
<b>T-4</b>	CI-6102 WG	75 + 45 + 15	0	0	0	0	0	0
<b>T-5</b>	CI-6102 WG	50 + 30	0	0	0	0	0	0
<b>T-6</b>	CI-6102 WG	62.5 + 37.5	0	0	0	0	0	0
<b>T-7</b>	CI-6102 WG	75 + 45	0	0	0	0	0	0
<b>T-8</b>	Clothianidin 50 WDG	125	0	0	0	0	0	0
<b>T-9</b>	Chlorantraniliprole 18.50 SC	69.37	3	0	0	0	0	0

DAA – Days after application,

**Example 5****Bio efficacy studies with CI-0741 WG (Clothianidin 07% + Chlorantraniliprole 14% + Kojic acid 1% WG)**

In further trials, the insecticidal combination was tested at varied percent active ingredient (a.i.) content. Effect of insecticidal treatment on stem borer at 20 days after application was tested for CI- 0741 WG comprising Clothianidin 07% w/w + Chlorantraniliprole 14% w/w + Kojic acid 1% w/w

Table- 5(a)Effect of Insecticide treatment on stem borer at 20 days after 1<sup>st</sup> and 2<sup>nd</sup> Application - CI-0741 WG

Treatments	Dose (a.i./ha)	Dead hearts and Percent control over Untreated (Average 10 Hills/Treatments)			
		20 DA1A	% Control	20 DA2A	% Control
Untreated	-	55.5	-	70.0	-
CI-0741 WG	35 + 20 + 5	14	74.77	19.0	72.85
CI -0741 WG	43.75 + 25 + 6.25	12	78.37	18.0	74.28
CI -0741 WG	52.5 + 30 + 7.5	10.0	81.98	16.0	77.14
CI -0741 WG	35 + 20	22.0	60.36	29.0	58.57
CI -0741 WG	43.75 + 25	19.0	65.76	27.0	61.42
CI -0741 WG	52.5 + 30	18.0	67.56	25.0	64.28
Clothianidin 50 WDG	125	15.5	72.07	18.0	70.0

Treatments	Dose (a.i./ha)	Dead hearts and Percent control over Untreated (Average 10 Hills/Treatments)			
		20 DA1A	% Control	20 DA2A	% Control
Chlorantraniliprole 18.50 SC	69.37	8.5	84.68	10.5	85.0

As seen in Table 5(a) showing maximum percent of control of Rice stem borer in CI- 0741 WG @ 43.75 + 25 + 62.5 gm a.i per hectare with no phytotoxicity (Shown in the below table)

Table-5(b) Phytotoxic effect of various treatments on Rice crop after 3 DAA

Treatments	Treatment details	Dose/hectare	3 DAA					
			L	N	V	E	H	W
<b>T-1</b>	Untreated	-	-	-	-	-	-	-
<b>T-2</b>	CI -0741 WG	35 + 20 + 5	0	0	0	0	0	0
<b>T-3</b>	CI -0741 WG	43.75 + 25 + 6.25	0	0	0	0	0	0
<b>T-4</b>	CI -0741 WG	52.5 + 30 + 7.5	0	0	0	0	0	0
<b>T-5</b>	CI -0741 WG	35 + 20	0	0	0	0	0	0
<b>T-6</b>	CI -0741 WG	43.75 + 25	0	0	0	0	0	0
<b>T-7</b>	CI -0741 WG	52.5 + 30	0	0	0	0	0	0
<b>T-8</b>	Clothianidin 50 WDG	125	0	0	0	0	0	0
<b>T-9</b>	Chlorantraniliprole 18.50 SC	69.37	0	0	0	0	0	0

DAA – Days after application,

L- Leaf injury on tips/surface, N-Necrosis, V- Vein clearing, E- Epinasty, H- Hyponasty, W-wilting

Table- 5 c Phytotoxic effect of various treatments on Rice crop after 7 DAA

Treatments	Treatment details	Dose/hectare	7 DAA					
			L	N	V	E	H	W
<b>T-1</b>	Untreated	-	-	-	-	-	-	-
<b>T-2</b>	CI -0741 WG	35 + 20 + 5	0	0	0	0	0	0
<b>T-3</b>	CI -0741 WG	43.75 + 25 + 6.25	0	0	0	0	0	0
<b>T-4</b>	CI -0741 WG	52.5 + 30 + 7.5	0	0	0	0	0	0
<b>T-5</b>	CI -0741 WG	35 + 20	0	0	0	0	0	0
<b>T-6</b>	CI -0741 WG	43.75 + 25	0	0	0	0	0	0
<b>T-7</b>	CI -0741 WG	52.5 + 30	0	0	0	0	0	0
<b>T-8</b>	Clothianidin 50 WDG	125	0	0	0	0	0	0
<b>T-9</b>	Chlorantraniliprole 18.50 SC	69.37	0	0	0	0	0	0

DAA – Days after application,

L- Leaf injury on tips/surface, N-Necrosis, V- Vein clearing, E- Epinasty, H- Hyponasty, W-wilting

5 Table-5.d Phytotoxic effect of various treatments on Rice crop after 10 DAA

Treatments	Treatment details	Dose/hectare	10 DAA					
			L	N	V	E	H	W
<b>T-1</b>	Untreated	-	-	-	-	-	-	-
<b>T-2</b>	CI -0741 WG	35 + 20 + 5	0	0	0	0	0	0

<b>T-3</b>	CI -0741 WG	43.75 + 25 + 6.25	0	0	0	0	0	0
<b>T-4</b>	CI -0741 WG	52.5 + 30 + 7.5	0	0	0	0	0	0
<b>T-5</b>	CI -0741 WG	35 + 20	0	0	0	0	0	0
<b>T-6</b>	CI -0741 WG	43.75 + 25	0	0	0	0	0	0
<b>T-7</b>	CI -0741 WG	52.5 + 30	0	0	0	0	0	0
<b>T-8</b>	Clothianidin 50 WDG	125	0	0	0	0	0	0
<b>T-9</b>	Chlorantraniliprole 18.50 SC	69.37	0	0	0	0	0	0

DAA – Days after application,

#### Example 6

insecticidal treatment on stem borer at 20 days after 1<sup>st</sup> and 2<sup>nd</sup> application was tested for CI 1483 WG comprising clothianidin 14% + Chlorantraniliprole 08% + Kojic acid 03% WG

5 Table-6.a: Effect of Insecticide treatment on stem borer at 20 days after 1<sup>st</sup> and 2<sup>nd</sup> Application

Treatments	Dose (a.i./ha)	Dead hearts and Percent control over Untreated (Average 10 Hills/Treatments)			
		20 DA1A	% Control	20 DA2A	% Control
Untreated	-	55.5	-	70.0	-
CI-1483 WG	70 + 40 + 15	4.5	91.89	8.5	87.85
CI-1483 WG	87.5 + 50 + 18.75	4.0	92.79	7.5	89.28

CI-1483 WG	105 + 60 + 22.5	3.0	94.59	7.0	90.00
CI-1483 WG	70 + 40	12.0	78.37	14.0	80.00
CI-1483 WG	87.5 + 50	10.0	81.98	12.0	82.85
CI-1483 WG	105 + 60	8.0	85.58	9.0	87.14
Clothianidin 50 WDG	125	15.5	72.07	18.0	70.0
Chlorantraniliprole 18.50 SC	69.37	8.5	84.68	10.5	85.0

As seen in Table 6 a showing maximum percent of control of Rice stem borer in CI- 1483 WG @ 87.5 + 50 + 18.75 gm a.i per hectare gm a.i per hectare with phytotoxicity in higher doses (Shown in the below table ) .

Table-6. b Example-1 Phytotoxic effect of various treatments on Rice crop after 3 DAA

Treatments	Treatment details	Dose/hectare	3 DAA					
			L	N	V	E	H	W
<b>T-1</b>	Untreated	-	-	-	-	-	-	-
<b>T-2</b>	CI-1483 WG	70 + 40 + 15	1	1	0	0	0	0
<b>T-3</b>	CI-1483 WG	87.5 + 50 + 18.75	1	1	0	0	0	0
<b>T-4</b>	CI-1483 WG	105 + 60 + 22.5	1	1	0	0	0	0
<b>T-5</b>	CI-1483 WG	70 + 40	1	1	0	0	0	0
<b>T-6</b>	CI-1483 WG	87.5 + 50	1	1	0	0	0	0

<b>T-7</b>	CI-1483 WG	105 + 60	1	1	0	0	0	0
<b>T-8</b>	Clothianidin 50 WDG	125	0	0	0	0	0	0
<b>T-9</b>	Chlorantraniliprole 18.50 SC	69.37	0	0	0	0	0	0

DAA – Days after application,

L- Leaf injury on tips/surface, N-Necrosis, V- Vein clearing, E- Epinasty, H- Hyponasty, W-wilting

Table-6. c Example-2 Phytotoxic effect of various treatments on Rice crop after 7 DAA

Treatments	Treatment details	Dose/hectare	7 DAA					
			L	N	V	E	H	W
<b>T-1</b>	Untreated	-	-	-	-	-	-	-
<b>T-2</b>	CI-1483 WG	70 + 40 + 15	1	1	0	0	0	0
<b>T-3</b>	CI-1483 WG	87.5 + 50 + 18.75	1	1	0	0	0	0
<b>T-4</b>	CI-1483 WG	105 + 60 + 22.5	1	1	0	0	0	0
<b>T-5</b>	CI-1483 WG	70 + 40	1	1	0	0	0	0
<b>T-6</b>	CI-1483 WG	87.5 + 50	1	1	0	0	0	0
<b>T-7</b>	CI-1483 WG	105 + 60	1	1	0	0	0	0
<b>T-8</b>	Clothianidin 50 WDG	125	0	0	0	0	0	0
<b>T-9</b>	Chlorantraniliprole 18.50 SC	69.37	0	0	0	0	0	0

DAA – Days after application,

L- Leaf injury on tips/surface, N-Necrosis, V- Vein clearing, E- Epinasty, H- Hyponasty, W-wilting



Table-6. d Example-3 Phytotoxic effect of various treatments on Rice crop after 10 DAA

Treatments	Treatment details	Dose/hectare	10 DAA					
			L	N	V	E	H	W
<b>T-1</b>	Untreated	-	-	-	-	-	-	-
<b>T-2</b>	CI-1483 WG	70 + 40 + 15	0	0	0	0	0	0
<b>T-3</b>	CI-1483 WG	87.5 + 50 + 18.75	0	0	0	0	0	0
<b>T-4</b>	CI-1483 WG	105 + 60 + 22.5	1	1	0	0	0	0
<b>T-5</b>	CI-1483 WG	70 + 40	0	0	0	0	0	0
<b>T-6</b>	CI-1483 WG	87.5 + 50	0	0	0	0	0	0
<b>T-7</b>	CI-1483 WG	105 + 60	1	1	0	0	0	0
<b>T-8</b>	Clothianidin 50 WDG	125	0	0	0	0	0	0
<b>T-9</b>	Chlorantraniliprole 18.50 SC	69.37	0	0	0	0	0	0

DAA – Days after application,

L- Leaf injury on tips/surface, N-Necrosis, V- Vein clearing, E- Epinasty, H- Hyponasty, W-wilting

#### Example -7: Phytotoxicity of 2 different formulations (CI-6102 WG & CI-6102 EC)

5 Table-7. a Example-1 Phytotoxic effect of 2 different formulations of CI-6102 WG & CI-6102 EC on Rice crop after 3 DAA

Treatments	Treatment details	Dose/hectare	3 DAA					
			L	N	V	E	H	W
<b>T-1</b>	Untreated	-	-	-	-	-	-	-
<b>T-2</b>	CI-6102 WG	50 + 30 + 10	0	0	0	0	0	0

<b>T-3</b>	CI-6102 WG	62.5 + 37.5 + 12.5	0	0	0	0	0	0
<b>T-4</b>	CI-6102 WG	75 + 45 + 15	0	0	0	0	0	0
<b>T-5</b>	CI-6102 EC	50 + 30 + 10	1	3	1	1	0	0
<b>T-6</b>	CI-6102 EC	62.5 + 37.5 + 12.5	3	3	1	1	0	0
<b>T-7</b>	CI-6102 EC	75 + 45 + 15	3	3	1	1	0	0

DAA – Days after application,

L- Leaf injury on tips/surface, N-Necrosis, V- Vein clearing, E- Epinasty, H- Hyponasty, W-wilting

As seen in Table 11, the trimix of the present invention in WG form preferably shows no phytotoxic effect on rice crop as compared to EC form, which shows moderate phytotoxicity in terms of necrosis, and measurable phytotoxicity to leaf tips, and veins, suggesting that the WG formulation is safer and suitable for application as compared to EC formulation

Table-7. b Example-1 Phytotoxic effect of 2 different formulations of CI-6102 WG & CI-6102 EC on Rice crop after 7 DAA

Treatments	Treatment details	Dose/hectare	7 DAA					
			L	N	V	E	H	W
<b>T-1</b>	Untreated	-	-	-	-	-	-	-
<b>T-2</b>	CI-6102 WG	72 + 132 + 8.4	0	0	0	0	0	0
<b>T-3</b>	CI-6102 WG	78 + 143 + 9.1	0	0	0	0	0	0
<b>T-4</b>	CI-6102 WG	84 + 154 + 9.8	0	0	0	0	0	0
<b>T-5</b>	CI-6102 EC	72 + 132 + 8.4	1	3	1	1	0	0
<b>T-6</b>	CI-6102 EC	78 + 143 + 9.1	3	3	1	1	0	0

Treatments	Treatment details	Dose/hectare	7 DAA					
			L	N	V	E	H	W
<b>T-7</b>	CI-6102 EC	84 + 154 +9.8	3	3	1	1	0	0

DAA – Days after application,

L- Leaf injury on tips/surface, N-Necrosis, V- Vein clearing, E- Epinasty, H- Hyponasty, W-wilting

As seen in Table ... the trimix of the present invention in WG form preferably shows no phytotoxic effect compared to EC form, which across all dosages shows moderate phytotoxicity in terms of necrosis and leaf tip damage, while measurable damage to veins, suggesting that the WG formulation is safer and suitable for application compared to EC formulation

Table-7. c Example-1 Phytotoxic effect of 2 different formulations of CI-6102 WG & CI-6102 EC on Rice crop after 10 DAA

Treatments	Treatment details	Dose/hectare	10 DAA					
			L	N	V	E	H	W
<b>T-1</b>	Untreated	-	-	-	-	-	-	-
<b>T-2</b>	CI-6102 WG	72 + 132 + 8.4	0	0	0	0	0	0
<b>T-3</b>	CI-6102 WG	78 + 143 + 9.1	0	0	0	0	0	0
<b>T-4</b>	CI-6102 WG	84 + 154 +9.8	0	0	0	0	0	0
<b>T-5</b>	CI-6102 EC	72 + 132 + 8.4	0	0	0	0	0	0
<b>T-6</b>	CI-6102 EC	78 + 143 + 9.1	0	0	0	0	0	0
<b>T-7</b>	CI-6102 EC	84 + 154 +9.8	1	0	0	0	0	0

DAA – Days after application,

L- Leaf injury on tips/surface, N-Necrosis, V- Vein clearing, E- Epinasty, H- Hyponasty, W-wilting

As seen in Table 7.(c), the trimix of the present invention in WG form preferably shows no phytotoxic effect compared to EC form, which across all dosages shows measurable phytotoxicity as evaluated by leaf injury to tips/surface, necrosis and vein clearing, suggesting that the WG formulation is safer and suitable for application compared to EC formulation.

5 Overall, the present invention provides an insecticidal trimix composition comprising the actives a) Clothianidin as the first active ingredient b) Chlorantraniliprole as the second active ingredient and c) Kojic acid as the UV photo degradation arrester, wherein clothianidin is present in an amount ranging from 1-25% w/w, Chlorantraniliprole is present in an amount ranging from 5-45% w/w and Kojic acid is present in an amount ranging from 1-30% w/w, which exhibits  
10 surprising and unexpected synergism/functional interrelationship especially when applied to Rice crop for control of insects. The net combined effect of the trimix is better than the effect of each of the actives if they were to be applied concurrently at the recommended dosages. The same is evidenced from the data presented herein, where the total concentration of the actives in the trimix composition is significantly less than the concentration of actives if they were to be  
15 applied as per recommended dosages. Furthermore, as per the trial data, a WG formulation of the claimed composition shows better safety profile than EC.

**I/We Claim:**

1. An insecticidal composition comprising a) Clothianidin as first active ingredient b) Chlorantraniliprole as second active ingredient and c) Kojic acid as UV photodegradation arrester, wherein Clothianidin is present in an amount ranging from 5-20% w/w, Chlorantraniliprole is present in an amount ranging from 4-12% w/w and Kojic acid is present in an amount ranging from 0.1-4%w/w of the insecticidal composition.
2. The insecticidal composition as claimed in claim 1, further comprising at least one agriculturally acceptable excipient.
3. The insecticidal composition as claimed in claim 1, wherein said insecticidal composition is formulated in a form selected from the group consisting of water-soluble concentrates (SL), emulsifiable concentrates (EC), emulsions (EW), micro-emulsions (ME), Suspension concentrates (SC), oil-based suspension concentrates (OD), flowable suspensions (FS), water-dispersible granules (WG), water-soluble granules (SG), wettable powders (WP), water soluble powders (SP), dry flowables (DF), granules (GR), encapsulated granules (CG), fine granules (FG), macrogranules (GG), aqueous Suspo-emulsions (SE), capsule suspensions (CS) and microgranules (MG).
4. The insecticidal composition as claimed in claim 3, wherein said insecticidal composition is in the form of wettable granule (WG) formulation.
5. The insecticidal composition as claimed in claim 4, wherein said insecticidal composition further comprises at least a wetting agent, at least a dispersing agent, at least a defoaming agent.
6. The insecticidal composition as claimed in claim 5, wherein said at least a dispersing agent is selected from the group consisting of sodium lignosulphonates, sodium naphthalene sulphonate- formaldehyde condensates, aliphatic alcohol ethoxylates, tristyrylphenol ethoxylates and esters, ethylene oxide/propylene oxide block copolymers, and combinations thereof; and wherein said at least a dispersing agent is present in an amount ranging from 3-8% w/w of the insecticidal composition.
7. The insecticidal composition as claimed in claim 5, wherein said at least a wetting agent is selected from the group consisting of blend of alkyl naphthalene sulfonate, sodium salt, sodium laurel sulphate, and combinations thereof; and wherein said wetting agent is present in an amount ranging from 1-5% w/w of the insecticidal composition.

- 5           **8.** The insecticidal composition as claimed in claim 5, wherein said at least an defoaming agent is selected from the group consisting of silicone emulsions, long-chain alcohols, fatty acids, organic fluorine compounds, and combinations thereof; and wherein said at least a defoaming agent is present in an amount ranging from 0.2-1% w/w of the insecticidal composition.
- 9.** The insecticidal composition as claimed in claim 1, wherein clothianidin is present in an amount of 10% w/w, Chlorantraniliprole is present in an amount of 6% w/w and Kojic acid is present in an amount of 2% w/w of the insecticidal composition.
- 10          **10.** A method for controlling insects in rice, said method comprising treating rice crop with an insecticidal composition as claimed in claim 1.
- 11.** A process for preparing an insecticidal composition as claimed in claim 1 as a water-dispersible formulation, said process comprising:
- 15           a. preparing a blend of clothianidin, Chlorantraniliprole, Kojic acid, and suitable agriculturally acceptable excipients to obtain a first pre-mix;
- b. grinding the first pre-mix by jet-milling to obtain a second pre-mix having mean particle size of less than 10 microns;
- c. preparing a dough from the second pre-mix;
- d. subjecting the second pre-mix to an extruder to obtain granules; and
- e. drying the granules to obtain the water-dispersible formulation.

## INTERNATIONAL SEARCH REPORT

International application No.  
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A. CLASSIFICATION OF SUBJECT MATTER  
A01N51/00 Version=2021.01

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)

PatSeer, IPO Internal Database

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	WO2019236717A1 NOVOZYMES BIOAG AS (DK) 12 DECEMBER 2019 (2019-12-12) paragraphs [006], [0022]; page 6 lines 16-27; Experiment 2; table 2; claims 6 and 26	1-11
Y	US4956353A US AGRICULTURE (US); 11 SEPTEMBER 1990; (1990-09-11) abstract, page 2 lines 5-42, claims 1, 9, 14-15; NONE	1-11

☐ Further documents are listed in the continuation of Box C. ☒ See patent family annex.

* Special categories of cited documents:	"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
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**INTERNATIONAL SEARCH REPORT**  
Information on patent family members

International application No.  
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Citation	Pub.Date	Family	Pub.Date
WO 2019236717 A1	12-12-2019	EP 3801017 A1	14-04-2021
		US 2021251238 A1	19-08-2021
		CA 3101519 A1	12-12-2019