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(71) Applicant: **INSECTICIDES (INDIA) LTD.** [IN/IN]; 401-402 Lusa Tower, Azadpur Commercial Complex, Delhi 110033 (IN).

(72) Inventors: **AGGARWAL, Rajesh**; 401-402 Lusa Tower, Azadpur Commercial Complex, Delhi 110033 (IN). **ROHELA, L.C.**; 401-402 Lusa Tower, Azadpur Commercial Complex, Delhi 110033 (IN).

(74) Agent: **GARG, Vidisha** et al.; Anand & Anand Advocates, B-41, Nizamuddin East, New Delhi 110013 (IN).

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(54) Title: FUNGICIDAL COMPOSITIONS AND METHODS RELATED THERETO

(57) Abstract: There is a need in the art for a pesticidal composition which exhibit excellent fungicidal activity towards a wide range of agricultural pests even at a low concentration, better selectivity, lower undesirable impact, lower production and market cost etc. The present invention satisfies the existing needs, as well as others, and generally overcome the deficiencies found in the prior art.

Fungicidal compositions and methods related thereto

FIELD OF THE INVENTION

The present invention relates to a pesticidal composition comprising combination of fungicide and bactericide for effective management of fungal and bacterial diseases of field crops. Specifically, the present invention relates to pesticidal compositions comprising a bactericide, Kasugamycin and a fungicide, Thifluzamide in various types of formulations.

BACKGROUND OF THE INVENTION

This section includes information that may be useful in understanding the present invention. It is not an admission that any of the information provided herein is prior art or relevant to the present invention, or that any publication specifically or implicitly if referenced is prior art.

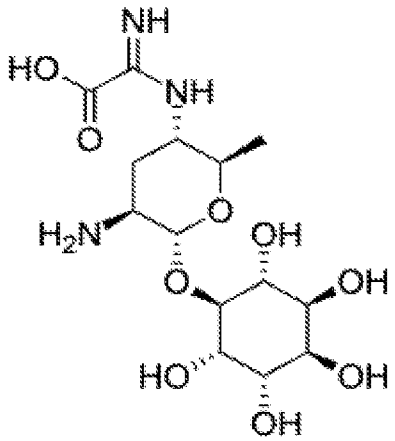
Fungicides and bactericides are an integral and important tool for farmers to control crops diseases, as well as to improve yields and quality of the crops. A wide range of active fungicidal and bactericidal compounds have been developed and used to control pests in the agriculture. These compounds are commercially available in various forms. The efficacy of pesticide compounds is limited in controlling certain pests and the problem of controlling a broad spectrum of pests still exists. In addition, many pesticidal formulations presently in the market are used in relatively high concentrations and volumes which cause high cost, phytotoxic problem, damage to desirable plant species and their residues detrimental to the succeeding crop.

To enable the efficient elimination or controlling of unwanted pests in agriculture and related endeavors, it is desirable to use effective pesticides. Formulations containing pesticides are desirable in agricultural and related endeavors in order to effectively distribute the active ingredient to the area where pest control is desired. Physico-chemical stability is most important in this type of formulation in order to ensure the small amount of the pesticide is fully effective. With crop tolerances decreasing, lower use rates being imposed and resistance being increasingly observed, 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.

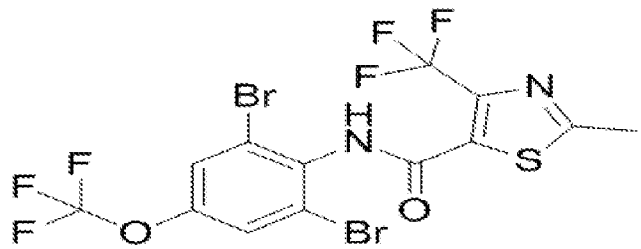
A pesticide suspension is a homogeneous mixture of small solid particles of pesticide suspended in a liquid medium. During periods of non-agitation, the solid particles with a density greater than the density of the liquid medium will settle toward the bottom of the container and homogeneity is not maintained. Similarly, solid particles with a density less than the density of the liquid medium will settle toward the top of the container and homogeneity is not maintained. The loss of homogeneity can cause product failure if it results in non-uniform applications of the pesticide, and plugging of strainers and nozzles used with application equipment. The production

of a stable suspension concentrate formulation most of times are difficult due to the formation of large crystals while large crystals grow larger or form agglomerates in the process known as Ostwald ripening. The drawbacks to Ostwald ripening include limited utility of the formulation because an acceptable shelf-life may not be obtained. For instance, a two year shelf-life is typically needed for a successful commercial product in agrochemicals. Also, crystals that grow too large may plug spray equipment and may not stay suspended in the spray tank or concentrated formulation. Consequently, there is a need for maintaining a stable suspension concentrate with a uniform particle size distribution (i.e. no large crystals) is most important for this type of formulation in order to prevent settling of particles during storage.

Kasugamycin (Ksg) is an aminoglycoside antibiotic that was originally isolated in 1965, from Streptomyces kasugaensis, a Streptomyces strain found near the Kasuga shrine in Nara, Japan. Kasugamycin was discovered by Hamao Umezawa, who also discovered kanamycin and bleomycin, as a drug that prevent growth of a fungus causing rice blast disease. It was later found to inhibit bacterial growth also. Kasugamycin is available as Kasugamycin hydrochloride hydrate and has a good solubility in water.



Thifluzamide is an aromatic amide which usually used to control Rhizoctonia spp. diseases on rice, potatoes, maize, grass and other crops. It has a role as an EC 1.3.5.1 [succinate dehydrogenase (quinone)] inhibitor and an antifungal agrochemical. It is an aromatic amide, an aromatic ether, an organofluorine compound, a member of 1,3-thiazoles, a dibromobenzene and an anilide fungicide.



Thifluzamide is a compound that has high pesticidal activity against leaf spot withering disease. Because the time to use an exterminating agent to exterminate rice leaf spot withering disease in rice plant cultivation is when the paddy field has lesser amount of water, dissolution /diffusion rate of the effective ingredient can affect the exterminating effect significantly. However, since the solubility of thifluzamide in water is extremely low, about 1.6 mg/l (20°C), it is desirable to develop a method that enhances its rate of release in water and improves its efficacy.

There is a need in the art for a pesticidal composition comprising combination of fungicide and bactericide for effective management of fungal and bacterial diseases of field cropswhich in combination exhibit excellent fungicidal and bactericidal activity towards a wide range of agricultural pests even at a low concentration, better selectivity, lower undesirable impact, lower production and market cost etc. The present invention satisfies the existing needs, as well as others, and generally overcome the deficiencies found in the prior art.

SUMMARY OF THE INVENTION

The embodiments of the present invention relate to pesticidal composition comprising a bactericide, Kasugamycin and a fungicide, Thifluzamide which ameliorate many problems such as mentioned herein below:

The embodiments of the present invention provide pesticidal composition comprising kasugamycin and thifluzamide that possess an enhanced efficacy over the individual pesticide whenused in isolation.

Another object of the present invention is to provide pesticidal composition comprising kasugamycin and thifluzamide in a stable suspension concentrate formulation wherein the active Kasugamycin hydrochloride hydrate is soluble in water. When one active is soluble in water, it leads to crystal growth in suspension concentrate formulation. The present invention

demonstrates inhibition of Ostwald ripening in suspension concentrate formulation of Kasugamycin and Thifluzamide on long term storage. In principle to formulate a water based suspension concentrate, the active ingredient must remain insoluble under all temperature conditions.

Another object of the present invention is to provide pesticidal compositions comprising kasugamycin and thifluzamide that causes an enhanced phytotonic effect to the crops to which it is administered.

Another object of the present invention is to provide pesticidal compositions comprising kasugamycin and thifluzamide that causes late senescence to the crop to which it is applied thereby resulting into an increasing yield of the crop.

Yet another object of the present invention is to provide pesticidal compositions comprising kasugamycin and thifluzamide that results into reduced fungal and bacterial disease incidence in the crops to which it is applied.

Another object of the present invention is to provide pesticidal compositions comprising kasugamycin and thifluzamide that increase yield in the crops to which it is applied.

Some or all these and other objects of the invention are can be achieved by way of the invention described hereinafter.

In accordance with an embodiment of the invention, there is provided a pesticidal composition comprising of Kasugamycin and Thifluzamide.

In accordance with an embodiment of the invention, the Kasugamycin can be present in an amount of from about 1% weight to about 10% of weight of all components in the composition. The Thifluzamide can be present in an amount of from about 10% weight to about 50% of weight of all components in the composition.

In accordance with an embodiment of the invention, the weight ratio of Kasugamycin and Thifluzamide relative to each other in the composition is in the range of from about 1:1 to about 1:50.

In accordance with another embodiment of the present invention, there is provided a method of treating fungi and bacteria comprising of applying an effective amount of Kasugamycin in an amount of from about 1% weight to about 10% of all components in the composition and Thifluzamide in an amount of from about 10% weight to about 50% of all components in the composition or/and the weight ratio of Kasugamycin and Thifluzamide relative to each other in

the composition is in the range of from about 1:1 to about 1:50 to a locus where fungal and bacterial control is needed or expected to be needed.

In accordance with one embodiment of the present invention, the composition comprising Kasugamycin and Thifluzamide also contain one or more adjuvants. The adjuvants employed in the composition will depend upon the type of formulation and/or the manner in which the formulation is to be applied by the end user. Depending on the aims to be achieved and the given circumstances, the fungicidal composition according to the invention are formulated into emulsifiable concentrates, suspension concentrates, directly sprayable or dilutable solutions, spreadable pastes, dilute emulsions, sprayable powders, soluble powders, dispersible powders, wettable powders, dusts, granules or encapsulations in polymeric substances.

In accordance with preferred embodiment of the present invention, the composition comprising Kasugamycin and Thifluzamide is formulated in suspension concentrate (SC) form.

Kasugamycin hydrochloride is highly soluble in water and thifluzamide has very poor solubility in water. When the toxicant solubility is high in water, it has tendency to separate out in presence of non compatible ingredients over a period of time and destabilize the suspension and even may form aggregates and exhibit Ostwald ripening and polymorphism. After extensive research, it has unexpectedly found that in suspension concentrate (SC) formulation comprising Kasugamycin and Thifluzamide, the addition of only specific inert components such as ethoxylated branched C11-C14, C13 rich alcohol, acrylic graft polymer formed from condensation of alpha-acrylamido-2-methylpropane sulfonic acid and ethoxylated lauryl methacrylate, smectite clay can achieve the desired stability and fungicidal activity.

The features of the invention will become more apparent from the detailed description set forth herein below.

DESCRIPTION OF THE INVENTION

The following is a description of embodiments of the present invention. The embodiments are in such details as to clearly communicate the invention. However, the amount of detail offered is not intended to limit the anticipated variations of embodiments; on the contrary, the intention is to cover all modifications, equivalents, and alternatives falling within the spirit and scope of the present invention.

As used herein, the term "stable composition" refers to compositions that are stable physically and / or chemically for defined periods of time to the environments in which they are produced, transported and/ or stored. Aspects of "stable compositions" include, but are not limited to: physical stability at temperatures that range from about 0° C. to about 54° C , homogeneity, pourability, liquids that do not exhibit appreciable sedimentation or Ostwald ripening of the

dispersed particles, compositions that form little or no precipitated solids or exhibit phase separation, compositions that readily disperse when poured into a spray tank of water and retain their biological efficacy when applied, for example, by spray application to target pests.

Unless specifically defined otherwise, all technical and scientific terms used herein shall be taken to have the same meaning as commonly understood by one of ordinary skill in the art (e.g., in agricultural chemistry, dendrimer chemistry, pesticide chemistry and formulations, and biochemistry).

Unless the context requires otherwise, throughout the specification which follow, the word "comprise" and variations thereof, such as, "comprises" and "comprising" are to be construed in an open, inclusive sense that is as "including", but not limited to."

As used in the description herein and throughout the claims that follow, the meaning of "a", "an" and "the" includes plural reference unless the context clearly dictates otherwise.

In some embodiments, the numerical parameters set forth in the written description are approximations that can vary depending upon the desired properties sought to be obtained by a particular embodiment.

In accordance with an embodiment of the invention, the active ingredients/compounds used in the present invention are in the form of a free acid, salt, or an ester.

In accordance with an embodiment of the invention, in the compositions comprising Kasugamycin and Thifluzamide in suspension concentrate (SC) form, the wetting agents are selected from the group consisting of aliphatic sulphates in sodium salt form, dioctyl sulfosuccinates sodium salt, taurates, alkyl ethoxylates, nonyl phenol ethoxylates etc.

All of said and various others wetting agents are tested but only ethoxylated branched C11- C13 rich alcohol C14, CAS = 78330-21-9 was found to give better wetting and less foaming as compared to linear alcohol ethoxylated. Only the C13 rich ethoxylated alcohol provided the stabilisation to compositions comprising Kasugamycin and Thifluzamide in suspension concentrate (SC) form.

In accordance with an embodiment of the invention, in the compositions comprising Kasugamycin and Thifluzamide in suspension concentrate (SC) form, the wetting agent is ethoxylated branched C11- C13 rich alcohol C14.

In accordance with an embodiment of the invention, in the compositions comprising Kasugamycin and Thifluzamide in suspension concentrate (SC) form, the dispersing agents are

selected from the group consisting of lignosulphonates, naphthalene sulfonates, phosphate esters of nonyl phenol ethoxylates, amine salts of tristyrene nonyl phenol ethoxylates, methylnaphthalene sulfonic acid formaldehyde condensate sulphates, polycarboxylic acid sodium, saponin, sod. Dodecane sulphonate, alcohol polyoxyethylene ether ammonium, sodium sulphate, etc..

All the above and various others dispersing agents were tried in the compositions comprising Kasugamycin and Thifluzamide in suspension concentrate (SC) form but the best suspension stability was observed by the use of acrylic graft polymer formed from condensation of alpha-acrylamido-2-methylpropane sulfonic acid and ethoxylated lauryl methacrylate.

In accordance with preferred embodiment of the invention, in the compositions comprising Kasugamycin and Thifluzamide in suspension concentrate (SC) form, the dispersing agent is acrylic graft polymer formed from condensation of alpha-acrylamido-2-methylpropane sulfonic acid and ethoxylated lauryl methacrylate.

In accordance with an embodiment of the invention, in the compositions comprising Kasugamycin and Thifluzamide in suspension concentrate (SC) form, the rheology modifiers are selected from silica, bentonite, alumina, smectite clay etc..

For viscosity stabilization in the compositions comprising Kasugamycin and Thifluzamide in suspension concentrate (SC) form, various rheology modifiers such as silica, bentonite, alumina and others were used but the best stabilization was provided by smectite clay.

In accordance with preferred embodiment of the invention, in the compositions comprising Kasugamycin and Thifluzamide in suspension concentrate (SC) form, the rheology modifier is smectite clay.

The pesticidal composition of the present invention may also comprise further pesticide.

The following example of formulation of Kasugamycin and Thifluzamide according to the invention is presented to illustrate, but not to restrict, this invention:

Table 1: Kasugamycin 6% + Thifluzamide 26% SC (w/v)

Component	Concentration (w/v)
Kasugamycin a.i.	6.0 g
Thifluzamide a.i.	26.0 g
C_{11}-C_{14} isoalcohol, C_{13} rich, ethoxylated	2.36 g

Acrylic graft copolymer in propylene glycol	3.54 g
Propylene glycol	7.08 g
Dimethyl siloxane reaction with silica octamethyl cyclotetrasilane	0.59 g
Xanthan gum	0.29 g
1,2 benzisothiazol-3-(2H)-one sodium hydrate	0.03 g
Smectite clay	0.35 g
Phosphoric acid	0.14 g
Water	qs to make 100 ml

2.36 g of ethoxylated branched C11- C13 rich alcohol C14and 3.54 g of acrylic graft copolymer in propylene glycol were added to 40 liter of DM water and mixed to form a solution. 7.08 g of propylene glycol and 0.59 g of octamethyl cyclotetrasilane were added to arrest the foaming. 26 g thifluzamide and 6.9 g of Kasugamycin hydrochloride hydrate (6 g of kasugamycin) were added to the above prepared mixture and mixed to form a uniform suspension. 0.35 g of smectite clay was added followed by 0.03 g of 1,2-benzisothiazol to prevent formation of any foul smell. The above mixed material was pulverized in horizontal pressurized bead mill to obtain particle D90 less than 8 micron. After obtaining the desired particle size, the material was mixed with 14.5 g of 2% aqueous solution of xanthan gum. The material was mixed to obtain stable viscosity of the material (400-600 cps). Finally, pH of the material was adjusted by adding 0.14 g of phosphoric acid and water is added to make it 100 ml.

The compositions comprising Kasugamycin and Thifluzamide of the present invention are found physico-chemical stable and also tested to evaluate the bio-efficacies for the control of the shealth blight and blast diseases on paddy crop. The expected efficacies for active ingredient combinations were determined using Colby's formula and compared with observed efficacies and it was found that the compositions comprising Kasugamycin and Thifluzamide of the present invention were effective against fungal diseases, in fact, synergistic effect was obtained. The yield output of paddy crop and cost benefit ratio obtained by using the composition of Table 1 of the present invention (referred as IIL-KT) is given in Table 2. Further, no phtotoxicity symptoms were recorded on paddy crop.

Although the foregoing invention has been described in some detail by way of illustration and example for purposes of clarity of understanding, it will be readily apparent to those of ordinary skill in the art in light of the teachings of this invention that certain changes and modifications may be made thereto.

Treatment	Dose		Yield Output			Economic Significance					
	g. a.i./ha	Formulation g or ml/ha	1000 Seed wt (g)	Crop yield (q/ha)	Per cent yield increase over check	Rate of product (Rs per lit or kg)	Cost of application (Rs/ha)	Gross profit from produce (Rs/ha)	Additional profit from produce over control (Rs/ha)	Net profit from produce (Rs/ha)	Cost: Benefit Ratio
IIL - KT	18 + 78	300	26.19 (5.12)	46.80 (6.84)	**24.90**	5000	4500	102960	20526	16026	**1: 3.56**
IIL - KT	20.70 + 89.70	345	26.35 (5.13)	48.45 (6.96)	**29.30**	5000	5175	106590	24156	18981	**1:3.67**
Thifluzamide 24% SC	90	375	26.72 (5.17)	42.57 (6.52)	**13.61**	2550	2869	93654	11220	8351	**1:2.91**
Kasugamycin 3 % SL	50	1500	25.31 (5.03)	40.73 (6.38)	**8.70**	710	3195	89606	7172	3977	**1:1.24**
Untreated Control	-	-	24.16 (4.92)	37.47 (6.12)	-	-	-	82434	-	-	-

Table 2: Results of trials conducted to determine yield of paddy grains and cost benefit ratio by using the compositions of the present invention.

We Claim:

1. A pesticidal composition comprising:
   (a) Thifluzamide;
   (b) Kasugamycin; and
   (c) At least one agriculturally acceptable adjuvant.

2. The pesticidal composition as claimed in claim 1, wherein weight ratio of Kasugamycin and Thifluzamide is in a range from about 1:1 to 1:50.

3. The pesticidal composition as claimed in any of the preceding claims, wherein the Thifluzamide is present in an amount of 10-50% by weight of the composition.

4. The pesticidal composition as claimed in any of the preceding claims, wherein the Kasugamycin is present in an amount of 1-10% by weight of the composition.

5. The pesticidal composition as claimed in any of the preceding claims, wherein the composition is formulated as a wettable powder (WP), suspension concentrate (SC), a micro emulsion (ME), an encapsulated suspension (CS), or a water dispersible granule (WG).

6. The pesticidal composition as claimed in any of the preceding claims, wherein the composition is formulated as a suspension concentrate (SC).

7. The pesticidal composition as claimed in any of the preceding claims, wherein said agriculturally acceptable adjuvant includes wetting agent, dispersing agent and rheology modifier.

8. The pesticidal composition as claimed in any of the preceding claims, wherein said wetting agent is ethoxylated branched C11- C13 rich alcohol C14.

9. The pesticidal composition as claimed in any of the preceding claims, wherein said dispersing agent is acrylic graft polymer.

10. The pesticidal composition as claimed in any of the preceding claims, wherein said rheology modifier is smectite clay.

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INTERNATIONAL SEARCH REPORT

International application No.
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A. CLASSIFICATION OF SUBJECT MATTER
A01N43/78,A01N43/16,A01P3/00 Version=2020.01

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, A01P

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)

TotalPatent One, 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.
X	CN 101755825 B (FUJIAN SINO DASHING BIOENGINEERING CO., LTD. [CN]) 20 March 2013 (20.03.2013) Family None Abstract, paragraphs [0023]-[0037], examples 8, 21 and claims 1-3;	1-10
A	US 20040116296 A1 (HAE-JUN PARK [KR] et al.,) 17 June 2004 (17.06.2004) Claims 1-5;	1-10
A	CN 102239852 A (NANNING DEFENGFU CHEMICAL CO., LTD. [CN]) 16 November 2011 (16.11.2011) Family None Abstract and claims 1-6;	1-10
A	CN 105475303 A (SHAANXI MICROBE BIOTECHNOLOGY CO., LTD. [CN]) 13 April 2016 (13.04.2016) Family None Abstract and claims 1-9;	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
"D" document cited by the applicant in the international application
"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	Date of mailing of the international search report
25-09-2020	25-09-2020
Name and mailing address of the ISA/ Indian Patent Office Plot No.32, Sector 14,Dwarka,New Delhi-110075 Facsimile No.	Authorized officer Veera R Kattula Telephone No. +91-1125300200

INTERNATIONAL SEARCH REPORT
Information on patent family members

International application No.
PCT/IB2020/055941

Citation	Pub.Date	Family	Pub.Date
US 20040116296 A1	17-06-2004	AU 2275902 A	01-07-2002
		CN 1209012 C	06-07-2005
		WO 2002049430 A1	27-06-2002
		JP 3809866 B2	16-08-2006