



Señora

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Referencia: Solicitud de Patente de Invención PCT denominada
“**COMPOSICIONES INSECTICIDAS QUE COMPRENDEN
METOXIFENOZIDA, BENZOATO DE EMAMECTINA Y UNO
DE PIMETROZINA, FLUBENDIAMIDA, DELTAMETRINA Y
LAMBDA-CIALOTRINA**”

Asunto: Respuesta al oficio No. 75 de 5 de enero de 2024

Expediente No. NC2021/0011704

Juan Carlos Cuesta Quintero, mayor de edad, identificado como aparece al pie de mi firma, obrando en mi calidad de apoderado de **WILLOWOOD CHEMICALS PRIVATE LIMITED** doy respuesta al Oficio No. 75 de 5 de enero de 2024, bajo los siguientes términos:

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1. De la solicitud de patente

1.1. Reivindicaciones

La Oficina ha solicitado que el biocida mencionado en la reivindicación 1 debería ser enmendado a “una solución acuosa de dipropilenglicol al 20% de 1,2-bencisotiazolim-3-ona”. La enmienda se ha realizado tal y como solicitado, por lo que la objeción ha sido superada mediante enmienda.

La Oficina ha objetado que la reivindicación 6 no está completamente soportada por la descripción originalmente presentada, ya que sólo se ejemplifican composiciones formuladas como concentrados en suspensión. El solicitante toma nota debidamente de que el alcance de la reivindicación debe limitarse a una generalización razonable del tipo de composiciones que se han sintetizado y probado de acuerdo con los ejemplos de la descripción. Por lo tanto, el solicitante ha eliminado numerosos de los tipos de formulación alternativos enumerados en la reivindicación 6 original, según lo solicitado, a una lista de generalizaciones razonables de formulaciones con similitudes entre sí, para así superar esta objeción mediante enmienda.

El examinador sugirió modificar la reivindicación 8 para mencionar únicamente el componente respectivamente listado, junto con el rango de su contenido correspondiente, a fin de evitar materia redundante; el solicitante indica respetuosamente que la reivindicación ha sido modificada según lo solicitado y, por tanto, la objeción ha sido superada mediante dicha modificación.

2. Examen de patentabilidad

2.1. Nivel Inventivo



El examinador ha citado los siguientes documentos del estado de la técnica:

D1: IN201621027690

D2: CN101919386

El Solicitante amablemente se permite señalar que ambos documentos ya fueron citados en la solicitud de patente tal como fue presentada.

Se hace notar que, al evaluar el nivel inventivo, el Examinador está aplicando el método “problema-solución”, siguiendo la *Guía para Examen de Solicitudes de Patente de Invención y Modelo de Utilidad* publicada por la SIC. En esta guía, se describe el correcto análisis del nivel inventivo (2.13.5.1); en particular, se debe aplicar el citado método “problema-solución”, el cual comprende las siguientes etapas:

- 1) Identificar el estado de la técnica cercano a la invención reivindicada,
- 2) Determinar la diferencia entre la invención y estado de la técnica cercano,
- 3) Definir el problema técnico objetivo, y
- 4) Evaluar si la invención reivindicada habría sido obvia para la persona medianamente versada en la materia.

Tal y como indicado en el primer párrafo del apartado 2.13.5.1 de la *Guía*, es importante que se haga “*un análisis objetivo de las divulgaciones previas del estado de la técnica, no influenciadas por el conocimiento que ya se tiene de la invención que se está evaluando*”: se ha de minimizar la subjetividad, y es importante evitar cualquier retrospectiva (“*hindsight*” o “*a posteriori*”) durante el análisis.

Además, con respecto a la etapa 4) arriba indicada, con respecto al segundo documento del estado de la técnica citado, es importante responder a la pregunta de si este documento contiene una enseñanza que **indicaría** a la persona medianamente versada en la materia

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cómo modificar o adaptar el estado de la técnica más cercano para alcanzar la invención evaluada sin realizar ninguna actividad inventiva, y no solamente si este documento *podría indicar* a la persona medianamente versada en la materia cómo modificar el estado de la técnica más cercano.

El Solicitante señala respetuosamente que el documento D1 ya fue citado en el primer Requerimiento, emitido en julio de 2023, en donde este documento se consideró relevante para la actividad inventiva de la presente invención por sí solo, sin combinar su divulgación con ningún otro documento del estado de la técnica. En particular, el Examinador se refirió a una composición específicamente divulgada en D1 que comprende 17,5 % de metoxifenoazida, 1 % de benzoato de emamectina y 7,5 % de bifentrina.

En respuesta a esto, el Solicitante, en resumen, argumentó que D1 no es una divulgación habilitante para ninguna de las combinaciones reivindicadas en la presente solicitud, ya que, aunque se reivindican un total de 58 composiciones diferentes, de hecho solo se divulgan 8 ejemplos específicos, ninguno de los cuales comprende pimetrozina, flubendiamida, deltametrina o lambda-cihalotrina. Como tal, la presente invención es inventiva con respecto a la divulgación de D1: un experto en la técnica, aunque *podría* haber sustituido la bifentrina por uno de pimetrozina, flubendiamida, deltametrina, o lambda-cihalotrina, no *habría* estado motivado para hacerlo por la divulgación de D1, y aún menos habría tenido una expectativa de éxito razonable.

Como ya se indicó en la respuesta al anterior Requerimiento, aunque todos de bifentrina y deltametrina y lambda-cihalotrina son compuestos piretroides, dentro de los piretroides, mientras que la bifentrina pertenece a los piretroides de tipo I, la cihalotrina y la deltametrina pertenece a los piretroides de tipo II. Debido a diferentes modos de acción debido a las



diferencias existentes, un experto en la técnica habría estado bastante motivado para probar otros piretroides de tipo I, en vez de cualquier piretroide de tipo II.

PIRETROIDES DE TIPO I	PIRETROIDES DE TIPO II
Aletrina	Ciflutrina
Bifentrina	Cihalotrina
Permetrina	Cipermetrina
Fenotrina	Deltametrina
Resmetrina	Genvalerato
Sumitrina	Flumetrina
Teflutrina	Fluvalinato
Tetrametrina	Tralometrina

Es decir, un experto medio en la técnica no habría sustituido la bifentrina por ningún piretroide de tipo II, ya que, según su conocimiento de las diferencias entre los dos tipos de piretroides, el experto habría esperado que fueran necesarias experimentaciones más amplias; es decir, un experto en la técnica no habría llegado a la presente invención de una manera obvia, sin necesidad de aplicar una actividad inventiva.

De hecho, el Solicitante entiende que el examinador ha estado de acuerdo en que D1, por sí solo, no es lo suficientemente relevante para la actividad inventiva de la presente invención, ya que se ha citado un nuevo documento del estado de la técnica adicional D2 en este segundo Requerimiento, como supuestamente relevante en combinación con D1. Por lo tanto, en este segundo Requerimiento, el documento D1 se cita como el estado de la técnica más cercano (según el último párrafo de la página 4 del Requerimiento); sin embargo, se objeta la falta de actividad inventiva en vista de la divulgación del documento



D2, el cual ha sido citado indicando que la combinación de la divulgación de D1 con la de D2 habría llevado a un experto en la materia hasta la presente invención.

Como se indicó correctamente en el primer párrafo de la página 5 del Requerimiento, la diferencia entre la reivindicación 1 de la presente invención y la composición desvelada en D1 radica en que en la presente invención los componentes de la composición insecticida se seleccionan de uno de: pimetrozina, flubendiamida, deltametrina y lambda-cihalotrina.

Según el Examinador, el problema técnico objetivo a resolver por la presente invención podría ser establecido del siguiente modo: *¿cómo modificar la composición desvelada en D1 para proporcionar una composición sinérgica alternativa?*

Por lo tanto, según el examinador, la inclusión específica en la composición insecticida de uno de pimetrozina, flubendiamida, deltametrina y lambda-cihalotrina se sugiere por la divulgación de D2, para llegar al objeto de la presente reivindicación 1, así la invención se considera obvia.

El Solicitante solicita respetuosamente al Examinador que tenga en cuenta el extracto anteriormente citado de la *Guía para examen* con respecto al método problema-solución, especialmente el hecho de que el análisis se tenga que aplicar objetivamente y sin aplicar retrospectiva, y también evaluando si un experto en la técnica *habría* modificado el estado de la técnica más cercano para llegar a la invención en evaluación, y no solo si el experto *podría* haber llegado.

Se observa que D2 es un documento que sólo está disponible en chino, sin solicitud equivalente en inglés o idioma similar. Por lo tanto, el siguiente análisis se basará en la traducción automática al inglés que está disponible en el sitio web de la Oficina Europea de Patentes esp@cenet (copia adjunta por comodidad).



En primer lugar, el Solicitante desea señalar que D2 divulga una combinación plaguicida binaria y no divulga ninguna composición ternaria, y aún menos la combinación plaguicida ternaria que es el objeto de la presente solicitud de patente.

En D2 se divulga 3 combinaciones binarias:

1. metoxifenoza + etofenprox
2. metoxifenoza + bifentrina
3. metoxifenoza + lambda-cihalotrina

No hay nada en la divulgación de D2 que motivara al experto a añadir un tercer componente, y aún menos en particular el tercer componente benzoato de emamectina, para preparar la composición ternaria de la presente invención.

Además, D2 comprende varios ejemplos de formulación, concretamente “*Ejemplos de formulación*” 1 a 28. De estas 28 formulaciones, solo los Ejemplos de formulación 11, 15 y 20 comprenden metoxifenoza en combinación con lambda-cihalotrina; hay otros ejemplos de formulación que comprenden lambda-cihalotrina en combinación con otros insecticidas irrelevantes, tales como metilhidrazida (por ejemplo, Ejemplos de formulación 6, 24, 26) o trimetoprim (Ejemplo de formulación 8, 17, 28), u otros ejemplos de formulación, que comprenden metoxifenoza en combinación con otros insecticidas irrelevantes, tales como etetrina (Ejemplo de formulación 1, 4, 7, 9, 12, 13, 16, 18, 21, 22, 25) o bifentrina (Ejemplo de formulación 10). Además, la pimetrozina, la flubendiamida y la deltametrina ni siquiera se mencionan en D2.

Es decir, además del hecho de que un experto en la técnica no hubiera estado motivado para implementar ninguna divulgación dada por D2 a la preparación de una composición



ternaria, además, cuando se considera el número global de los Ejemplos de formulación para cada una de las combinaciones (que pueden proporcionar una idea de las combinaciones preferidas), un experto en la técnica no hubiera estado motivado en absoluto para seleccionar particularmente la combinación de metoxifenoazida y lambda-cihalotrina, para preparar una combinación plaguicida ternaria a partir de D1, si es que siquiera hubiera considerado preparar una composición ternaria. De hecho, la divulgación de D2 habría guiado a un experto en la técnica en contra de usar esta combinación particular para preparar una nueva composición plaguicida, ya que es evidentemente una de las combinaciones menos preferidas en D2.

Si lo anterior aún no se considerara suficiente argumento, es aún más relevante que, además de la (falta de) relevancia dada por el número de ejemplos con la combinación de activos potencialmente relevante, las explicaciones proporcionadas en D2 desaconsejan claramente el uso de la combinación de metoxifenoazida y lambda-cihalotrina, como se demostrará a continuación.

En la Tabla 5, se proporcionan los *“resultados de la prueba de control del barrenador del arroz”* (véase la página 11 de la traducción al inglés de D2).

El párrafo que sigue a la Tabla 5, se traduce como sigue: “Como puede observarse en la tabla anterior, el metóxido se combina con uno seleccionado del grupo que consiste en los insecticidas piretroides etere-trina, bifentrina o lambda-cihalotrina.”



Tabla 5 de D2:



[0125] 表 5 防治水稻二化螟试验结果

[0126]

处理 编号	药剂处理	施药剂量(制剂量) (克/亩)	保苗效果 (%)	杀虫效果 (%)
1	10%甲氧虫酰肼+10%醚菊酯 SC	20	76.15	79.86
2	10%甲氧虫酰肼+10%醚菊酯 SC	30	86.42	91.53
3	10%甲氧虫酰肼+10%醚菊酯 SC	40	90.31	94.85
4	10%甲氧虫酰肼+5%联苯菊酯 SC	20	72.28	76.08
5	10%甲氧虫酰肼+5%联苯菊酯 SC	30	85.23	90.54
6	10%甲氧虫酰肼+5%联苯菊酯 SC	40	87.46	92.32
7	24%甲氧虫酰肼+6%高效氯氟 氰菊酯 SC	9	73.28	78.08
8	24%甲氧虫酰肼+6%高效氯氟 氰菊酯 SC	12	87.23	91.54
9	24%甲氧虫酰肼+6%高效氯氟 氰菊酯 SC	15	88.46	93.32
10	24%甲氧虫酰肼 SC	30	79.62	83.98
11	10%醚菊酯 SC	100	75.21	78.37
12	2.5%联苯菊酯 EC	100	69.54	73.65
13	2.5%高效氯氟氰菊酯 EW	60	73.22	76.31
14	清水对照	/	/	/

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Se debe suponer que la primera columna son los números de “Ejemplos de formulación”. Como se observa anteriormente, solo los Ejemplos de formulación 11, 15 y 20 parecen relacionarse con la combinación de metoxifenoazida y lambda-cihalotrina. Suponiendo que la 4.^a y 5.^a columnas se refieren a la actividad, se observa que el Ejemplo 11, que sería la combinación de metoxifenoazida y lambda-cihalotrina, es una de las composiciones con la menor actividad divulgada. Es decir, un experto en la técnica habría elegido cualquiera de las combinaciones más activas, cuando se intentara preparar una nueva composición plaguicida.

Aunque lo anterior es algo basado en suposiciones, en vista del hecho de que el documento está en chino, se dice que la Tabla 6 refleja los “*resultados de la prueba de control del enrollador de las hojas del arroz*” (véase la página 12 de la traducción al inglés de D2). En el párrafo debajo de dicha tabla se establece que “*Como se puede apreciar de la tabla anterior, 40 % de metoxifenoazida + 40 % de etilmetrina WG, 12 % de metoxifenoazida + 3 % de bifentrina SE, 20 % de metoxifenoazida + 10 % de cloruro de etilo*” [...] tienen “*mejor efecto de control sobre el enrollador de la hoja del arroz a dosis de prueba medias y altas*” (véase la página 12 de la traducción al inglés de D2). No se menciona la lambda-cihalotrina. Además, se dice que la Tabla 7 se relaciona con “*los resultados de control de la polilla del rizado de las hojas del manzano*” (véase la página 12 de la traducción al inglés de D2), y a continuación de la tabla, se establece lo siguiente: “*Como se puede apreciar de la tabla anterior, 5 % de metoxifenoazida + 5 % de etilmetrina OF, 9 % de metoxifenoazida + 3 % de bifentrina ME, 15 % de metoxifenoazida + 5 % de cloro ciflutrina altamente eficiente tiene mejor efecto de control sobre la polilla del enrollador de las hojas del manzano a dosis de prueba medias y altas*” (véase la página 13 de la traducción al inglés de D2). No se menciona la lambda-cihalotrina.



En vista de lo anterior, un experto en la técnica, basándose en la divulgación de D2, habría estado motivado para explorar cualquiera de las combinaciones preferidas divulgadas en el presente documento, y en ningún caso habría estado motivado a explorar la combinación de metoxifenoazida y lambda-cihalotrina; de hecho, un experto en la técnica habría visto desaconsejado, basándose en la divulgación de D2, de explorar aún más la combinación de metoxifenoazida y lambda-cihalotrina; es decir, la divulgación de D2 desaconseja a un experto en la técnica de la modificación de D1 combinando metoxifenoazida y lambda-cihalotrina, para alcanzar la composición de la presente invención, cuando el componente c) es lambda-cihalotrina. Aún menos el experto en la técnica habría estado motivado para combinar metoxifenoazida con cualquiera de pimetrozina, flubendiamida o deltametrina, por cualquiera de los documentos del estado de la técnica citados.

Así parecería que el Examinador puede haber aplicado retrospectiva cuando se evalúa la actividad inventiva de la presente invención, en vista de las divulgaciones de los documentos D1 y D2, contrario a las indicaciones de la *Guía para Examen* en Colombia. Un experto en la técnica, a partir de la divulgación del documento del estado de la técnica más cercano D1, no hubiera tenido ninguna motivación ni indicio para modificar la composición de D1 combinando metoxifenoazida con cualquiera de pimetrozina, flubendiamida, deltametrina o lambda-cihalotrina. Se alega respetuosamente que la presente invención no es obvia en vista de las divulgaciones de D1 y D2, y por lo tanto es inventiva.

Se ruega respetuosamente al Examinador que considere todo lo anterior, junto con los argumentos aportados en respuesta al primer Requerimiento, y reconsidere que la invención es realmente inventiva a la vista del estado de la técnica.



En caso de que aún queden dudas, se solicita respetuosamente que se emita un nuevo Requerimiento, con el fin de dar al Solicitante la oportunidad de proporcionar más aclaraciones.

3. Conclusiones

En consecuencia y luego de haber presentado los argumentos necesarios y haber realizado las modificaciones pertinentes en el capítulo reivindicatorio de la presente invención, queda evidenciado que la invención cumple con los requisitos de patentabilidad definidos en la Decisión 486 de 2000.

Cordialmente,

Juan Carlos Cuesta Quintero

C.C. 79.339.809 de Bogotá

T.P. 43.273 del C.S. de la J.

Anexos: Comprobante de pago por modificación de la solicitud.
Capítulo reivindicatorio modificado con control de cambios.
Capítulo reivindicatorio modificado en limpio.

REIVINDICACIONES

1. Una composición insecticida sinérgica que comprende:
 - a) de 15 al 25 % p/p de metoxifenoazida;
 - 5 b) de 0,1 al 3 % p/p de benzoato de emamectina;
 - c) de 0,5 al 20 % p/p de uno de pimetrozina, flubendiamida, deltametrina y lambda-cialotrina; y
 - d) uno o más excipientes seleccionados de agente dispersante seleccionado del grupo que comprende sal de amina de fosfato de triestirilo fenol etoxilado, copolímero acrílico, copolímero de injerto y combinaciones de los mismos, agente humectante seleccionado del grupo que comprende éster de fosfato de poliarilfenol etoxilado, sulfosuccinato de dioctilo, etoxilado no iónico y combinaciones de los mismos, agente anticongelante seleccionado del grupo que comprende propilenglicol, dietilenglicol, monoetilenglicol y combinaciones de los mismos, antiespumante que es una emulsión de dimetil polisiloxano, biocida seleccionado del grupo que comprende una solución acuosa de dipropilenglicol al 20% de 1,2-bencisotiazolim-3-ona, formaldehído y combinaciones de los mismos, y espesante que es goma de xantano.
- 10
- 15
- 20
2. La composición insecticida sinérgica de acuerdo con la reivindicación 1, que comprende 21 % p/p de metoxifenoazida, 1,1 % p/p de benzoato de emamectina y 15 % p/p de pimetrozina.
- 25 3. La composición insecticida sinérgica de acuerdo con la reivindicación 1,

que comprende 21 % p/p de metoxifenoza, 1,1 % p/p de benzoato de emamectina y 3,5 % p/p de flubendiamida.

4. La composición insecticida sinérgica de acuerdo con la reivindicación 1,
5 que comprende 21 % p/p de metoxifenoza, 1,1 % p/p de benzoato de emamectina y 1,25 % p/p de deltametrina.

5. La composición insecticida sinérgica de acuerdo con la reivindicación 1,
que comprende 21 % p/p de metoxifenoza, 1,1 % p/p de benzoato de
10 emamectina y 1,5 % p/p de lambda-cialotrina.

6. La composición insecticida sinérgica de acuerdo con la reivindicación 1,
en donde la formulación se formula como ~~una suspensión en cápsula (CS),~~
~~concentrado dispersable (DC), polvo espolvoreable (DP), polvo para el~~
15 ~~tratamiento de semillas en seco (DS),~~ concentrado emulsionable (EC), ~~gránulo~~
~~emulsionable (EG), emulsión agua/aceite (EO), polvo emulsionable (EP),~~
emulsión para el tratamiento de semillas (ES), emulsión aceite/agua (EW),
concentrado fluido para el tratamiento de semillas (FS), gránulos (GR),
microemulsión (ME), dispersión oleosa (OD), ~~concentrado fluido miscible con~~
20 ~~aceite (OF), líquido miscible con aceite (OL), polvo dispersable en aceite (OP),~~
concentrado en suspensión (SC), concentrado en suspensión para aplicación
directa (SD), ~~o~~ suspoemulsión (SE), ~~gránulo soluble en agua (SG), concentrado~~
~~soluble (SL), aceite para dispersión (SO), polvo soluble en agua (SP),~~
~~comprimido soluble en agua (ST), suspensión de volumen ultrabajo (ULV),~~
25 ~~comprimido (TB), líquido de volumen ultrabajo (ULV), gránulos dispersables en~~

~~agua (WG), polvo humectable (PH), polvo dispersable en agua para el tratamiento de semillas en suspensión (WS), comprimido dispersable en agua (WT), una formulación mixta de CS y SC (ZC) o una formulación mixta de CS y SE (ZE), una formulación mixta de CS y EW (ZW).~~

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7. La composición insecticida sinérgica de acuerdo con la reivindicación 6, en donde la composición se formula como concentrado en suspensión.

8. La composición insecticida sinérgica de acuerdo con la reivindicación 1, en donde

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a. el agente dispersante ~~se selecciona del grupo que comprende sal de amina de fosfato de triestirilo fenol etoxilado, copolímero acrílico, copolímero de injerto y combinaciones de los mismos,~~ y está presente en una cantidad en el intervalo de 2 al 10 % p/p;.

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b. el agente humectante ~~se selecciona del grupo que comprende éster de fosfato de poliarilfenol etoxilado, sulfosuccinato de dioctilo, etoxilado no iónico y combinaciones de los mismos,~~ y está presente en una cantidad en el intervalo de 1 al 5 % p/p;

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c. el agente anticongelante ~~se selecciona del grupo que comprende propilenglicol, dietilenglicol, monoetilenglicol y combinaciones de los mismos,~~ y está presente en una cantidad en el intervalo de 2 al 10 % p/p;

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d. el antiespumante ~~es una emulsión de dimetil polisiloxano,~~ y está presente

en una cantidad en el intervalo de 0,01 al 0,5 % p/p;

- e. el biocida ~~se selecciona del grupo que consiste en una solución acuosa de dipropilenglicol al 20 % de 1,2-bencisotiazolim 3-ona, formaldehído y combinaciones de los mismos,~~ y está presente en una cantidad en el intervalo de 0,01 al 0,50 % p/p;
- 5
- f. el espesante ~~es goma de xantano y~~ está presente en una cantidad en el intervalo de 0,01 al 0,50 % p/p.

REIVINDICACIONES

1. Una composición insecticida sinérgica que comprende:
 - a) de 15 al 25 % p/p de metoxifenoazida;
 - 5 b) de 0,1 al 3 % p/p de benzoato de emamectina;
 - c) de 0,5 al 20 % p/p de uno de pimetrozina, flubendiamida, deltametrina y lambda-cialotrina; y
 - d) uno o más excipientes seleccionados de agente dispersante seleccionado del grupo que comprende sal de amina de fosfato de triestirilo fenol etoxilado, copolímero acrílico, copolímero de injerto y combinaciones de los mismos, agente humectante seleccionado del grupo que comprende éster de fosfato de poliarilfenol etoxilado, sulfosuccinato de dioctilo, etoxilado no iónico y combinaciones de los mismos, agente anticongelante seleccionado del grupo que comprende propilenglicol, dietilenglicol, monoetilenglicol y combinaciones de los mismos, antiespumante que es una emulsión de dimetil polisiloxano, biocida seleccionado del grupo que comprende una solución acuosa de dipropilenglicol al 20% de 1,2-bencisotiazolim-3-ona, formaldehído y combinaciones de los mismos, y espesante que es goma de xantano.
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- 15
- 20
2. La composición insecticida sinérgica de acuerdo con la reivindicación 1, que comprende 21 % p/p de metoxifenoazida, 1,1 % p/p de benzoato de emamectina y 15 % p/p de pimetrozina.
- 25
3. La composición insecticida sinérgica de acuerdo con la reivindicación 1,

que comprende 21 % p/p de metoxifenoza, 1,1 % p/p de benzoato de emamectina y 3,5 % p/p de flubendiamida.

4. La composición insecticida sinérgica de acuerdo con la reivindicación 1,
5 que comprende 21 % p/p de metoxifenoza, 1,1 % p/p de benzoato de emamectina y 1,25 % p/p de deltametrina.

5. La composición insecticida sinérgica de acuerdo con la reivindicación 1,
que comprende 21 % p/p de metoxifenoza, 1,1 % p/p de benzoato de
10 emamectina y 1,5 % p/p de lambda-cialotrina.

6. La composición insecticida sinérgica de acuerdo con la reivindicación 1,
en donde la formulación se formula como concentrado emulsionable (EC),
emulsión para el tratamiento de semillas (ES), emulsión aceite/agua (EW),
15 concentrado fluido para el tratamiento de semillas (FS), gránulos (GR),
microemulsión (ME), dispersión oleosa (OD), concentrado en suspensión (SC),
concentrado en suspensión para aplicación directa (SD), o suspoemulsión (SE).

7. La composición insecticida sinérgica de acuerdo con la reivindicación 6,
20 en donde la composición se formula como concentrado en suspensión.

8. La composición insecticida sinérgica de acuerdo con la reivindicación 1,
en donde
a. el agente dispersante está presente en una cantidad en el intervalo de 2
25 al 10 % p/p;

- b. el agente humectante está presente en una cantidad en el intervalo de 1 al 5 % p/p;
- 5 c. el agente anticongelante está presente en una cantidad en el intervalo de 2 al 10 % p/p;
- d. el antiespumante está presente en una cantidad en el intervalo de 0,01 al 0,5 % p/p;
- 10 e. el biocida está presente en una cantidad en el intervalo de 0,01 al 0,50 % p/p;
- f. el espesante está presente en una cantidad en el intervalo de 0,01 al 0,50 % p/p.
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(54) 发明名称

一种含甲氧虫酰肼和拟除虫菊酯类杀虫剂的
杀虫组合物

(57) 摘要

本发明公开了一种含甲氧虫酰肼和醚菊酯、
联苯菊酯或高效氯氟氰菊酯拟除虫菊酯类杀虫剂
的杀虫组合物。该组合物中甲氧虫酰肼与拟除虫
菊酯类杀虫剂的重量比是 40 : 1 ~ 1 : 10, 优选
为 20 : 1 ~ 1 : 5。本杀虫组合物可以配制成水
分散粒剂、可湿性粉剂、悬浮剂、悬浮乳剂、可分散
油悬浮剂、乳油、微乳剂、微囊悬浮剂剂型。本发明
用于防治水稻二化螟、三化螟、稻纵卷叶螟、棉花
棉铃虫、苹果卷叶蛾、蔬菜甜菜夜蛾鳞翅目害虫的
应用, 组分合理, 杀虫效果好, 用药成本低, 且其活
性和杀虫效果不是各组分活性的简单叠加, 而是
有显著的增效作用, 可减缓抗性的产生, 对作物安
全性好, 符合农药制剂的安全性要求。

1. 一种含甲氧虫酰肼和拟除虫菊酯类杀虫剂的杀虫组合物,其特征在于包括:

A) 第一活性成分甲氧虫酰肼;

B) 第二活性成分为拟除虫菊酯类杀虫剂;

第一活性成分与第二活性成分的重量比为 40 : 1 ~ 1 : 10。

2. 根据权利要求 1 所述的含甲氧虫酰肼和拟除虫菊酯类杀虫剂的杀虫组合物,其特征在于:第二活性成分拟除虫菊酯类杀虫剂选自醚菊酯、联苯菊酯、高效氯氟氰菊酯。

3. 根据权利要求 2 所述的含甲氧虫酰肼和拟除虫菊酯类杀虫剂的杀虫组合物,其特征在于:第一活性成分和第二活性成分的重量比是 20 : 1 ~ 1 : 5。

4. 根据权利要求 1 至 3 任意之一所述的含甲氧虫酰肼和拟除虫菊酯类杀虫剂的杀虫组合物,其特征在于:第一活性成分与第二活性成分的累积量为所述组合物总重量的 1% ~ 80%。

5. 根据权利要求 4 所述的含甲氧虫酰肼和拟除虫菊酯类杀虫剂的杀虫组合物,其特征在于:第一活性成分与第二活性成分的累积量为所述组合物总重量的 5% ~ 50%。

6. 根据权利要求 1 所述含甲氧虫酰肼和拟除虫菊酯类杀虫剂的杀虫组合物,其特征在于所述组合物可配制成农业上允许的任意剂型。

7. 根据权利要求 6 所述的含甲氧虫酰肼和拟除虫菊酯类杀虫剂的杀虫组合物,其特征在于:所述组合物可配制的制剂剂型是水分散粒剂、可湿性粉剂、悬浮剂、悬浮乳剂、可分散油悬浮剂、乳油、微乳剂、微囊悬浮剂。

8. 权利要求 1 所述含甲氧虫酰肼和拟除虫菊酯类杀虫剂的杀虫组合物在防治水稻二化螟、三化螟、稻纵卷叶螟、棉花棉铃虫、苹果卷叶蛾、蔬菜甜菜夜蛾鳞翅目害虫中的应用。

一种含甲氧虫酰肼和拟除虫菊酯类杀虫剂的杀虫组合物

技术领域

[0001] 本发明涉及一种杀虫组合物,特别是一种第一活性成分为甲氧虫酰肼,第二活性成分选自醚菊酯、联苯菊酯或高效氯氟氰菊酯拟除虫菊酯类杀虫剂的杀虫组合物,属于复配农药技术领域。

背景技术

[0002] 甲氧虫酰肼,英文通用名称: methoxyfenozide,其他英文名: Intrepid, Runner, 化学名称: N-叔丁基-N'-(3-甲基-2-甲氧羰基)-3,5-二甲基苯甲酰肼。甲氧虫酰肼属双酰肼类杀虫剂,是一种昆虫生长调节剂,为一种非固醇型结构的蜕皮激素,模拟天然昆虫蜕皮激素——20-羟基蜕皮激素,激活并附着蜕皮激素受体蛋白,促使鳞翅目幼虫在成熟前提早进入蜕皮过程而又不能形成健康的新表皮,从而导致幼虫提早停止取食、最终死亡。鳞翅目幼虫摄食甲氧虫酰肼后的反应是快速的。一般摄食4~16小时后幼虫即停止取食,出现中毒症状。目前市场上销售的甲氧虫酰肼为240克/升的悬浮剂,作为一种仿生激素类杀虫剂,其具有低药量、持效长、稳定、高效,环境友好性等特点,是符合人们对现代农药的要求和发展趋势的,但甲氧虫酰肼在使用过程中存在着使用成本高,抗性增大的风险的缺陷。

[0003] 醚菊酯(英文通用名称 etofenprox,化学分子式 $C_{25}H_{28}O_3$)是一种结构中无菊酸但因空间结构和拟除虫菊酯有相似之处的类似拟除虫菊酯类的杀虫剂,具有杀虫谱广、杀虫活性高、击倒速度快、对稻田蜘蛛等天敌杀伤力较小,对作物安全等优点。有触杀和胃毒作用,对害虫无内吸传导作用。广泛用于水稻、蔬菜、棉花上,对鳞翅目、半翅目、直翅目、鞘翅目、双翅目和等翅目等多种害虫有高效。但在实际应用过程中,单独使用醚菊酯单剂,害虫易产生抗性,使用成本日趋上升。

[0004] 联苯菊酯,英文通用名: bifenthrin,化学名称: (1R, S)-顺式(Z)2,2-二甲基-3-(2-氯-3,3,3-三氟-1-丙烯基)环丙烷羧酸-2-甲基-3-苯基苄酯,分子式: $C_{23}H_{22}ClF_3O_2$ 。联苯菊酯是一种拟除虫菊酯类杀虫、杀螨剂。具有触杀、胃毒作用,无内吸、熏蒸作用,杀虫谱广、作用迅速。在土壤中不移动,对环境较为安全,残效期较长。适用于棉花、果树、蔬菜、茶叶等作物上防治鳞翅目幼虫、粉虱、蚜虫、潜叶蛾、叶蝉、叶螨等害虫、害螨。用于虫、螨并发时,省时省药。

[0005] 高效氯氟氰菊酯,英文通用名: lambda-cyhalothrin,化学名称: α -氰基-3-苯氧苄基-3-(2-氯-3,3,3-三氟-1-丙烯基)-2,2-二甲基环丙烷羧酸酯(Z)-(1R, 3R), S-酯及(Z), (1S, 3S), R-酯的1:1混合物;分子式: $C_{23}H_{19}O_3NClF_3$ 。高效氯氟氰菊酯是一种拟除虫菊酯类杀虫剂,具有触杀、胃毒作用,无内吸、熏蒸作用,杀虫谱广、作用迅速,作用机理为抑制昆虫神经轴突部位的传导,对昆虫具有趋避、击倒及毒杀的作用,作用特点为杀虫谱广,活性较高,药效迅速,喷洒后耐雨水冲刷,但长期使用易对其产生抗性,对刺吸式口器的害虫及害螨有一定防效,但对螨的使用剂量要比常规用量增加1-2倍,适用于花生、大豆、棉花、果树、蔬菜的害虫。防治对象:用于小麦、玉米、果树、棉花、十字花科蔬菜等防治麦

芽、吸浆虫、粘虫、玉米螟、甜菜夜蛾、食心虫、卷叶蛾、潜夜蛾、凤蝶、吸果夜蛾、棉铃虫、红龄虫、菜青虫等,用于草原、草地、旱田作物防治草地螟等。

[0006] 目前,以甲氧虫酰肼为有效成份的农药制剂以 240 克 / 升甲氧虫酰肼悬浮剂为主,在实际使用过程中,持效性长,但速效性一般,使用成本高;醚菊酯、联苯菊酯、高效氯氟氰菊酯作为拟除虫菊酯类杀虫剂,目前市场上销售大多为单剂,杀虫效果好,速效性快,但存在使用成本升高,易对其产生抗性,持效期一般的缺陷,发明人意外发现将甲氧虫酰肼与拟除虫菊酯类杀虫剂混配使用,具有显著的增效效果,可降低使用成本,减缓害虫抗性的产生的作用。

发明内容

[0007] 本发明的目的在于提供一种组分合理,显著增效,杀虫效果好,用药成本低,不易产生抗药性的含甲氧虫酰肼和拟除虫菊酯类杀虫剂的杀虫组合物。

[0008] 为了克服现有单一制剂的缺陷,本发明的技术方案是这样解决的:

[0009] 含甲氧虫酰肼和拟除虫菊酯类杀虫剂的杀虫组合物,包括:

[0010] A) 第一活性成分甲氧虫酰肼;

[0011] B) 第二活性成分为拟除虫菊酯类杀虫剂;

[0012] 第二活性成分拟除虫菊酯类杀虫剂选自醚菊酯、联苯菊酯、高效氯氟氰菊酯。

[0013] 第一活性成分与第二活性成分的重量比为 40 : 1 ~ 1 : 10,优选为 20 : 1 ~ 1 : 5。

[0014] 第一活性成分与第二活性成分的累积量为所述组合物总重量的 1% ~ 80%,优选为 5% ~ 50%,活性成分累积量的大小也与剂型密切相关,如液体剂型悬浮剂、悬浮乳剂、可分散油悬浮剂、乳油、微乳剂、微囊悬浮剂的活性成分累积量不超过 50%。

[0015] 本发明含甲氧虫酰肼和拟除虫菊酯类杀虫剂的杀虫组合物按照本领域技术人员所公知的方法可以配制的制剂剂型是水分散粒剂、可湿性粉剂、悬浮剂、悬浮乳剂、可分散油悬浮剂、乳油、微乳剂、微囊悬浮剂。

[0016] 对于水分散粒剂来说,本领域技术人员很熟悉使用相应的助剂完成本发明。分散剂选自聚羧酸盐、木质素磺酸盐、烷基萘磺酸盐、烷基酚聚氧乙烯醚、E0/P0 嵌段聚醚;润湿剂选自烷基硫酸盐、烷基磺酸盐、萘磺酸盐、木质素磺酸钠、脂肪醇聚氧乙烯醚、烷基酚聚氧乙烯醚;崩解剂选自硫酸铵、硫酸钠、氯化钠、氯化铵、尿素、蔗糖、葡萄糖、羧甲基纤维素、可溶淀粉、聚乙烯吡咯烷酮;粘结剂选自聚乙烯醇、可溶性淀粉、糊精、黄原胶、羧甲基(乙基)纤维素类;填料选自硅藻土、高岭土、白炭黑、轻质碳酸钙、滑石粉、凹凸棒土、陶土。

[0017] 对可湿性粉剂,可使用的助剂有:分散剂选自聚羧酸盐、木质素磺酸盐、烷基萘磺酸盐;润湿剂选自烷基硫酸盐、烷基磺酸盐、萘磺酸盐;填料选自硫酸铵、尿素、蔗糖、葡萄糖、硅藻土、高岭土、白炭黑、轻质碳酸钙、滑石粉、凹凸棒土、陶土。

[0018] 对悬浮剂,可使用的助剂有:分散剂选自聚羧酸盐、木质素磺酸盐、烷基萘磺酸盐、TERSPERSE 2425(美国亨斯迈公司出品,烷基萘磺酸盐类);乳化剂选自农乳 700#(通用名:烷基酚甲醛树脂聚氧乙烯醚)、农乳 2201、斯盘-60#(通用名:山梨醇酐单硬脂酸酯)、乳化剂 T-60(通用名:失水山梨醇单硬脂酸酯聚氧乙烯醚)、农乳 1601#(通用名:苯乙基苯酚聚氧乙烯聚氧丙烯醚)、TERSPERSE 4894(美国亨斯迈公司出品);润湿剂选自烷基酚聚氧乙

烯基醚甲醛缩合物硫酸盐、烷基酚聚氧乙烯基醚磷酸酯、苯乙基酚聚氧乙烯基醚磷酸酯、烷基硫酸盐、烷基磺酸盐、萘磺酸盐、TERSPERSE 2500(美国亨斯迈公司出品);增稠剂选自黄原胶、聚乙烯醇、膨润土、硅酸镁铝;防腐剂选自甲醛、苯甲酸、苯甲酸钠;消泡剂为有机硅类消泡剂;防冻剂选自乙二醇、丙二醇、甘油、尿素、无机盐类(如氯化钠)。

[0019] 对悬浮乳剂,可使用的助剂有:分散剂选自聚羧酸盐、木质素磺酸盐、烷基萘磺酸盐(扩散剂 NNO)、TERSPERSE 2425;乳化剂选自农乳 700#、农乳 2201、斯盘-60#、吐温-60#(通用名:失水山梨醇单硬脂酸酯聚氧乙烯醚)、农乳 1601#、三苯乙基酚聚氧乙烯基醚磷酸酯、TERSPERSE 4894;润湿剂选自烷基酚聚氧乙烯基醚甲醛缩合物硫酸盐、烷基酚聚氧乙烯基醚磷酸酯、苯乙基酚聚氧乙烯基醚磷酸酯、烷基硫酸盐、烷基磺酸盐、萘磺酸盐、TERSPERSE 2500;溶剂选自二甲苯、甲苯、环己酮、溶剂油(S-150、S-180、S-200);增稠剂选自黄原胶、聚乙烯醇、膨润土、硅酸镁铝;防腐剂选自甲醛、苯甲酸、苯甲酸钠;消泡剂为有机硅类消泡剂;防冻剂选自乙二醇、丙二醇、甘油、尿素、无机盐类如氯化钠;稳定剂选自亚磷酸三苯酯、环氧氯丙烷、醋酐;水为去离子水。

[0020] 对可分散油悬浮剂,可使用的助剂有:分散剂选自聚羧酸盐、木质素磺酸盐、烷基萘磺酸盐(扩散剂 NNO)、TERSPERSE 2425;乳化剂选自 BY(蓖麻油聚氧乙烯醚)系列乳化剂(BY-110、BY-125、BY-140)、农乳 700#、农乳 2201、斯盘-60#、吐温-60#、农乳 1601#、TERSPERSE 4894;润湿剂选自烷基酚聚氧乙烯基醚甲醛缩合物硫酸盐、烷基酚聚氧乙烯基醚磷酸酯、苯乙基酚聚氧乙烯基醚磷酸酯、烷基硫酸盐、烷基磺酸盐、萘磺酸盐、TERSPERSE 2500;增稠剂选自白炭黑、聚乙烯醇、膨润土、硅酸镁铝;防冻剂选自乙二醇、丙二醇、甘油、尿素、无机盐类(如氯化钠);稳定剂选自环氧大豆油、环氧氯丙烷、磷酸三苯酯;分散介质选自大豆油、菜籽油、玉米油、油酸甲酯、柴油、机油、矿物油。

[0021] 对于乳油来说,可使用的助剂有:乳化剂选自十二烷基苯磺酸钙(农乳 500#)、农乳 700#、农乳 2201#、Span-60#(通用名:山梨醇酐单硬脂酸酯)、乳化剂 T-60、TX-10(通用名:辛基酚聚氧乙烯(10)醚)、农乳 1601#、农乳 600#、农乳 400#;溶剂选自二甲苯、溶剂油(S-150、S-180、S-200)、甲苯、生物柴油、甲酯化植物油;助溶剂:乙酸乙酯、甲醇、二甲基甲酰胺、环己酮、丙酮;稳定剂选自亚磷酸三苯酯、环氧氯丙烷、醋酐。

[0022] 对微乳剂,可使用的助剂有:乳化剂选自十二烷基苯磺酸钙(农乳 500#)、农乳 700#、农乳 2201#、斯盘-60#、吐温-60#、TX-10、农乳 1601、农乳 600#、农乳 400#;助乳化剂选自甲醇、异丙醇、正丁醇、乙醇;溶剂选自环己酮、N-甲基吡咯烷酮、二甲苯、甲苯、溶剂油(牌号:S-150、S-180、S-200);稳定剂选自亚磷酸三苯酯、环氧氯丙烷。

[0023] 对于微囊悬浮剂,可使用的助剂及填料有:溶剂、乳化剂、成囊单体、润湿分散剂、防冻剂、增稠剂和水。溶剂选自甲苯、二甲苯、二甲基甲酰胺、环己酮、丙酮、二甲基亚砷、N-甲基吡咯烷酮;所述的乳化剂选自农乳 700#、农乳 2201、斯盘-60#、吐温-60#、TX-10、农乳 1601;所述的成囊单体是聚醚多元醇(规格为 6305(GB12008.2-89))、多元醇改性 MDI(产品规格是 PMM-20, NCO(游离异氰脲酸)含量为 27.0-28.0%,酸份 $\leq 0.1\%$,粘度 $150 \pm 50 \text{ mPas}/25^\circ\text{C}$)中的一种;润湿分散剂选自烷基酚甲醛树脂聚氧乙烯醚硫酸钠(SOPA)、烷基酚聚氧乙烯基醚磷酸酯、苯乙基酚聚氧乙烯基醚磷酸酯;防冻剂选自乙二醇、丙二醇、甘油;增稠剂选自黄原胶、聚乙烯醇、膨润土;防腐剂选自甲醛、苯甲酸、苯甲酸钠;水为去离子水。制备过程:将第一活性成分和第二活性成分用溶剂溶解后,加入成囊单体和

乳化剂混合均匀作为油相,以去离子水为水相。将油相加入水相中,在高速剪切机下剪切,使乳状液的粒径至 1 ~ 3 微米,然后通过控制温度和搅拌速度,使微囊固化,待体系粒径符合需要的大小时停止成囊反应,然后将分散剂、增稠剂、防腐剂、防冻剂加入,搅拌均匀。

[0024] 本发明组分合理,杀虫效果好,用药成本低,且其活性和杀虫效果不是各组分活性的简单叠加,而是有显著的增效作用,可减缓抗性的产生,对作物安全性好,符合农药制剂的安全性要求。本发明对水稻二化螟、三化螟、稻纵卷叶螟、棉花棉铃虫、苹果卷叶蛾、蔬菜甜菜夜蛾鳞翅目害虫具有较好的防治效果。

具体实施方式

[0025] 为了防治农业生产上的水稻二化螟、三化螟、稻纵卷叶螟、棉花棉铃虫、苹果小卷叶蛾、蔬菜甜菜夜蛾害虫,发明人将甲氧虫酰肼与醚菊酯、联苯菊酯或高效氯氟氰菊酯等成分进行了相互复配的增效研究,具体方法为:

[0026] 1) 水稻二化螟学名 (*Chilo suppressalis*) 鳞翅目,螟蛾科,别名钻心虫;三化螟 (*Tryporyza incertulas*(walker)) 鳞翅目,螟蛾科;稻纵卷叶螟 (*Cnaphalocrocis medialis* Guenee), 鳞翅目,螟蛾科,药剂对水稻二化螟室内毒力测定结果可代表对水稻稻纵卷叶螟、三化螟室内毒力结果,因此本试验以水稻二化螟为室内毒力测定试验试虫,试验药剂甲氧虫酰肼原药采用美国陶氏益农有限公司生产的原药,醚菊酯原药采用江苏辉丰农化股份有限公司生产的原药,联苯菊酯原药采用江苏扬农化工股份有限公司生产的原药,高效氯氟氰菊酯原药采用江苏常隆化工有限公司生产的原药,由陕西上格之路生物科学有限公司技术部将原药配制成需要的试验药剂,试验方法参考《中华人民共和国农业行业标准 NY/T1154. 2-2006》。首先将单剂及各混配药剂设置 5 个不同浓度梯度(在预备试验结果的基础上,水稻二化螟死亡率在 5% ~ 90% 的范围内按等比级数设定),试虫为采自田间的二化螟,在实验室挑选龄期均匀一致的二化螟,饥饿 4 ~ 8 小时,用直径 1 厘米的打孔器打皮叶碟,放入培养皿,并注意保湿。用毛细管点滴器从低浓度开始,每叶碟点滴水 1 μ L ~ 2 μ L 药液,待溶剂挥发后和另一片涂有淀粉糊的叶碟对合制成夹毒叶碟,制作完毕放于 12 孔组织培养板的孔内。每处理 4 次重复,每重复不少于 12 个夹毒叶碟,并设不含药剂的相应的有机溶剂的处理作为对照。组织超前意识板每个孔内接 1 头试虫,置于正常条件下培养。接虫 2 ~ 4 小时后,待试虫取食完含药叶碟后,在培养板孔内加入清洁饲料继续饲养至调查,淘汰未食完一张完整叶碟的试虫,24 小时检查死虫数,计算死亡率。

[0027] 2) 棉花棉铃虫 (*Heliothis armigera* Hiibner), 试验药剂来源如 1) 所述,由陕西上格之路生物科学有限公司技术部将原药配制成需要的试验药剂,试验方法为夹毒叶片法,以《中华人民共和国农业行业标准 NY/T1154. 2-2006》为参考,首先将单剂及各混配药剂设置 5 个不同浓度梯度(在预备试验结果的基础上,棉铃虫死亡率在 5% ~ 90% 的范围内按等比级数设定),试虫为采自田间的棉铃虫,在实验室挑选三龄期均匀一致的棉铃虫,饥饿 4 ~ 8h,选取 50 头幼虫,用电子天平称重,计算每头平均质量。接入组织培养板中,每空 1 头虫子,每处理重复 4 次,于正常条件下培养,4d 检查死虫数,记录总虫数和死虫数。空白对照为等量不含药剂的相应的有机溶剂的处理。

[0028] 3) 苹果小卷叶蛾 (*Adoxophyes orana*), 试验药剂来源如 1) 所述,由陕西上格之路生物科学有限公司技术部将原药配制成需要的试验药剂,试验方法参考《中华人民共和国

农业行业标准 NY/T1154. 2-2006》。

[0029] 4) 花椰菜甜菜夜蛾学名 *Laphygma exigua* Hubner 鳞翅目, 夜蛾科。别名贪夜蛾。试验药剂来源如 1) 所述, 由陕西上格之路生物科学有限公司技术部将原药配制成需要的试验药剂, 试验方法参考《中华人民共和国农业行业标准 NY/T1154. 2-2006》。

[0030] 混剂的共毒系数 (CTC 值) 按下列公式计算:

$$[0031] \quad ATI = \frac{S}{M} \times 100$$

[0032] 式中: ATI——混剂实测毒力指数;

[0033] S——标准杀虫剂 LC_{50} , 单位为毫克每升 (mg/L);

[0034] M——混剂的 LC_{50} , 单位为毫克每升 (mg/L);

$$[0035] \quad TTI = TI_A \times P_A + TI_B \times P_B$$

[0036] 式中: TTI——混剂理论毒力指数;

[0037] TI_A ——A 药剂毒力指数;

[0038] P_A ——A 药剂在混剂中的百分含量, 单位为百分率 (%);

[0039] TI_B ——B 药剂毒力指数;

[0040] P_B ——B 药剂在混剂中的百分含量, 单位为百分率 (%);

$$[0041] \quad CTC = \frac{ATI}{TTI} \times 100$$

[0042] 式中: CTC——共毒系数;

[0043] ATI——混剂实测毒力指数;

[0044] TTI——混剂理论毒力指数;

[0045] 复配制剂的共毒系数 (CTC) ≥ 120 表现为增效作用; CTC ≤ 80 表现为拮抗作用; $80 < CTC < 120$ 表现为相加作用。

[0046] 4) 测定结果分别见表 1 ~ 4。

[0047] 表 1 甲氧虫酰肼与拟除虫菊酯类杀虫剂组合对水稻二化螟的室内毒力测定

[0048]

药 剂	毒力回归方程	相关性(r)	EC ₅₀ . (μg/ml)	EC ₅₀ 95%的 置信区间	CTC
甲氧虫酰肼 (A)	Y=3.0564+2.2982X	0.9995	7.01	6.2418 ~ 7.9634	——
醚菊酯 (B)	Y=2.7939+2.0002X	0.9951	12.67	8.2688 ~ 23.0363	——
联苯菊酯 (C)	Y=4.6233+1.9271X	0.9961	1.57	1.3110 ~ 1.9227	——
高效氯氟氰菊酯 (D)	Y=4.8547+2.0682X	0.9996	1.18	1.1149 ~ 1.2404	——
A+B (40: 1)	Y=3.5625+1.9476X	0.9979	5.47	4.4080 ~ 7.1247	129.3
A+B (20: 1)	Y=3.5585+1.9968X	0.9983	5.27	4.3564 ~ 6.6159	136.0
A+B (10: 1)	Y=3.6192+1.9559X	0.9972	5.08	3.9928 ~ 6.8857	143.7
A+B (5: 1)	Y=3.6530+1.9164X	0.9977	5.05	4.0451 ~ 6.6261	150.2
A+B (2.5: 1)	Y=3.7008+1.9130X	0.9976	4.78	3.8326 ~ 6.2736	168.4
A+B (1: 1)	Y=3.5144+1.9909X	0.9970	5.57	4.3267 ~ 7.6890	162.0
A+B (1: 2.5)	Y=3.2605+2.0027X	0.9978	7.39	5.7707 ~ 10.0311	138.9
A+B (1: 5)	Y=3.1561+1.9789X	0.9986	8.55	6.9208 ~ 10.9794	130.6
A+B (1: 10)	Y=2.9815+2.0690X	0.9994	9.45	8.1653 ~ 11.1362	124.8
A+C (40: 1)	Y=3.6766+1.9651X	0.9975	4.71	3.7898 ~ 6.1748	137.2
A+C (20: 1)	Y=3.8801+1.9904X	0.9973	3.65	2.9804 ~ 4.6913	164.8

[0049]

A+C (10: 1)	$Y=4.0296+1.9995X$	0.9985	3.06	2.6453 ~ 3.6140	173.9
A+C (5: 1)	$Y=4.2703+1.9794X$	0.9979	2.34	2.0137 ~ 2.7759	190.1
A+C (2.5: 1)	$Y=4.3374+1.9850X$	0.9983	2.16	1.8911 ~ 2.5027	163.0
A+C (1: 1)	$Y=4.4878+2.0729X$	0.9998	1.77	1.7571 ~ 1.7760	144.9
A+C (1: 2.5)	$Y=4.6595+1.9766X$	0.9992	1.49	1.3704 ~ 1.6199	135.4
A+C (1: 5)	$Y=4.7122+1.9914X$	0.9993	1.39	1.2959 ~ 1.5057	129.7
A+C (1: 10)	$Y=4.7098+2.0374X$	0.9989	1.39	1.2674 ~ 1.5274	121.6
A+D (40: 1)	$Y=3.8071+1.8685X$	0.9952	4.35	3.2518 ~ 6.4376	143.8
A+D (20: 1)	$Y=3.8756+2.0380X$	0.9987	3.56	3.0882 ~ 4.1982	159.2
A+D (10: 1)	$Y=4.1643+1.9943X$	0.9976	2.62	2.2228 ~ 3.1937	184.4
A+D (5: 1)	$Y=4.2279+2.0623X$	0.9979	2.37	2.0388 ~ 2.8165	162.0
A+D (2.5: 1)	$Y=4.4639+1.8057X$	0.9988	1.98	1.7689 ~ 2.2451	146.8
A+D (1: 1)	$Y=4.7132+1.8749X$	0.9977	1.42	1.2402 ~ 1.6493	142.1
A+D (1: 2.5)	$Y=4.8607+1.8380X$	0.9952	1.19	0.9778 ~ 1.4677	130.0
A+D (1: 5)	$Y=4.9334+1.9120X$	0.9943	1.08	0.8755 ~ 1.3498	126.8
A+D (1: 10)	$Y=4.9617+1.9198X$	0.9956	1.05	0.8687 ~ 1.2654	121.6

[0050] 表 2 甲氧虫酰肼与拟除虫菊酯类杀虫剂组合对棉花棉铃虫的室内毒力测定

[0051]

药 剂	毒力回归方程	相关性 (r)	EC ₅₀ ($\mu\text{g/ml}$)	EC ₅₀ 95%的 置信区间	CTC
甲氧虫酰肼 (A)	$Y=2.7506+2.0052X$	0.9988	13.24	10.5667 ~ 17.2480	——
醚菊酯 (B)	$Y=2.7013+1.8675X$	0.9926	17.02	9.6219 ~ 41.0249	——
联苯菊酯 (C)	$Y=4.0855+2.1507X$	0.9989	2.66	2.3840 ~ 3.0100	——
高效氯氟氰菊酯 (D)	$Y=4.6650+1.9405X$	0.9964	1.49	1.2532 ~ 1.8028	——
A+B (40: 1)	$Y=2.9882+2.0008X$	0.9978	10.13	7.6975 ~ 14.2419	131.3

[0052]

A+B (20: 1)	$Y=3.0506+1.9583X$	0.9976	9.89	7.4080 14.2583 ~	135.3
A+B (10: 1)	$Y=3.0445+2.0087X$	0.9974	9.41	7.0484 13.5591 ~	143.6
A+B (5: 1)	$Y=3.0682+2.0715X$	0.9979	8.56	6.6532 11.6800 ~	160.6
A+B (2.5: 1)	$Y=3.2298+1.9781X$	0.9988	7.85	6.4663 ~ 9.8576	180.1
A+B (1: 1)	$Y=3.2723+1.9792X$	0.9992	7.46	6.4244 ~ 8.8420	199.8
A+B (1: 2.5)	$Y=3.3027+1.9629X$	0.9987	7.32	6.0584 ~ 9.1469	215.3
A+B (1: 5)	$Y=3.0836+1.9782X$	0.9988	9.30	7.6213 11.7555 ~	174.9
A+B (1: 10)	$Y=2.9535+1.9692X$	0.9980	10.95	8.3432 15.3007 ~	151.7
A+C (40: 1)	$Y=3.1656+1.9110X$	0.9990	9.12	7.5880 11.2762 ~	132.3
A+C (20: 1)	$Y=3.3835+1.9353X$	0.9988	6.84	5.6940 ~ 8.4881	162.7
A+C (10: 1)	$Y=3.6201+1.8997X$	0.9976	5.32	4.2415 ~ 7.0610	182.7
A+C (5: 1)	$Y=3.9087+1.8143X$	0.9972	3.99	3.2053 ~ 5.2571	199.5
A+C (2.5: 1)	$Y=4.0684+1.9381X$	0.9974	3.02	2.5057 ~ 3.8005	205.2
A+C (1: 1)	$Y=4.2163+2.0017X$	0.9986	2.46	2.1689 ~ 2.8452	180.1
A+C (1: 2.5)	$Y=4.3357+1.9371X$	0.9932	2.20	1.7001 ~ 3.0832	156.7
A+C (1: 5)	$Y=4.3167+2.0127X$	0.9967	2.18	1.8222 ~ 2.7136	140.8
A+C (1: 10)	$Y=4.3326+1.8628X$	0.9963	2.28	1.8690 ~ 2.9098	125.8
A+D (40: 1)	$Y=3.2739+1.9241X$	0.9952	7.89	5.4856 13.0462 ~	140.7
A+D (20: 1)	$Y=3.4185+1.9828X$	0.9981	6.27	5.0531 ~ 8.1604	153.5
A+D (10: 1)	$Y=3.6841+1.9617X$	0.9973	4.68	3.7348 ~ 6.2178	164.7
A+D (5: 1)	$Y=3.9561+2.0703X$	0.9985	3.19	2.7581 ~ 3.7831	179.3
A+D (2.5: 1)	$Y=4.2577+1.9868X$	0.9971	2.36	1.9814 ~ 2.9170	172.4
A+D (1: 1)	$Y=4.5619+1.9429X$	0.9980	1.68	1.4746 ~ 1.9424	159.4

[0053]

A+D (1: 2.5)	$Y=4.6747+2.0011X$	0.9956	1.45	1.2065 ~ 1.7923	137.7
A+D (1: 5)	$Y=4.7524+1.8634X$	0.9984	1.36	1.2107 ~ 1.5336	128.6
A+D (1: 10)	$Y=4.7754+1.8577X$	0.9983	1.32	1.1735 ~ 1.4971	122.8

[0054] 表 3 甲氧虫酰肼与拟除虫菊酯类杀虫剂组合对苹果小卷叶蛾的室内毒力测定
[0055]

药剂	毒力回归方程	相关性(r)	EC ₅₀ ($\mu\text{g/ml}$)	EC ₅₀ 95%的 置信区间	CTC
甲氧虫酰肼 (A)	$Y=3.8552+1.9370X$	0.9979	3.90	3.2311 ~ 4.8916	——
醚菊酯 (B)	$Y=3.2824+1.9872X$	0.9982	7.32	5.8605 ~ 9.5694	——
联苯菊酯 (C)	$Y=3.6069+2.0288X$	0.9977	4.86	3.9355 ~ 6.2921	——
高效氯氟氰菊酯 (D)	$Y=4.4795+2.0224X$	0.9968	1.81	1.5303 ~ 2.1925	——
A+B (40: 1)	$Y=3.9633+2.0643X$	0.9993	3.18	2.8755 ~ 3.5494	124.0
A+B (20: 1)	$Y=3.9669+2.1062X$	0.9997	3.09	2.9008 ~ 3.3132	129.1
A+B (10: 1)	$Y=4.0378+2.0144X$	0.9995	3.00	2.7742 ~ 3.2727	135.8
A+B (5: 1)	$Y=4.0259+1.9933X$	0.9989	3.08	2.7235 ~ 3.5413	137.3
A+B (2.5: 1)	$Y=4.0214+1.9400X$	0.9974	3.19	2.6364 ~ 4.0355	141.1
A+B (1: 1)	$Y=4.0209+1.8458X$	0.9962	3.39	2.6755 ~ 4.5967	150.1
A+B (1: 2.5)	$Y=3.8877+1.9409X$	0.9973	3.74	3.0389 ~ 4.8377	156.5
A+B (1: 5)	$Y=3.7177+1.9374X$	0.9970	4.59	3.6202 ~ 6.1961	139.1
A+B (1: 10)	$Y=3.5681+1.9598X$	0.9974	5.38	4.2363 ~ 7.2529	126.0
A+C (40: 1)	$Y=4.0767+1.8557X$	0.9968	3.14	2.5435 ~ 4.0955	124.8
A+C (20: 1)	$Y=4.0772+1.9375X$	0.9974	2.99	2.4813 ~ 3.7600	131.7
A+C (10: 1)	$Y=4.0827+1.9807X$	0.9972	2.90	2.4017 ~ 3.6598	136.9
A+C (5: 1)	$Y=4.1450+1.9396X$	0.9982	2.76	2.3692 ~ 3.2957	146.1
A+C (2.5: 1)	$Y=4.1568+2.0121X$	0.9980	2.62	2.2515 ~ 3.1380	157.7

[0056]

A+C (1: 1)	$Y=4.0404+1.9423X$	0.9961	3.12	2.4844 ~ 4.1628	138.7
A+C (1: 2.5)	$Y=3.8882+2.1025X$	0.9989	3.38	2.9701 ~ 3.9121	134.3
A+C (1: 5)	$Y=3.9009+1.9487X$	0.9994	3.66	3.3195 ~ 4.0859	127.6
A+C (1: 10)	$Y=3.8599+1.9242X$	0.9978	3.91	3.2397 ~ 4.9144	121.6
A+D (40: 1)	$Y=4.0227+2.0682X$	0.9977	2.97	2.4965 ~ 3.6496	127.7
A+D (20: 1)	$Y=4.1292+2.0439X$	0.9955	2.67	2.1286 ~ 3.5472	138.4
A+D (10: 1)	$Y=4.2165+2.0898X$	0.9983	2.37	2.0740 ~ 2.7610	148.9
A+D (5: 1)	$Y=4.3551+2.0427X$	0.9990	2.07	1.8712 ~ 2.3090	158.0
A+D (2.5: 1)	$Y=4.4268+2.0253X$	0.9977	1.92	1.6578 ~ 2.2658	152.7
A+D (1: 1)	$Y=4.5559+2.0223X$	0.9982	1.66	1.4676 ~ 1.8955	148.9
A+D (1: 2.5)	$Y=4.5367+2.0283X$	0.9975	1.69	1.4636 ~ 1.9903	126.5
A+D (1: 5)	$Y=4.5772+2.0301X$	0.9950	1.61	1.3185 ~ 2.0461	123.4
A+D (1: 10)	$Y=4.6036+2.0094X$	0.9941	1.57	1.2654 ~ 2.0356	121.2

[0057] 表 4 甲氧虫酰肼与拟除虫菊酯类杀虫剂组合对花椰菜甜菜夜蛾的室内毒力测定

[0058]

药剂	毒力回归方程	相关性 (r)	EC ₅₀ . (μg/ml)	EC ₅₀ 95%的 置信区间	CTC
甲氧虫酰肼 (A)	Y=5.5684+2.0628X	0.9979	0.53	0.4536 ~ 0.6090	——
醚菊酯 (B)	Y=4.4246+1.9964X	0.9964	1.94	1.6175 ~ 2.4095	——
联苯菊酯 (C)	Y=6.4986+2.0397X	0.9987	0.184	0.1514 ~ 0.2180	——
高效氯氟氰菊酯 (D)	Y=6.7699+2.0773X	0.9979	0.141	0.1052 ~ 0.1781	——
A+B (40: 1)	Y=5.6670+1.8142X	0.9954	0.429	0.3247 ~ 0.5356	125.8
A+B (20: 1)	Y=5.6977+1.8429X	0.9982	0.418	0.3542 ~ 0.4832	131.4
A+B (10: 1)	Y=5.7296+1.9066X	0.9979	0.414	0.3465 ~ 0.4831	137.1
A+B (5: 1)	Y=5.7717+1.9479X	0.9976	0.402	0.3304 ~ 0.4740	150.0

[0059]

A+B (2.5: 1)	$Y=5.7137+1.9354X$	0.9969	0.428	0.3440 ~ 0.5132	156.3
A+B (1: 1)	$Y=5.5564+1.9515X$	0.9969	0.519	0.4258 ~ 0.6144	160.4
A+B (1: 2.5)	$Y=5.3330+1.9521X$	0.9962	0.675	0.5547 ~ 0.8049	163.3
A+B (1: 5)	$Y=5.0658+1.8768X$	0.9975	0.922	0.7993 ~ 1.0616	145.8
A+B (1: 10)	$Y=4.8923+2.1184X$	0.9993	1.12	1.0451 ~ 1.2106	139.4
A+C (40: 1)	$Y=5.7856+2.0799X$	0.9992	0.419	0.3774 ~ 0.4611	121.0
A+C (20: 1)	$Y=5.8620+2.0527X$	0.9982	0.380	0.3216 ~ 0.4395	128.0
A+C (10: 1)	$Y=6.0421+2.0309X$	0.9968	0.307	0.2363 ~ 0.3786	147.4
A+C (5: 1)	$Y=6.2167+2.0060X$	0.9968	0.247	0.1846 ~ 0.3120	163.4
A+C (2.5: 1)	$Y=6.3066+2.0185X$	0.9975	0.225	0.1728 ~ 0.2793	153.3
A+C (1: 1)	$Y=6.4676+2.0073X$	0.9984	0.186	0.1486 ~ 0.2241	146.9
A+C (1: 2.5)	$Y=6.4757+1.9379X$	0.9972	0.173	0.1260 ~ 0.2227	128.7
A+C (1: 5)	$Y=6.5761+2.0145X$	0.9920	0.165	0.0897 ~ 0.2479	125.1
A+C (1: 10)	$Y=6.6605+2.0988X$	0.9919	0.162	0.0880 ~ 0.2429	120.8
A+D (40: 1)	$Y=5.8332+1.9447X$	0.9978	0.373	0.3067 ~ 0.4399	133.2
A+D (20: 1)	$Y=5.9542+2.0697X$	0.9975	0.346	0.2805 ~ 0.4122	135.4
A+D (10: 1)	$Y=6.1182+2.0297X$	0.9965	0.281	0.2112 ~ 0.3528	150.8
A+D (5: 1)	$Y=6.3710+1.9793X$	0.9987	0.203	0.1672 ~ 0.2395	178.8
A+D (2.5: 1)	$Y=6.4324+1.9802X$	0.9992	0.189	0.1620 ~ 0.2168	156.8
A+D (1: 1)	$Y=6.6521+2.1353X$	0.9980	0.168	0.1298 ~ 0.2087	132.6
A+D (1: 2.5)	$Y=6.7710+2.0572X$	0.9994	0.138	0.1192 ~ 0.1569	129.3
A+D (1: 5)	$Y=6.8357+2.0505X$	0.9986	0.127	0.1003 ~ 0.1557	126.5
A+D (1: 10)	$Y=6.8193+2.0067X$	0.9976	0.124	0.0886 ~ 0.1621	121.8

[0060] 室内毒力测定结果表明：甲氧虫酰肼与醚菊酯、联苯菊酯或高效氯氟氰菊酯拟除虫菊酯类杀虫剂混用对水稻二化螟、三化螟、稻纵卷叶螟、棉花棉铃虫、苹果小卷叶蛾、蔬菜

甜菜夜蛾有较好的防治效果,重量比为 40 : 1 ~ 1 : 10 时,表现为增效显著。

[0061] 下面结合制剂实施例对本发明内容作进一步说明。

[0062] 制剂实施例 1 :

[0063] 称取 40% 甲氧虫酰肼、40% 醚菊酯、3% TERSPERSE 2700 (聚羧酸盐,美国亨斯迈公司出品)、2% 扩散剂 NNO (烷基萘磺酸盐甲醛缩合物)、1% 拉开粉 BX (二丁基萘磺酸钠)、1% K-12 (十二烷基硫酸钠)、3% 可溶淀粉、3% 葡萄糖、高岭土加至 100% 重量份。上述原料经常规制取水分散粒剂的方法即混合、超微气流粉碎、混合、造粒步骤制取 80% 甲氧虫酰肼·醚菊酯水分散粒剂。

[0064] 制剂实施例 2

[0065] 称取 40% 甲氧虫酰肼、10% 联苯菊酯、3% TERSPERSE 2700、4% 木质素磺酸钠、1% 拉开粉 BX、1% K-12、1% 羧甲基纤维素、3% 尿素、硅藻土加至 100% 重量份。上述原料经常规制取水分散粒剂的方法即混合、超微气流粉碎、混合、造粒步骤制取 50% 甲氧虫酰肼·联苯菊酯水分散粒剂。

[0066] 制剂实施例 3 :

[0067] 称取 50% 甲氧虫酰肼、5% 高效氯氟氰菊酯、4% TERSPERSE 2700、1% 拉开粉 BX、1% K-12、0.8% 糊精、5% 硫酸铵、凹凸棒土加至 100% 重量份。上述原料经常规制取水分散粒剂的方法即混合、超微气流粉碎、混合、造粒步骤制取 55% 甲氧虫酰肼·高效氯氟氰菊酯水分散粒剂。

[0068] 制剂实施例 4 :

[0069] 称取 4% 甲氧虫酰肼、40% 醚菊酯、4% MorwetD-425 (烷基萘磺酸盐,阿克苏诺贝尔公司出品)、3% 扩散剂 NNO (烷基萘磺酸盐甲醛缩合物)、2% 拉开粉 BX、2% K-12、2% 聚乙烯吡咯烷酮、0.5% 黄原胶、高岭土加至 100% 重量份。上述原料经常规制取水分散粒剂的方法即混合、超微气流粉碎、混合、造粒步骤制取 44% 甲氧虫酰肼·醚菊酯水分散粒剂。

[0070] 制剂实施例 5

[0071] 称取 60% 甲氧虫酰肼、1.5% 联苯菊酯、5% 木质素磺酸钙、2% 拉开粉 BX、1% K-12、3% 白炭黑、高岭土加至 100% 重量份。上述原料经混合,气流粉碎后制得 61.5% 甲氧虫酰肼·联苯菊酯可湿性粉剂。

[0072] 制剂实施例 6

[0073] 称取 50% 甲氧虫酰肼、2.5% 高效氯氟氰菊酯、4% 木质素磺酸钙、3% TERSPERSE2700、2% 拉开粉 BX、1% K-12、3% 白炭黑、轻质碳酸钙加至 100% 重量份。上述原料经混合,气流粉碎后制得 52.5% 甲氧虫酰肼·高效氯氟氰菊酯可湿性粉剂。

[0074] 制剂实施例 7

[0075] 称取 5% 甲氧虫酰肼、10% 醚菊酯、4% 木质素磺酸钙、4% 扩散剂 NNO (烷基萘磺酸盐甲醛缩合物)、2% 拉开粉 BX、2% K-12、5% 白炭黑、凹凸棒土加至 100% 重量份。上述原料经混合,气流粉碎后制得 15% 甲氧虫酰肼·醚菊酯可湿性粉剂。

[0076] 制剂实施例 8

[0077] 称取 20% 甲氧虫酰肼、10% 高效氯氟氰菊酯、4% 木质素磺酸钠、2% TERSPERSE2700、2% K-12、3% 白炭黑、陶土加至 100% 重量份。上述原料经混合,气流粉碎后制得 30% 甲氧虫酰肼·高效氯氟氰菊酯可湿性粉剂。

[0078] 制剂实施例 9

[0079] 称取 10% 甲氧虫酰肼、15% 醚菊酯、3% TERSPERSE 4894、2% TERSPERSE 2500、1% TERSPERSE 2425、0.2% 黄原胶、5% 乙二醇、0.3% 苯甲酸、0.5% 有机硅消泡剂（商品名：s-29 南京四新应用化学品公司出品）去离子水加至 100% 重量份。上述原料经混合，高速剪切分散 30min，用砂磨机砂磨后制得 25% 甲氧虫酰肼·醚菊酯悬浮剂。

[0080] 制剂实施例 10：

[0081] 称取 10% 甲氧虫酰肼、5% 联苯菊酯、1.5% NNO、1% TERSPERSE 2500、2% 乳化剂 T-60、3% 农乳 700#、0.1% 黄原胶、5% 丙二醇、0.5% 甲醛、0.3% 有机硅消泡剂去离子水加至 100% 重量份。上述原料经混合，高速剪切分散 30min，用砂磨机砂磨后制得 15% 甲氧虫酰肼·联苯菊酯悬浮剂。

[0082] 制剂实施例 11：

[0083] 称取 24% 甲氧虫酰肼、6% 高效氯氟氰菊酯、3% TERSPERSE 4894、1% TERSPERSE2500、1% TERSPERSE 2425、0.2% 黄原胶、1% 硅酸镁铝、5% 乙二醇、0.3% 苯甲酸钠、0.5% 有机硅消泡剂去离子水加至 100% 重量份。上述原料经混合，高速剪切分散 30min，用砂磨机砂磨后制得 30% 甲氧虫酰肼·高效氯氟氰菊酯悬浮剂。

[0084] 制剂实施例 12：

[0085] 称取 10% 甲氧虫酰肼、10% 醚菊酯、3% 农乳 700#、2% 农乳 1601#、1.5% TERSPERSE2500、0.5% TERSPERSE 2425、0.2% 黄原胶、3% 白炭黑、5% 乙二醇、0.3% 苯甲酸、0.5% 有机硅消泡剂去离子水加至 100% 重量份。上述原料经混合，高速剪切分散 30min，用砂磨机砂磨后制得 20% 甲氧虫酰肼·醚菊酯悬浮剂。

[0086] 制剂实施例 13

[0087] 称取 5% 甲氧虫酰肼、5% 醚菊酯、2% TERSPERSE 2425、4% 斯盘 -60#、3% 吐温 -60#、2% 农乳 700#、1% TERSPERSE 2500、0.5% 白炭黑、5% 丙二醇、1% 环氧氯丙烷、油酸甲酯加至 100% 重量份。上述原料经混合，高速剪切分散 30min，用砂磨机砂磨后制得 10% 甲氧虫酰肼·醚菊酯可分散油悬浮剂。

[0088] 制剂实施例 14

[0089] 称取 18% 甲氧虫酰肼、2% 联苯菊酯、2% 扩散剂 NNO、3% 农乳 2201#、8% BY-110、1% TERSPERSE 2500、1% 硅酸镁铝、3% 丙二醇、15% 环氧大豆油、大豆油加至 100% 重量份。上述原料经混合，高速剪切分散 30min，用砂磨机砂磨后制得 20% 甲氧虫酰肼·联苯菊酯可分散油悬浮剂。

[0090] 制剂实施例 15

[0091] 称取 5% 甲氧虫酰肼、10% 高效氯氟氰菊酯、3% TERSPERSE 2425、3% 斯盘 -60#、3% 吐温 -60#、8% BY-110、1% TERSPERSE 2500、0.5% 白炭黑、5% 丙二醇、20% 环氧大豆油、油酸甲酯加至 100% 重量份。上述原料经混合，高速剪切分散 30min，用砂磨机砂磨后制得 15% 甲氧虫酰肼·高效氯氟氰菊酯可分散油悬浮剂。

[0092] 制剂实施例 16

[0093] 称取 5% 甲氧虫酰肼、15% 醚菊酯、3% 扩散剂 NNO、5% BY-125、4% 斯盘 -60#、4% 农乳 1601#、1% 苯乙基酚聚氧乙烯基醚磷酸酯、0.5% 膨润土、5% 甘油、3% 磷酸三苯酯、菜籽油加至 100% 重量份。上述原料经混合，高速剪切分散 30min，用砂磨机砂磨后制得 20%

甲氧虫酰肼·醚菊酯可分散油悬浮剂。

[0094] 制剂实施例 17

[0095] 称取 15% 甲氧虫酰肼、5% 高效氯氟氰菊酯、3% TERSPERSE 2425、4% 农乳 2201#、2% 农乳 700#、5% BY-110、1% TERSPERSE 2500、0.5% 白炭黑、5% 丙二醇、20% 环氧大豆油、油酸甲酯加至 100% 重量份。上述原料经混合，高速剪切分散 30min，用砂磨机砂磨后制得 20% 甲氧虫酰肼·高效氯氟氰菊酯可分散油悬浮剂。

[0096] 制剂实施例 18

[0097] 称取 5% 甲氧虫酰肼、5% 醚菊酯、4% TX-10、5% 农乳 700#、6% 农乳 500#、4% 农乳 1601#、15% 环己酮、5% N-甲基吡咯烷酮、5% 正丁醇、1% 环氧氯丙烷、经溶解完全并混合均匀，去离子水加至 100% 重量份，搅拌后制得 10% 甲氧虫酰肼·醚菊酯微乳剂。

[0098] 制剂实施例 19

[0099] 称取 9% 甲氧虫酰肼、3% 联苯菊酯、5% 农乳 400#、4% 农乳 500#、6% 农乳 1601#、25% 环己酮、5% 二甲苯、5% 异丙醇、3% 亚磷酸三苯酯、经溶解完全并混合均匀，去离子水加至 100% 重量份，搅拌后制得 12% 甲氧虫酰肼·联苯菊酯微乳剂。

[0100] 制剂实施例 20

[0101] 称取 3% 甲氧虫酰肼、2% 高效氯氟氰菊酯、6% 农乳 2201#、5% 农乳 700#、6% 农乳 600#、10% 环己酮、5% N-甲基吡咯烷酮、5% 甲苯、5% 乙醇、1% 环氧氯丙烷、经溶解完全并混合均匀，去离子水加至 100% 重量份，搅拌后制得 5% 甲氧虫酰肼·高效氯氟氰菊酯微乳剂。

[0102] 制剂实施例 21

[0103] 称取 8% 甲氧虫酰肼、5% 醚菊酯、4% TX-10、8% 农乳 600#、6% 农乳 500#、20% 环己酮、10% N-甲基吡咯烷酮、5% 甲苯、1% 环氧氯丙烷、经溶解完全并混合均匀，去离子水加至 100% 重量份，搅拌后制得 13% 甲氧虫酰肼·醚菊酯微乳剂。

[0104] 制剂实施例 22

[0105] 称取 8% 甲氧虫酰肼、10% 醚菊酯、4% 农乳 500#、2% 农乳 600#、3% 农乳 2201#、3% 亚磷酸三苯酯、16% 二甲基甲酰胺、二甲苯加至 100% 重量份。上述原料经混合，搅拌溶解完全后制得 18% 甲氧虫酰肼·醚菊酯乳油。

[0106] 制剂实施例 23

[0107] 称取 6% 甲氧虫酰肼、10% 联苯菊酯、4% 农乳 500#、3% 农乳 400#、5% 农乳 1601#、1% 环氧氯丙烷、10% 乙酸乙酯、10% 丙酮、S-150 加至 100% 重量份。上述原料经混合，搅拌溶解完全后制得 16% 甲氧虫酰肼·联苯菊酯乳油。

[0108] 制剂实施例 24

[0109] 称取 10% 甲氧虫酰肼、3% 高效氯氟氰菊酯、4% Span-60#、3% 乳化剂 T-60、3% 农乳 2201#、3% 醋酐、25% 丙酮、10% 乙酸乙酯、生物柴油加至 100% 重量份。上述原料经混合，搅拌溶解完全后制得 13% 甲氧虫酰肼·高效氯氟氰菊酯乳油。

[0110] 制剂实施例 25

[0111] 称取 5% 甲氧虫酰肼、7.5% 醚菊酯、1.5% 扩散剂 NNO、4% TERSPERSE 4894、2.5% 三苯乙基酚聚氧乙烯基醚磷酸酯、5.0% 环己酮、5% 甲苯、2.5% 斯盘-60#、3.5% 吐温-60#、1% TERSPERSE 2500、2% 硅酸镁铝、0.2% 黄原胶、5% 乙二醇、1% 环氧氯丙烷、

0.3%苯甲酸、0.5%有机硅消泡剂,去离子水加至100%重量份。操作步骤:1、上述原料中醚菊酯和溶剂溶解完全后加入乳化剂混合均匀,2、其他物料经混合均匀,3、将1中混合均匀物料投入2中物料中,高速剪切分散30min,用砂磨机砂磨后制得12.5%甲氧虫酰肼·醚菊酯悬浮乳剂。

[0112] 制剂实施例 26

[0113] 称取12%甲氧虫酰肼,3%高效氯氟氰菊酯,1.0%TERSPERSE 2700,4%TERSPERSE4894,2.5%农乳1601#,6%二甲苯,3.5%农乳2201,1%TERSPERSE 2500,0.2%黄原胶,5%丙二醇,1%环氧氯丙烷,0.3%苯甲酸钠、0.3%有机硅消泡剂,去离子水加至100%重量份。操作步骤:1、上述原料中高效氯氟氰菊酯和溶剂溶解完全后加入乳化剂混合均匀,2、其他物料经混合均匀,3、将1中混合均匀物料投入2中物料中,高速剪切分散30min,用砂磨机砂磨后制得15%甲氧虫酰肼·高效氯氟氰菊酯悬浮乳剂。

[0114] 制剂实施例 27

[0115] 称取8%甲氧虫酰肼、4%联苯菊酯,溶剂甲苯8%、N-甲基吡咯烷酮20%,成囊单体PMM-203%、乳化剂TX-104%、乳化剂2201 2%、分散剂烷基酚聚氧乙烯基醚磷酸酯4%,增稠剂黄元胶0.3%,防冻剂乙二醇4%,0.5%苯甲酸、去离子水补足100%。将甲氧虫酰肼和联苯菊酯两种活性组分用溶剂甲苯、N-甲基吡咯烷酮加热溶解后,加入成囊单体和乳化剂混合均匀作为油相,以余量水为水相。在高速剪切机8000转/分钟速度下,将油相加入到水相中剪切10-15分钟得到乳液,取样测定乳液粒径,平均粒径在3微米即可。然后将乳状液转移至带有温度计和搅拌器的反应釜中,加热温度至45℃,搅拌速度500转/分钟,使成囊单体在乳状液微粒上缓慢发生界面聚合反应,在微粒表面上逐渐形成微孔结构囊皮。待体系反应3-3.5小时后,取样测定粒径,测定平均粒径在5微米左右,成囊反应阶段结束。然后将温度升温到65℃,体系反应由成囊反应阶段转入囊皮固化阶段,固化阶段时间为1-1.5小时。囊皮固化阶段结束后,体系温度恢复到室温,然后将润湿分散剂、增稠剂、防冻剂、防腐剂加入,在800转/分钟的速度下搅拌30分钟即得到12%甲氧虫酰肼·联苯菊酯微囊悬浮剂。

[0116] 制剂实施例 28

[0117] 称取5%甲氧虫酰肼、2.5%高效氯氟氰菊酯,溶剂二甲苯10%、20%N-甲基吡咯烷酮,成囊单体聚醚多元醇63054%、乳化剂农乳700#3%、乳化剂22012%、分散剂苯乙基酚聚氧乙烯基醚磷酸酯4%,烷基酚甲醛树脂聚氧乙烯醚硫酸钠(SOPA)2%,增稠剂黄元胶0.3%,防冻剂丙二醇4%,0.5%苯甲酸钠、去离子水补足100%。将甲氧虫酰肼和高效氯氟氰菊酯两种活性组分用溶剂二甲苯和N-甲基吡咯烷酮加热溶解后,加入成囊单体和乳化剂混合均匀作为油相,以余量水为水相。在高速剪切机8000转/分钟速度下,将油相加入到水相中剪切10-15分钟得到乳液,取样测定乳液粒径,平均粒径在3微米即可。然后将乳状液转移至带有温度计和搅拌器的反应釜中,加热温度至45℃,搅拌速度500转/分钟,使成囊单体在乳状液微粒上缓慢发生界面聚合反应,在微粒表面上逐渐形成微孔结构囊皮。待体系反应3-3.5小时后,取样测定粒径,测定平均粒径在5微米左右,成囊反应阶段结束。然后将温度升温到65℃,体系反应由成囊反应阶段转入囊皮固化阶段,固化阶段时间为1-1.5小时。囊皮固化阶段结束后,体系温度恢复到室温,然后将润湿分散剂、增稠剂、防冻剂、防腐剂加入,在800转/分钟的速度下搅拌30分钟即得到7.5%甲氧虫酰肼·高效

氯氟氰菊酯微囊悬浮剂。

[0118] 生物实施例 1 :防治水稻二化螟田间试验

[0119] 发明人于 2010 年 6 月 15 日在江西省芦溪县进行了防治水稻二化螟的田间药效试验,验证了该药剂对二化螟的防治效果、有效剂量及对水稻的安全性。试验作物为水稻,防治对象二化螟 (*Chilo suppressalis*)。采用常规喷雾法,于水稻分蘖期,一代二化螟卵发育末期到卵孵初期,采用手动喷雾器常规喷雾,每亩喷液量 45 升,于一代二化螟螟害稳定时调查各小区的防治效果。采取平衡跳跃式随机取样,每小区调查 25 丛稻,统计枯心率及死亡率。同时在药后 2、7、14 天观察水稻生长情况,目测试验药剂对水稻有无药害产生。

[0120] 计算方法:

[0121]

$$\text{枯心率 (\%)} = \frac{\text{调查枯心数}}{\text{调查总株数}} \times 100$$

[0122]

$$\text{保苗效果 (\%)} = \frac{\text{CK}_0 - \text{Pt}_0}{\text{CK}_0} \times 100$$

[0123] 式中: Pt_0 ----- 药剂处理区药后枯心率;

[0124] CK_0 ----- 空白对照区药后枯心率

[0125] 表 5 防治水稻二化螟试验结果

[0126]

处理 编号	药剂处理	施药剂量(制剂量) (克/亩)	保苗效果 (%)	杀虫效果 (%)
1	10%甲氧虫酰肼+10%醚菊酯 SC	20	76.15	79.86
2	10%甲氧虫酰肼+10%醚菊酯 SC	30	86.42	91.53
3	10%甲氧虫酰肼+10%醚菊酯 SC	40	90.31	94.85
4	10%甲氧虫酰肼+5%联苯菊酯 SC	20	72.28	76.08
5	10%甲氧虫酰肼+5%联苯菊酯 SC	30	85.23	90.54
6	10%甲氧虫酰肼+5%联苯菊酯 SC	40	87.46	92.32
7	24%甲氧虫酰肼+6%高效氯氟 氰菊酯 SC	9	73.28	78.08
8	24%甲氧虫酰肼+6%高效氯氟 氰菊酯 SC	12	87.23	91.54
9	24%甲氧虫酰肼+6%高效氯氟 氰菊酯 SC	15	88.46	93.32
10	24%甲氧虫酰肼 SC	30	79.62	83.98
11	10%醚菊酯 SC	100	75.21	78.37
12	2.5%联苯菊酯 EC	100	69.54	73.65
13	2.5%高效氯氟氰菊酯 EW	60	73.22	76.31
14	清水对照	/	/	/

[0127] 从上表可以看出,甲氧虫酰肼与选自醚菊酯、联苯菊酯或高效氯氟氰菊酯拟除虫菊酯类杀虫剂中一种进行组合复配,在二化螟卵发育末期到卵孵初期施药一次可有效控制二化螟为害,且用药量少于对照药剂用量,有明显的增效效果,可用于水稻二化螟的防治,且对水稻安全。

[0128] 生物实施例 2:防治水稻稻纵卷叶螟田间试验

[0129] 发明人于 2009 年 7 月 16 日在浙江省仙居县进行了防治水稻稻纵卷叶螟的田间药效试验,验证了该药剂对稻纵卷叶螟的防治效果、有效剂量及对水稻的安全性。试验作物为

水稻,防治对象稻纵卷叶螟 (*Cnaphalocrocis medinalis*)。采用手动喷雾器常规喷雾,每亩喷液量 30 升,防治水稻三代稻纵卷叶螟,于药后 3 天、7 天、10 天进行调查。采取平衡跳跃式随机取样,每小区 5 点取样共查 25 丛稻,统计卷叶率,与对照区卷叶率比较,计算相对防效,同时调查卷叶内有虫率。同时在药后 3、7、10 天观察水稻生长情况,目测试验药剂对水稻有无药害产生。

[0130] 计算方法:

[0131]

$$\text{卷叶率 (\%)} = \frac{\text{调查卷叶数}}{\text{调查总叶数}} \times 100$$

[0132]

$$\text{防治效果 (\%)} = \frac{\text{CK} - \text{Pt}}{\text{CK}} \times 100$$

[0133] 式中:Pt----- 药剂处理区药后卷叶率;CK----- 空白对照区药后卷叶率

[0134] 表 6 防治水稻稻纵卷叶螟试验结果

[0135]

处理 编号	药剂处理	施药剂量 (制剂量) (克/亩)	防效(%)		
			3 天	7 天	10 天
1	40%甲氧虫酰肼+40%醚菊酯 WG	6	82.76	87.65	85.56
2	40%甲氧虫酰肼+40%醚菊酯 WG	8	87.43	94.31	93.87
3	40%甲氧虫酰肼+40%醚菊酯 WG	10	91.31	96.85	94.88
4	12%甲氧虫酰肼+3%联苯菊酯 SE	20	79.86	83.87	84.87
5	12%甲氧虫酰肼+3%联苯菊酯 SE	30	85.23	88.54	90.52
6	12%甲氧虫酰肼+3%联苯菊酯 SE	40	90.63	94.32	92.27
7	20%甲氧虫酰肼+10%高效氯氟氰菊酯 WP	9	80.86	86.87	85.87
8	20%甲氧虫酰肼+10%高效氯氟氰菊酯 WP	12	86.23	89.54	90.52
9	20%甲氧虫酰肼+10%高效氯氟氰菊酯 WP	15	91.63	94.32	93.27
10	24%甲氧虫酰肼 SC	30	68.21	82.65	83.31
12	10%醚菊酯 SC	100	82.57	83.98	74.89

[0136]

13	2.5%联苯菊酯 EC	100	75.32	79.74	69.64
14	2.5%高效氯氟氰菊酯 EW	100	80.65	81.47	71.53
18	清水对照	/	/	/	

[0137] 从上表可以看出,40%甲氧虫酰肼+40%醚菊酯 WG、12%甲氧虫酰肼+3%联苯菊酯 SE、20%甲氧虫酰肼+10%高效氯氟氰菊酯 WP 在中高试验剂量下对稻纵卷叶螟具有较好的防效,速效性与持效期优于对照单剂,可有效降低各有效成分的用量,具有明显的增效效果,对水稻安全。

[0138] 生物实施例 3:防治苹果小卷叶蛾田间试验

[0139] 发明人于 2010 年 4 月在陕西省礼泉县进行了防治苹果小卷叶虫的田间药效试验,验证了该药剂对苹果小卷叶虫的防治效果、有效剂量及对苹果的安全性。试验作物为苹果,防治对象苹果小卷叶虫 (*Adoxophyes orana*)。采用机动喷雾器常规喷雾,于花前用药,防治苹果小卷叶虫,于药后 3 天、7 天、10 天进行调查。每小区调查两株,记录上部树冠 100~200 个枝条的虫包数,药前基数调查时,在整个实验区另剥查 100 个虫包的有虫率,以校正虫包基数。施药后的调查,则在对应的时间和小区内剥查 30~50 个虫包的有虫(幼虫、蛹及蛹壳)率,以校正对应的虫包数,最后一次调查还需记录被害枝条数。同时在药后 3、7、10 天观察苹果生长情况,目测试验药剂对苹果有无药害产生。

[0140] 计算方法:

[0141]

$$\text{枝条受害率 (\%)} = \frac{\text{调查的总枝条数} - \text{受害枝条数}}{\text{调查的总枝条数}} \times 100$$

[0142]

$$\text{防治效果 (\%)} = \frac{\text{CK} - \text{Pt}}{\text{CK}} \times 100$$

[0143] 式中:Pt----- 药剂处理区虫数;CK----- 空白对照区虫数。

[0144] 表 7 防治苹果小卷叶蛾试验结果

[0145]

试验处理 (稀释倍数)		第一次施药后 3 天	第一次施药后 7 天	第二次施药后 10 天
		防效 (%)	防效 (%)	防效 (%)
5% 甲氧虫酰肼 +5% 醚菊酯 OF	1500	86.72	88.63	93.74
	1200	89.86	91.53	96.85
	900	92.75	94.01	100
9% 甲氧虫酰肼 +3% 联苯菊酯 ME	2800	83.74	86.52	91.76
	2300	87.34	89.01	94.79
	1800	89.42	92.04	96.53
15% 甲氧虫酰肼 +5% 高效氯 氟氰菊酯 OF	5000	81.74	84.52	88.76
	4000	85.34	87.01	92.79
	3000	88.42	91.04	94.74
24% 甲氧虫酰肼 SC	3000	73.21	85.15	86.51
10% 醚菊酯 SC	500	85.64	76.53	78.64
2.5% 联苯菊酯 EC	500	82.23	72.50	75.71
2.5% 高效氯氟氰菊酯 EW	500	83.95	74.22	73.65
清水对照 (CK)		/	/	/

[0146] 从上表可以看出,5% 甲氧虫酰肼 +5% 醚菊酯 OF、9% 甲氧虫酰肼 +3% 联苯菊酯 ME、15% 甲氧虫酰肼 +5% 高效氯氟氰菊酯 OF 在中高试验剂量下对苹果小卷叶蛾具有较好的防效,速效性与持效期优于对照单剂,可有效降低各有效成分的用量,具有明显的增效效果,对苹果安全。

[0147] 生物实例 4 防治花椰菜甜菜夜蛾田间药效试验

[0148] 防治花椰菜甜菜夜蛾参照“田间药效试验准则 (一) GB/T 17980.13-2000 杀虫剂防治十字花科蔬菜的鳞翅目幼虫”试验药剂、对照药剂和空白对照的小区处理采用随机区组排列,小区面积 30m²,4 次重复,施药前调查虫情基数,施药前及施药后 3、10 天调查防治效果。每小区随机取五点调查,每点查两株,调查每株上的不同龄期全部活幼虫数。

[0149] 药效计算方法:虫口减退率 (%) = $\frac{\text{施药前虫数} - \text{施药后虫数}}{\text{施药前虫数}}$

[0150]

$$\text{防治效果 (\%)} = (1 - \frac{CK_0 \times PT_1}{CK_1 \times PT_0}) \times 100$$

[0151] 式中:CK₀、CK₁ 分别为空白对照区施药前、后的虫数;

[0152] PT₀、PT₁ 分别为药剂处理区施药前、后的虫数。

[0153] 表 8 防治花椰菜甜菜夜蛾的试验结果

[0154]

试验处理 (g/亩)		施药后 3 天 防效 (%)	施药后 10 天 防效 (%)
5%甲氧虫酰肼+7.5%醚菊酯 SE	40	86.72	85.65
	50	92.86	90.13
	60	96.95	95.41
40%甲氧虫酰肼+10%联苯菊酯 WG	6	87.48	85.22
	8	93.39	91.51
	10	95.42	96.64
10%甲氧虫酰肼+3%高效氯氟氰菊酯 EC	20	86.48	85.22
	25	90.39	89.51
	30	93.42	92.64
24%甲氧虫酰肼 SC	20	71.21	84.35
10%醚菊酯 SC	80	83.23	72.50
2.5%联苯菊酯 EC	80	81.95	69.22
2.5%高效氯氟氰菊酯 EW	60	84.54	70.75
清水对照(CK)		/	/

[0155] 表 4 田间试验表明：甲氧虫酰肼与醚菊酯、联苯菊酯或高效氯氟氰菊酯拟除虫菊酯类杀虫剂混配后，明显提高了对花椰菜甜菜夜蛾的速效性及持效期，降低了用药量及用药成本，对花椰菜生长安全。

[0156] 生物实例 5 防治棉花棉铃虫田间药效试验

[0157] 防治棉花棉铃虫参照“田间药效试验准则（一）GB/T 17980.5-2000 杀虫剂防治棉铃虫”试验药剂、对照药剂和空白对照的小区处理采用随机区组排列，小区面积 50m²，并保证 5-6 行棉株，4 次重复，每小区中间数行随机取样挂牌固定 5 点，每点固定 5 株有棉铃虫卵和幼虫的棉株，共 25 株，药前记录幼虫数和卵数，药后记录幼虫数。从调查株以外的棉株上采卵至少 50 粒带回在室温下保湿培养，测定不同时间的卵孵化率。或以田间空白对照区卵孵化率化率为准。施药后最后一次调查时，需统计蕾铃数被害率和保蕾效果。若蕾铃较少需统计顶芽被害率和保顶效果，即定株上的总蕾铃数和被害蕾铃数，总株数和顶芽被害株数。基数调查，施药前调查定株上幼虫数和卵数。第一次调查药后 1 天，第二次调查药后 3 天，第三次调查药 7 天，同时调查蕾铃被害率或顶芽被害率。

[0158] 药效计算方法：

[0159] 卵孵化率 (%) = (已孵化卵数 / 总卵数) × 100%

[0160]

$$\text{第一天虫口减退率 } \% = \frac{(\text{药前幼虫数} + \text{药前卵数} \times \text{药后1天孵化率}) - \text{药后幼虫数}}{\text{药前幼虫数} + \text{药前卵数} \times \text{药后1天孵化率}}$$

[0161]

$$\text{第三天虫口减退率 } \% = \frac{(\text{药前幼虫数} + \text{药前卵数} \times \text{药后3天孵化率}) - \text{药后幼虫数}}{\text{药前幼虫数} + \text{药前卵数} \times \text{药后3天孵化率}}$$

[0162]

$$\text{第七天虫口减退率 } \% = \frac{(\text{药前幼虫数} + \text{药前卵数} \times \text{药后7天孵化率}) - \text{药后幼虫数}}{\text{药前幼虫数} + \text{药前卵数} \times \text{药后7天孵化率}}$$

[0163]

$$\text{校正防效 (\%)} = \frac{\text{PT} - \text{CK}}{100 - \text{CK}} \times 100$$

[0164] 式中:PT 药剂处理区虫口减退率;CK 空白对照区虫口减退率

[0165] 花蕾被害率(%) = 累计被害花蕾数 × 100 / 调查花蕾总数

[0166] 保蕾效果(%) = (空白对照区花蕾被害率 - 药剂处理区花蕾被害率) × 100 / 空白对照区花蕾被害率

[0167] 顶芽被害率(%) = 生长点被害株数 × 100 / 调查总株数

[0168] 保顶效果(%) = (空白对照区顶芽被害率 - 药剂处理区顶芽被害率) × 100 / 空白对照区顶芽被害率

[0169] 表 9 防治棉花棉铃虫的试验结果

[0170]

试验处理 (g/亩)		施药后 3 天	施药后 10 天	
		防效 (%)	防效 (%)	保蕾效果(%)
10%甲氧虫酰肼+10%醚菊酯 SC	40	73.72	75.69	92.75
	60	79.94	80.48	97.43
	80	84.75	83.61	100
10%甲氧虫酰肼+5%联苯菊酯 SC	40	71.91	73.52	90.86
	60	76.34	78.01	95.79
	80	82.42	82.04	97.43
24%甲氧虫酰肼+6%高效氯氟氰菊酯 SC	20	74.91	72.52	94.86
	30	82.34	80.01	98.79
	40	87.42	86.04	100
24%甲氧虫酰肼 SC	60	61.29	74.75	87.46
10%醚菊酯 SC	120	75.23	60.50	84.44
2.5%联苯菊酯 EC	200	72.54	58.53	81.74
2.5%高效氯氟氰菊酯 EW	120	76.55	62.26	85.58
清水对照(CK)		/	/	/

[0171] 表 5 田间试验表明:甲氧虫酰肼与醚菊酯、联苯菊酯或高效氯氟氰菊酯拟除虫菊酯类杀虫剂混配后,明显提高了对棉花棉铃虫的速效性及持效期,降低了用药量及用药成本,对棉花生长安全。

[0172] 综上所述,本发明的组合物是采用两种活性成分复配方案,其活性和杀虫效果不是各组分活性的简单叠加,与现有的单一制剂相比,除对水稻二化螟、三化螟、稻纵卷叶螟、棉花棉铃虫、苹果卷叶蛾、蔬菜甜菜夜蛾鳞翅目害虫具有显著的杀虫效果外,而且有显著的增效作用,可减缓抗性的产生,对作物安全性好,用药成本低,符合农药制剂的安全性要求。

CN101919386A Insecticidal composition containing methoxyfenozide and synthetic pyrethroid insecticide

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Insecticide composition containing methoxid and pyrethroid insecticide

Technical field

The invention relates to an insecticidal composition, in particular a first active ingredient is methoxid, and the second active ingredient is selected from the group consisting of etherethrin, bifenthrin or beta-cyhalothrin pyrethroids. The insecticidal composition of an insecticide belongs to the technical field of compound pesticides.

Background technique

Methofenozide, English Common Name: methoxyfenozide, Other English Names: Intrepid, Runner, Chemical Name: N-tert-butyl-N'-(3-methyl-2-toluoyl)-3,5-di Methylbenzoylhydrazine. Methofenozide is a bishydrazide insecticide. It is an insect growth regulator. It is a non-sterol-type ecdysone, which mimics the natural insect ecdysone, 20-hydroxyecdysone, and activates and attaches. The ecdysone receptor protein urges Lepidoptera larvae to enter the molting process before maturity without being able to form a healthy new epidermis, which causes the larvae to stop feeding early and eventually die. The response of Lepidoptera larvae after ingestion of methanid hydrazide is rapid. Usually 4 to 16 hours after feeding, the larvae stop feeding and symptoms of poisoning appear. Methofenozide is currently sold on the market as a suspension agent of 240 g / l. As a biomimetic hormone insecticide, it has the characteristics of low drug dose, long-lasting effect, stability, high efficiency, and environmental friendliness. It meets people's requirements and development trends of modern pesticides, but in the use of methoxyfenozide, it has the defects of high use cost and increased risk of resistance.

Etherethrin (English common name etofenprox, chemical formula C₂₅H₂₈O₃) is a pyrethroid-like insecticide that is free of inulin in structure but similar to pyrethroid in space structure. It has a broad insecticidal spectrum, It has high insecticidal activity, fast knock-down speed, less damage to natural enemies such as rice field spiders, and safety to crops. It has contact killing and stomach poisoning effects, and has no systemic conduction effect on pests. It is widely used in rice, vegetables, and cotton, and is highly effective against various pests such as Lepidoptera, Hemiptera, Orthoptera, Coleoptera, Diptera, and Isoptera. However, in the actual application process, if a single agent of permethrin is used, pests are prone to develop resistance, and the cost of use is increasing.

Bifenthrin, English common name: bifenthrin, Chemical name: (1R, S) -cis (Z) 2,2-dimethyl-3- (2-chloro-3,3,3-trifluoro-1 -Propenyl) cyclopropanecarboxylic acid-2-methyl-3-phenylbenzyl ester, molecular formula: C₂₃H₂₂ClF₃O₂. Bifenthrin is a pyrethroid insecticide and acaricide. It has contact and stomach poisoning effects, no systemic and fumigation effects, and has a wide range of insecticidal effects and rapid effects. It does not move in the soil, is safer to the environment, and has a longer residual period. It is suitable for cotton, fruit trees, vegetables, tea and other crops to control pests and pests such as Lepidoptera larvae, whitefly, aphids, leaf miners, leafhoppers, leaf mites, and spider mites. It is used to save time and medicine when worms and mites are concurrent.

Lambda-cyhalothrin, common name in English: lambda-cyhalothrin, chemical name: α-cyano-3-phenoxybenzyl-3- (2-chloro-3,3,3-trichlorofluoro-1-propene) -2,2-dimethylcyclopropanecarboxylate (Z)-(1R, 3R), S-ester and (Z), (1S, 3S), R-ester 1: 1 mixture; Molecular formula: C₂₃H₁₉O₃NCIF₃. Lambda-cyhalothrin is a pyrethroid insecticide with contact killing, stomach toxicity, no systemic and fumigation effects, broad insecticidal spectrum and rapid action, and its mechanism of action is to inhibit the conduction of insect axonal sites. It has the functions of avoiding, knocking down and poisoning insects, and has the characteristics of broad insecticidal spectrum, high activity, rapid medicinal effect, and resistance to rain erosion after spraying, but it is easy to be resistant to long-term use. Mouth pests and mites have a certain control effect, but the dosage of mites should be increased by 1-2 times than the normal dosage, and it is suitable for pests of peanut, soybean, cotton,

fruit trees and vegetables. Objects to be prevented: Used for wheat, corn, fruit trees, cotton, cruciferous vegetables, etc. to control malt, midges, armyworms, corn borer, beet armyworm, heartworm, leaf curler, leafworm, swallowtail butterfly, swallowtail butterfly, cotton bollworm, cotton boll Insects, red instars, cabbage worms, etc., are used for grassland, grassland, dryland crops, etc.

At present, pesticide formulations that use methoxrazine as an active ingredient are mainly 240g / liter methoxazide suspensions. In actual use, the effect is long, but the quick-acting is average, and the use cost is high; ether As a pyrethroid insecticide, pyrethrin, bifenthrin, and lambda-cyhalothrin are currently sold on the market as single agents, which have good insecticidal effects and fast-acting properties. It has the disadvantages of resistance and general durability. The inventors have unexpectedly found that the use of methoxid and pyrethroid insecticides has a significant synergistic effect, which can reduce the cost of use and slow the resistance to pests. The resulting effect.

Summary of the invention

The purpose of the present invention is to provide an insecticidal composition containing methoxid and pyrethroid insecticides which has reasonable components, significant synergism, good insecticidal effect, low drug cost, and low resistance to drug resistance.

In order to overcome the shortcomings of the existing single preparation, the technical solution of the present invention is solved as follows:

An insecticidal composition containing methoxyfenozide and a pyrethroid insecticide, including:

- A) the first active ingredient methoxyfenozide;
- B) the second active ingredient is a pyrethroid insecticide;

The second active ingredient, the pyrethroid insecticide, is selected from the group consisting of **etherethrin, bifenthrin, and lambda-cyhalothrin.**

The weight ratio of the first active ingredient to the second active ingredient is 40: 1 to 1:10, and preferably 20: 1 to 1: 5.

The cumulative amount of the first active ingredient and the second active ingredient is 1% to 80%, preferably 5% to 50% of the total weight of the composition. The cumulative amount of the active ingredient is also closely related to the dosage form, such as a liquid dosage form suspension The cumulative amount of active ingredients in agents, suspension emulsions, dispersible oil suspensions, emulsifiable concentrates, microemulsions, and microcapsule suspensions does not exceed 50%.

The insecticidal composition containing methoxid and pyrethroid insecticides according to the present invention can be formulated according to methods known to those skilled in the art. The formulations are water-dispersible granules, wettable powders, suspensions, and suspension emulsions. , Dispersible oil suspension, emulsifiable concentrate, microemulsion, microcapsule suspension.

For water-dispersible granules, those skilled in the art are familiar with using the corresponding adjuvants to complete the present invention. The dispersant is selected from polycarboxylate, ligninsulfonate, alkylnaphthalenesulfonate, alkylphenol polyoxyethylene ether, EO / PO block polyether; wetting agent is selected from alkyl sulfate, alkyl Sulfonate, naphthalene sulfonate, sodium lignin sulfonate, fatty alcohol polyoxyethylene ether, alkylphenol polyoxyethylene ether; disintegrants selected from ammonium sulfate, sodium sulfate, sodium chloride, ammonium chloride, urea , Sucrose, glucose, carboxymethyl cellulose, soluble starch, polyvinylpyrrolidone; the binder is selected from polyvinyl alcohol, soluble starch, dextrin, xanthan gum, carboxymethyl (ethyl) cellulose; fillers It is selected from diatomite, kaolin, white carbon black, light calcium carbonate, talc, attapulgite, and clay.

For wettable powders, the auxiliary agents that can be used are: the dispersant is selected from the group consisting of polycarboxylate, ligninsulfonate, and alkylnaphthalenesulfonate; the wetting agent is selected from alkyl sulfate, alkylsulfonate, Naphthalene sulfonate; the filler is selected from ammonium sulfate, urea, sucrose, glucose, diatomaceous earth, kaolin, white carbon black, light calcium carbonate, talc, attapulgite, and clay.

For the suspending agent, the auxiliary agents that can be used are: the dispersant is selected from the group consisting of polycarboxylate, ligninsulfonate, alkyl naphthalenesulfonate, TERSPERSE 2425 (produced by Huntsman, USA, alkyl naphthalenesulfonates); The emulsifier is selected from agricultural milk 700 # (common name: alkyl phenol formaldehyde resin polyoxyethylene ether), agricultural milk 2201, Span-60 # (common name: sorbitan monostearate), emulsifier T-60 (generic name: sorbitan monostearate polyoxyethylene ether), agricultural milk 1601 # (generic name: phenethylphenol polyoxyethylene polyoxypropylene ether), TERSPERSE 4894 (Huntsman, USA) (Produced by the company); wetting agent selected from alkyl phenol polyoxy vinyl ether formaldehyde condensate sulfate, alkyl phenol polyoxy vinyl ether phosphate, phenethyl phenol polyoxy vinyl ether phosphate, alkyl sulfate, Alkyl sulfonate, naphthalene sulfonate, TERSPERSE 2500 (produced by Huntsman, USA); thickener selected from xanthan gum, polyvinyl alcohol, bentonite, magnesium aluminum silicate; preservative selected from formaldehyde, benzene Formic acid, sodium benzoate; defoamer is a silicone-based defoamer; antifreeze is selected from ethylene glycol, propylene glycol, glycerin, urea, inorganic salts (such as chlorine Sodium).

For suspension emulsions, the auxiliary agents that can be used are: the dispersant is selected from polycarboxylate, ligninsulfonate, alkyl naphthalenesulfonate (diffusing agent NNO), TERSPERSE 2425; the emulsifier is selected from agricultural milk 700 #, Agricultural milk 2201, Span-60 #, Tween-60 # (generic name: sorbitan monostearate polyoxyethylene ether), agricultural milk 1601 #, triphenyl ethyl phenol polyoxyethylene ether phosphate Ester, TERSPERSE 4894; wetting agent selected from alkylphenol polyoxyvinyl ether formaldehyde condensate sulfate, alkylphenol polyoxyvinyl ether phosphate, phenethylphenol polyoxyvinyl ether phosphate, alkyl sulfate Salt, alkyl sulfonate, naphthalene sulfonate, TERSPERSE 2500; solvent selected from xylene, toluene, cyclohexanone, solvent oil (S-150, S-180, S-200); thickener selected from yellow Raw gum, polyvinyl alcohol, bentonite, magnesium aluminum silicate; preservative selected from formaldehyde, benzoic acid, sodium benzoate; defoamer is a silicone-based defoamer; antifreeze is selected from ethylene glycol, propylene glycol, glycerin, urea Inorganic salts such as sodium chloride; stabilizers selected from triphenyl phosphite, epichlorohydrin, acetic anhydride; water is deionized water.

For dispersable oil suspending agents, the auxiliary agents that can be used are: the dispersant is selected from polycarboxylate, ligninsulfonate, alkyl naphthalenesulfonate (diffusing agent NNO), TERSPERSE 2425; Castor oil polyoxyethylene ether) series emulsifiers (BY-110, BY-125, BY-140), agricultural milk 700 #, agricultural milk 2201, Span-60 #, Tween-60 #, agricultural milk 1601 #, TERSPERSE 4894; wetting agent selected from alkylphenol polyoxyethylene ether formaldehyde condensate sulfate, alkylphenol polyoxyethylene ether phosphate, phenethylphenol polyoxyvinyl ether phosphate, alkyl sulfate, alkyl Sulfonate, naphthalene sulfonate, TERSPERSE 2500; thickener selected from white carbon black, polyvinyl alcohol, bentonite, magnesium aluminum silicate; antifreeze selected from ethylene glycol, propylene glycol, glycerin, urea, inorganic salts (Such as sodium chloride); the stabilizer is selected from epoxy soybean oil, epichlorohydrin, triphenyl phosphate; the dispersion medium is selected from soybean oil, rapeseed oil, corn oil, methyl oleate, diesel oil, engine oil, mineral oil .

For emulsifiable concentrates, the auxiliaries that can be used are: emulsifier selected from calcium dodecylbenzenesulfonate (Nongmao 500 #), Nongmao 700 #, Nongma 2201 #, Span-60 # (generic name: Yamanashi Alkyd monostearate), emulsifier T-60, TX-10 (common name: octylphenol polyoxyethylene (10) ether), agricultural milk 1601 #, agricultural milk 600 #, agricultural milk 400 #; solvent Selected from xylene, solvent oil (S-150, S-180, S-200), toluene, biodiesel, methylated vegetable oil; co-solvents: ethyl acetate, methanol, dimethylformamide, cyclohexanone, Acetone; the stabilizer is selected from triphenyl phosphite, epichlorohydrin, acetic anhydride.

For microemulsions, the auxiliary agents that can be used are: the emulsifier is selected from calcium dodecylbenzenesulfonate (agricultural milk 500 #), agricultural milk 700 #, agricultural milk 2201 #, Span-60 #, Tween-60 #, TX-10, agricultural milk 1601, agricultural milk 600 #, agricultural milk 400 #; co-emulsifier selected from methanol, isopropanol, n-butanol, ethanol; solvent selected from cyclohexanone, N-methylpyrrolidone, Xylene, toluene, and solvent oil (brands: S-150, S-180, S-200); the stabilizer is selected from triphenyl phosphite and epichlorohydrin.

For microcapsule suspending agents, the auxiliaries and fillers that can be used are: solvents, emulsifiers, capsular monomers, wetting and dispersing agents, antifreeze agents, thickeners and water. The solvent is selected from toluene, xylene, dimethylformamide, cyclohexanone, acetone, dimethyl sulfoxide, and N-methylpyrrolidone; the emulsifier is selected from agricultural milk 700 #, agricultural milk 2201, span -60 #,

Tween-60 #, TX-10, agricultural milk 1601; the capsule-forming monomers are polyether polyols (specification 6305 (GB12008.2-89)), polyol-modified MDI (products Specification is one of PMM-20, NCO (free isocyanuric acid) content is 27.0-28.0%, acid content $\leq 0.1\%$, viscosity $150 \pm 50\text{mPas}$ / $25\text{ }^{\circ}\text{C}$); wetting and dispersing agent is selected from alkylphenol formaldehyde Resin polyoxyethylene ether sodium sulfate (SOPA), alkylphenol polyoxyvinyl ether phosphate, phenethylphenol polyoxyvinyl ether phosphate; antifreeze selected from ethylene glycol, propylene glycol, glycerin; thickener selection From xanthan gum, polyvinyl alcohol, bentonite; the preservative is selected from formaldehyde, benzoic acid, sodium benzoate; water is deionized water. Preparation process: after dissolving the first active ingredient and the second active ingredient with a solvent, adding a capsule-forming monomer and an emulsifier and mixing them as an oil phase, and using deionized water as an aqueous phase. Add the oil phase to the water phase and cut under a high-speed shearing machine to make the particle size of the emulsion to 1-3 microns, and then control the temperature and stirring speed to solidify the microcapsules. Stop the encapsulation reaction within one hour, and then add the dispersant, thickener, preservative, and antifreeze, and stir well.

The composition of the invention is reasonable, the insecticidal effect is good, the drug cost is low, and its activity and insecticidal effect are not a simple superposition of the activities of the components, but have a significant synergistic effect, which can slow the generation of resistance and be safe for crops Good performance, meet the safety requirements of pesticide preparations. The invention has better control effect on rice cockroaches, rice cockroaches, rice leaf rollers, cotton bollworm, apple leaf roller moth, vegetable beet armyworm, lepidoptera pests.

detailed description

In order to control rice pests, rice borers, rice leaf rollers, cotton bollworm, apple leaf curler moth, vegetable beet armyworm pests in agricultural production, the inventors combined methoxid with dimethrin and biphenyl Synergistic studies have been carried out on ingredients such as permethrin or lambda-cyhalothrin, which are:

1)The scientific name of rice, *Chilo supersuperssalis*, Lepidoptera, Spodoptera, also known as the core borer; *Tryporyza incertulas* (walker), Lepidoptera, Spodoptera, and family; The results of the indoor virulence measurement of Lepidoptera, Spodoptera, and Pesticides on rice cockroaches could represent the results of indoor virulence on rice leaf rollers and rice cockroaches. The test insects, the test agent, methoxid hydrazide, the original drug produced by the American Dow AgroSciences Co., Ltd., and the ether pyrethrin, the original drug produced by Jiangsu Huifeng Agricultural Chemical Co., Ltd., bifenthrin The drug uses the original drug produced by Jiangsu Yangnong Chemical Co., Ltd., and the beta-cypermethrin original drug uses the original drug produced by Jiangsu Changlong Chemical Co., Ltd., and is formulated by the technical department of Shaanxi Shanggezhiu Biological Science Co., Ltd. For the required test agents and test methods, please refer to "Agricultural Industry Standard of the People's Republic of China NY / T1154.2-2006". First, set 5 different concentration gradients for each single agent and each mixed agent (based on the preliminary test results, the death rate of rice cockroach pupa is set in the range of equal proportions within the range of 5% to 90%). In order to collect *dioscorea sibiricum* from the field, select the *dioscorea sibiricum* with uniform age in the laboratory, starve for 4 to 8 hours, use a 1 cm diameter punch to peel the leaf dish, put it into the culture and place it in a dish, and pay attention Moisturizing. Use a capillary dropper to start from a low concentration, drip 1 μL to 2 μL of liquid medicine on each leaf dish, and after the solvent evaporates, combine with another leaf dish coated with starch paste to make a poisoned leaf dish. Place it in a 12-hole tissue after making it. Inside the wells of a culture plate. Each treatment was repeated 4 times, each repetition was no less than 12 poisonous leaf dishes, and the treatment with the corresponding organic solvent without medicament was set as a control. One test insect was inserted into each well of the tissue advance consciousness board and placed in normal Cultivate under conditions. After 2 to 4 hours of inoculation, wait for the test insects to eat the medicine-containing leaf dish, add clean feed to the wells of the culture plate and continue to raise them until the investigation. Check the number of dead insects 24 hours and calculate the mortality.

2)Cotton bollworm (*Helibthis armigera* Hiibner), the source of the test agent is as described in 1), the technical department of Shaanxi Shanggezhiu Biological Science Co., Ltd. formulates the original test agent into the required test agent. The Agricultural Industry Standard of the People's Republic of China, NY / T1154.2-2006 "is used as a reference. Firstly, a single agent and each compounded agent are set with 5 different concentration gradients. (Based on the preliminary test results, the cotton bollworm mortality is 5% to 90%. (% Range is set according to the scale of equal proportions). The test insects are cotton bollworms collected from the field. The cotton bollworms with uniform third instar period are selected in the laboratory. After starvation for 4-8 hours, 50 larvae are selected and weighed by electronic balance. Weight, calculate the average mass per head.

Connected to the tissue culture plate, every empty worm, each treatment was repeated 4 times, and cultured under normal conditions. The number of dead insects was checked at 4 days, and the total number of dead insects and the number of dead insects were recorded. The blank control was treated with an equivalent amount of the corresponding organic solvent without the agent.

3)The apple leaf curl moth (*Adoxophyes orana*), the source of the test agent is as described in 1). The technical department of Shaanxi Shanggezhi Road Biological Science Co., Ltd. formulates the original drug into the required test agent. /T1154.2-2006".

4)Cauliflower beet armyworm *Laphygma exigua* Hubner Lepidoptera, Noctuidae. Alias *Spodoptera exigua*. The source of the test agent is as described in 1). The technical department of Shaanxi Shanggezhi Road Biological Science Co., Ltd. formulates the original drug into the required test agent.

The co-toxicity coefficient (CTC value) of the mixture is calculated according to the following formula:

In the formula: ATI——Mixed virulence index;

S——standard insecticide LC50, unit is milligram per liter (mg / L);

M——mixture LC50, unit is milligram per liter (mg / L);

$$TTI = TIA \times PA + TIB \times PB$$

In the formula: TTI——theoretical virulence index of the mixture;

TIA——A drug virulence index;

PA——the percentage content of A agent in the mixture, the unit is percentage (%);

TIB——B drug virulence index;

PB——Percentage of B agent in the mixture, the unit is percentage (%);

In the formula: CTC——co-toxicity coefficient;

ATI——mixture measured virulence index;

TTI——Theoretical Toxicity Index of Mixtures;

The co-toxicity coefficient (CTC) ≥ 120 of the compound preparation is synergistic; CTC ≤ 80 is antagonistic; 80 < CTC < 120 is additive.

4)The measurement results are shown in Tables 1 to 4.

Table 1 Determination of indoor virulence of methamphetamine and pyrethroid insecticides on rice borer

Table 2 Indoor virulence measurement of cotton worms combined with methoxid and pyrethroid insecticides

Table 3 Indoor virulence measurement of methanil hydrazide and pyrethroid insecticides to the apple leaf roller moth

Table 4 Indoor virulence measurement of methoxid and pyrethroid insecticides against Cauliflower beet armyworm

The results of laboratory virulence tests showed that the combination of methotrexate with ethethrin, bifenthrin, or lambda-cyhalothrin pyrethroid insecticides was applied to rice diploid, trilobate, and longitudinal leaf curl. Pupae, cotton bollworm, apple leaf curler moth, and vegetable beet armyworm have good control effects. When the weight ratio is 40: 1 to 1:10, the performance is significantly enhanced.

The content of the present invention will be further described below with reference to formulation examples.

Formulation Example 1:

Weigh 40% Methofenazide, 40% Ethethrin, 3% TERSPERSE 2700 (Polycarboxylate, manufactured by Huntsman, USA), 2% Diffusion Agent NNO (Alkyl Naphthalene Sulfonate Formaldehyde Condensate) , 1% opened powder BX (sodium dibutyl naphthalenesulfonate), 1% K-12 (sodium lauryl sulfate), 3% soluble starch, 3% glucose, and kaolin added to 100% by weight. The above raw materials are prepared by a conventional method for preparing water-dispersed granules, that is, mixing, ultra-micro airflow pulverization, mixing, and granulation steps to obtain 80% methionazine · permethrin water-dispersed granules.

Formulation Example 2

Weigh 40% trimethoprim, 10% bifenthrin, 3% TERSPERSE 2700, 4% sodium lignin sulfonate, 1% pulverized powder BX, 1% K-12, 1% carboxymethyl cellulose, 3% urea, diatomaceous earth to 100% by weight. The above raw materials are prepared by conventional methods for preparing water-dispersed granules, that is, mixing, ultra-micro airflow pulverization, mixing, and granulation steps to prepare 50% methionazine · bifenthrin water-dispersed granules.

Formulation Example 3:

Weigh 50% Methofenozide, 5% Lambda-cyhalothrin, 4% TERSPERSE 2700, 1% pulverized powder BX, 1% K-12, 0.8% dextrin, 5% ammonium sulfate, attapulgit Add to 100% by weight. The above raw materials are prepared by conventional methods for preparing water-dispersible granules, namely mixing, ultra-micro airflow pulverization, mixing, and granulation steps to prepare 55% methicylhydrazine · cyhalothrin water-dispersing granules.

Formulation Example 4:

Weigh 4% Methofenozide, 40% Ethethrin, 4% Morwet D-425 (alkylnaphthalenesulfonate, produced by AkzoNobel), 3% diffusing agent NNO (alkylnaphthalenesulfonate formaldehyde condensation) Material), 2% pulverized powder BX, 2% K-12, 2% polyvinylpyrrolidone, 0.5% xanthan gum, and kaolin added to 100% by weight. The above raw materials are conventionally prepared into a water-dispersible granule by mixing, ultra-micro airflow pulverization, mixing, and granulating steps to prepare 44% methionazine · permethrin water-dispersible granules.

Formulation Example 5

Weigh 60% trimethoprim, 1.5% bifenthrin, 5% calcium lignosulfonate, 2% pulverized powder BX, 1% K-12, 3% white carbon black, and kaolin to 100% by weight Serving. The above raw materials were mixed and pulverized by air to obtain 61.5% methionazide · bifenthrin wettable powder.

Formulation Example 6

Weigh 50% methicylhydrazide, 2.5% lambda-cyhalothrin, 4% calcium lignosulfonate, 3% TERSPERSE 2700, 2% pulverized powder BX, 1% K-12, 3% white carbon black, Light calcium carbonate is added to 100% by weight. The above raw materials were mixed and pulverized by air to obtain 52.5% methicylhydrazine · cypermethrin wettable powder.

Formulation Example 7

Weigh 5% Methofenozide, 10% Ethethrin, 4% Calcium Lignin Sulfonate, 4% Diffusion Agent NNO (Alkyl Naphthalene Sulfonate Formaldehyde Condensate), 2% Open Powder BX, 2% K-12, 5% white carbon black, attapulgit are added to 100% by weight. The above raw materials were mixed and pulverized by air to obtain 15% methionazine · permethrin wettable powder.

Formulation Example 8

Weigh 20% trimethoprim, 10% lambda cyhalothrin, 4% sodium lignin sulfonate, 2% TERSPERSE 2700, 2% K-12, 3% white carbon black, and clay to 100% by weight . The above raw materials were mixed and pulverized by air to obtain 30% methoxidazin · cypermethrin wettable powder.

Formulation Example 9

Weigh 10% Methofenozide, 15% Ethethrin, 3% TERSPERSE 4894, 2% TERSPERSE 2500, 1% TERSPERSE 2425, 0.2% xanthan gum, 5% ethylene glycol, 0.3% benzoic acid, 0.5% Silicone defoamer (trade name: s-29 produced by Nanjing Sixin Application Chemical Co., Ltd.) is added to 100% by weight of deionized water. The above raw materials were mixed and dispersed at high speed for 30 minutes, and then sand-milled with a sand mill to obtain a 25% methionid-hydrazine suspension agent.

Formulation Example 10:

Weigh 10% Methofenozide, 5% Bifenthrin, 1.5% NNO, 1% TERSPERSE 2500, 2% Emulsifier T-60, 3% Agricultural Milk 700 #, 0.1% Xanthan Gum, 5% Propylene Glycol, 0.5% formaldehyde, 0.3% silicone defoamer deionized water was added to 100% by weight. The above raw materials were mixed and dispersed at high speed for 30 minutes, and then sand-milled with a sand mill to obtain a 15% methotrexate · bifenthrin suspension.

Formulation Example 11:

Weigh 24% Methofenazide, 6% **Lambda-cyhalothrin**, 3% TERSPERSE 4894, 1% TERSPERSE2500, 1% TERSPERSE 2425, 0.2% xanthan gum, 1% magnesium aluminum silicate, 5% ethylene glycol Alcohol, 0.3% sodium benzoate, 0.5% silicone defoamer deionized water were added to 100% by weight. The above raw materials were mixed and dispersed at high speed for 30 min, and then sand-milled with a sand mill to obtain 30% methicylhydrazine · cyhalothrin suspension.

Formulation Example 12:

Weigh 10% Methofenozide, 10% Ethethrin, 3% Agricultural Milk 700 #, 2% Agricultural Milk 1601 #, 1.5% TERSPERSE2500, 0.5% TERSPERSE 2425, 0.2% Xanthan Gum, 3% Silica Black, 5% ethylene glycol, 0.3% benzoic acid, 0.5% silicone defoamer deionized water was added to 100% by weight. The above raw materials were mixed, sheared and dispersed at high speed for 30 minutes, and then sand-milled with a sand mill to obtain a 20% methionid-hydrazine suspension agent.

Formulation Example 13

Weigh 5% Methofenozide, 5% Ethethrin, 2% TERSPERSE 2425, 4% Span-60 #, 3% Tween-60 #, 2% Agricultural Milk 700 #, 1% TERSPERSE 2500, 0.5 % White carbon black, 5% propylene glycol, 1% epichlorohydrin, and methyl oleate were added to 100% by weight. The above raw materials were mixed and dispersed at high speed for 30 minutes, and then sand-milled with a sand mill to obtain a 10% methionazine-permethrin dispersible oil suspension agent.

Formulation Example 14

Weigh 18% methicylhydrazide, 2% bifenthrin, 2% diffusing agent NNO, 3% agricultural milk 2201 #, 8% BY-110, 1% TERSPERSE2500, 1% magnesium aluminum silicate, 3% Propylene glycol, 15% epoxy soybean oil, and soybean oil were added to 100% by weight. The above raw materials were mixed and dispersed at high speed for 30 minutes, and then sand-milled with a sand mill to obtain a 20% methionazine · bifenthrin dispersible oil suspension agent.

Formulation Example 15

Weigh 5% Methofenozide, 10% **Lambda-cyhalothrin**, 3% TERSPERSE 2425, 3% Span-60 #, 3% Tween-60 #, 8% BY-110, 1% TERSPERSE 2500, 0.5% white carbon black, 5% propylene glycol, 20% epoxy soybean oil, and methyl oleate are added to 100% by weight. The above raw materials were mixed and dispersed at high speed for 30 minutes, and then sand-milled with a sand mill to obtain 15% methicylhydrazine · cyhalothrin dispersible oil suspension agent.

Formulation Example 16

Weigh 5% Methofenozide, 15% Ethethrin, 3% Diffusion NNO, 5% BY-125, 4% Span-60 #, 4% Agricultural Milk 1601 #, 1% Phenylphenol Oxyvinyl ether phosphate, 0.5% bentonite, 5% glycerol, 3% triphenyl phosphate, and rapeseed oil were added to 100% by weight. The above raw materials are mixed and dispersed at high speed for 30 minutes, and then sand-milled with a sand mill to obtain a 20% methionazine-permethrin dispersible oil suspension agent.

Formulation Example 17

Weigh 15% trimethoprim, 5% lambda-cyhalothrin, 3% TERSPERSE 2425, 4% agricultural milk 2201 #, 2% agricultural milk 700 #, 5% BY-110, 1% TERSPERSE 2500, 0.5 % White carbon black, 5% propylene glycol, 20% epoxy soybean oil, and methyl oleate were added to 100% by weight. The above raw materials were mixed

and dispersed at a high speed for 30 minutes, and then sand-milled with a sand mill to obtain 20% methicylhydrazine · cypermethrin dispersible oil suspension agent.

Formulation Example 18

Weigh 5% Methofenozide, 5% Ethethrin, 4% TX-10, 5% Agricultural Milk 700 #, 6% Agricultural Milk 500 #, 4% Agricultural Milk 1601 #, 15% Cyclohexanone, 5 % N-methylpyrrolidone, 5% n-butanol, 1% epichlorohydrin, completely dissolved and mixed uniformly, deionized water was added to 100% by weight, and 10% methacylhydrazine · ether was prepared after stirring Permethrin microemulsion.

Formulation Example 19

Weigh 9% trimethoprim, 3% bifenthrin, 5% agricultural milk 400 #, 4% agricultural milk 500 #, 6% agricultural milk 1601 #, 25% cyclohexanone, 5% xylene, 5 % Isopropanol and 3% triphenyl phosphite were completely dissolved and mixed uniformly, deionized water was added to 100% by weight, and a 12% fenozofen · bifenthrin microemulsion was prepared after stirring.

Formulation Example 20

Weigh 3% Methofenozide, 2% **Lambda-cyhalothrin**, 6% Agricultural Milk 2201 #, 5% Agricultural Milk 700 #, 6% Agricultural Milk 600 #, 10% Cyclohexanone, 5% N- Methylpyrrolidone, 5% toluene, 5% ethanol, 1% epichlorohydrin, completely dissolved and mixed uniformly, deionized water was added to 100% by weight, and 5% methofenazide · efficient chlorine was obtained after stirring. Cyhalothrin microemulsion.

Formulation Example 21

Weigh 8% Methofenozide, 5% Ethethrin, 4% TX-10, 8% Agricultural Milk 600 #, 6% Agricultural Milk 500 #, 20% Cyclohexanone, 10% N-methylpyrrolidone, 5% toluene, 1% epichlorohydrin, completely dissolved and mixed uniformly, and deionized water was added to 100% by weight, and then a 13% methionazine · permethrin microemulsion was prepared.

Formulation Example 22

Weigh 8% Methofenozide, 10% Ethethrin, 4% Agricultural Milk 500 #, 2% Agricultural Milk 600 #, 3% Agricultural Milk 2201 #, 3% Triphenyl Phosphite, 16% Dimethyl Formamide and xylene are added to 100% by weight. The above raw materials were mixed and stirred to dissolve completely to obtain 18% methoxyfenozide · permethrin EC.

Formulation Example 23

Weigh 6% trimethoprim, 10% bifenthrin, 4% agricultural milk 500 #, 3% agricultural milk 400 #, 5% agricultural milk 1601 #, 1% epichlorohydrin, 10% ethyl acetate , 10% acetone, S-150 to 100% by weight. The above raw materials were mixed, stirred and completely dissolved to obtain 16% methoxyfenozide · bifenthrin emulsifiable concentrate.

Formulation Example 24

Weigh 10% methicylhydrazide, 3% lambda-cyhalothrin, 4% Span-60 #, 3% emulsifier T-60, 3% agricultural milk 2201 #, 3% acetic anhydride, 25% acetone, 10% ethyl acetate and biodiesel were added to 100% by weight. The above raw materials were mixed and stirred to dissolve completely to obtain 13% methicylhydrazine · cypermethrin emulsifiable concentrate.

Formulation Example 25

Weigh 5% Methofenozide, 7.5% Ethethrin, 1.5% Diffusion NNO, 4% TERSPERSE 4894, 2.5% Triphenethylphenol polyoxyvinyl ether phosphate, 5.0% Cyclohexanone, 5% Toluene, 2.5% Span-60 #, 3.5% Tween-60 #, 1% TERSPERSE2500, 2% magnesium aluminum silicate, 0.2% xanthan gum, 5% ethylene glycol, 1% epichlorohydrin, 0.3 % Benzoic acid, 0.5% silicone defoamer, deionized water was added to 100% by weight. Operation steps: 1. Add the emulsifier and mix evenly after dissolving the etherethrin and the solvent in the above raw materials. 2. Mix the other materials evenly. 3. Put the mixed materials in 1 into the materials in 2

and shear and disperse for 30 minutes at high speed. After sanding with a sand mill, a 12.5% methicylhydrazine · permethrin suspension emulsion was prepared.

Formulation Example 26

Weigh 12% methicylhydrazide, 3% lambda-cyhalothrin, 1.0% TERSPERSE 2700, 4% TERSPERSE4894, 2.5% agricultural milk 1601 #, 6% xylene, 3.5% agricultural milk 2201, 1% TERSPERSE 2500, 0.2% xanthan gum, 5% propylene glycol, 1% epichlorohydrin, 0.3% sodium benzoate, 0.3% silicone defoamer, and deionized water was added to 100% by weight. Operation steps: 1. After the lambda-cyhalothrin and solvent in the above materials are completely dissolved, add emulsifier and mix well. 2. Other materials are mixed evenly. 3. Put the mixed materials in 1 into 2 materials and cut at high speed. Disperse for 30 minutes and sand mill with a sand mill to obtain a 15% methicylhydrazine · cypermethrin suspension emulsion.

Formulation Example 27

Weigh 8% trimethoprim, 4% bifenthrin, 8% toluene, 20% N-methylpyrrolidone, PMM-203% encapsulating monomer, TX-104% emulsifier, 2201 emulsifier %, Dispersant alkylphenol polyoxyvinyl ether phosphate 4%, thickener xanthan gum 0.3%, antifreeze ethylene glycol 4%, 0.5% benzoic acid and deionized water make up 100%. After dissolving the two active components of methicylhydrazine and bifenthrin with toluene, N-methylpyrrolidone as a solvent, the vesicular monomer and emulsifier are added and mixed as an oil phase. . At a high-speed shearing machine at a speed of 8000 rpm, the oil phase is added to the water phase and sheared for 10-15 minutes to obtain an emulsion. The particle size of the emulsion can be measured by sampling, and the average particle size can be 3 microns. The emulsion is then transferred to a reaction kettle with a thermometer and a stirrer, and the heating temperature is 45 ° C, and the stirring speed is 500 rpm, so that the capsular monomers slowly undergo interfacial polymerization on the emulsion particles, and on the surface of the particles Microporous structure capsules gradually formed. After the system is reacted for 3 to 3.5 hours, a sample is taken to measure the particle size, and the average particle size is measured to be about 5 microns, and the encapsulation reaction phase is completed. The temperature was then raised to 65 ° C, and the reaction of the system was transferred from the cyst formation reaction stage to the capsular skin curing stage, and the curing stage time was 1-1.5 hours. After the bladder solidification stage is completed, the system temperature is returned to room temperature, and then wetting and dispersing agents, thickeners, antifreeze agents, and preservatives are added, and the mixture is stirred at 800 rpm for 30 minutes to obtain 12% methionyl. Hydrazine Bifenthrin Microcapsule Suspension.

Formulation Example 28

Weigh 5% trimethoprim, 2.5% lambda-cyhalothrin, solvent xylene 10%, 20% N-methylpyrrolidone, encapsulating monomer polyether polyol 63054%, emulsifier agricultural milk 700 # 3%, emulsifier 22012%, dispersant phenethylphenol polyoxyethylene ether phosphate 4%, alkylphenol formaldehyde resin polyoxyethylene ether sodium sulfate (SOPA) 2%, thickener xanthan gum 0.3%, Antifreeze propylene glycol 4%, 0.5% sodium benzoate, deionized water make up 100%. After dissolving the two active components, methoxazide and lambda-cyhalothrin, with solvents xylene and N-methylpyrrolidone, heat them and dissolve them. Add the cyst-forming monomer and emulsifier and mix as an oil phase. For the water phase. At a high-speed shearing machine at a speed of 8000 rpm, the oil phase is added to the water phase and sheared for 10-15 minutes to obtain an emulsion. The particle size of the emulsion can be measured by sampling, and the average particle size can be 3 microns. The emulsion is then transferred to a reaction kettle with a thermometer and a stirrer, and the heating temperature is 45 ° C, and the stirring speed is 500 rpm, so that the capsular monomers slowly undergo interfacial polymerization on the emulsion particles, and on the surface of the particles Microporous structure capsules gradually formed. After the system is reacted for 3 to 3.5 hours, a sample is taken to measure the particle size, and the average particle size is measured to be about 5 microns, and the encapsulation reaction phase is completed. The temperature was then raised to 65 ° C, and the reaction of the system was transferred from the cyst formation reaction stage to the capsular skin curing stage, and the curing stage time was 1-1.5 hours. After the curing phase of the bladder is completed, the system temperature is returned to room temperature, and then a wetting and dispersing agent, a thickener, an antifreeze, and a preservative are added, and the mixture is stirred at 800 rpm for 30 minutes to obtain 7.5% methionyl. Hydrazine · Cypermethrin Microcapsule Suspension.

Biological Example 1: Field test for controlling rice cockroaches

The inventor carried out a field medicinal effect test on the control of rice cockroaches in Luxi County, Jiangxi Province on June 15, 2010, and verified the control effect, effective dose and safety of the medicament on the rice cockroaches. The test crop was rice, and the control target was *Chilo suppressalis*. Using conventional spraying method, in the tillering stage of rice, the end of the first generation of pupae of the second generation of pupae, from the end of the development of the eggs to the early stage of egg hatching, a conventional sprayer was used to spray 45 liters of liquid per acre. effect. Equilibrium jumping random sampling was used. 25 clumps of rice were surveyed in each plot, and the dry heart rate and mortality were counted. At the same time, the growth of rice was observed at 2, 7, and 14 days after the medicine, and the presence or absence of phytotoxicity to the rice was visually inspected.

Calculation method:

In the formula: Pt0 ----- Dry heart rate after treatment in the drug treatment area;

CK0 ----- Blank heart rate after treatment

Table 5 Test results of controlling rice borer

As can be seen from the table above, methoxid is combined with one selected from the group consisting of etherethrin, bifenthrin, or lambda-cyhalothrin pyrethroid insecticides. One application at the end of development to the beginning of egg hatching can effectively control the damage of *P. chinensis*, and the dosage is less than that of the control agent, which has a significant synergistic effect. It can be used for the control of *P. chinensis* and is safe for rice.

Biological Example 2: Field Test on Controlling Rice Leaf Roller

The inventor conducted a field medicinal effect test on rice leaf roller leaf roller in Xianju County, Zhejiang Province on July 16, 2009, and verified the control effect, effective dose and effect on rice of rice leaf roller leaf roller. safety. The test crop was rice, and the control target was *Cnaphalocrocis medinalis*. The manual sprayer was used for conventional spraying with a spray volume of 30 liters per acre to prevent and control the third-generation rice leaf curler. Investigation was conducted 3 days, 7 days, and 10 days after the application. Equilibrium jumping random sampling was adopted, and a total of 25 clusters of rice were sampled at 5 points in each plot, and the leaf rolling rate was calculated. Compared with the leaf rolling rate in the control area, the relative control effect was calculated. At the same time, the insect rate in the leaf was investigated. At the same time, the growth of rice was observed at 3, 7, and 10 days after the treatment, and the presence or absence of phytotoxicity to the rice was visually inspected.

Calculation method:

In the formula: Pt ----- leaf curling rate after drug treatment in the treatment area; CK ----- leaf curling rate after drug treatment in the blank control area

Table 6 Test results of controlling rice leaf roller

As can be seen from the table above, 40% Methofenazide + 40% Ethylmethrin WG, 12% Methofenazide + 3% Bifenthrin SE, 20% Methofenazide + 10% Ethyl Chloride The cyfluthrin WP has better control effect on rice leaf curler at middle and high test doses, and its fast-acting and holding period is better than that of the control single agent, which can effectively reduce the amount of each active ingredient and has a significant synergistic effect. , Safe for rice.

Biological Example 3: Field test on controlling apple leaf curl moth

The inventor conducted a field efficacy test on the apple leaf curler in Liquan County, Shaanxi Province in April 2010, and verified the control effect, effective dose and safety of the agent on apple leaf curler. The test crop was apple, and the subject was controlled for *Adoxophyes orana*. The motor sprayer was used for conventional spraying, and the medicine was applied before flowering to control the apple leaf curler. Investigation was conducted 3 days, 7 days, and 10 days after the application. Two plants were surveyed in each plot, and the number of worm packs of 100 to 200 branches in the upper canopy was recorded. During the pre-drug base survey, another 100 worm packs were stripped for the worm rate in the entire experimental area to correct the worm pack base. After the application of the investigation, the worms (larvae, pupae, and pupae) of 30 to 50 worm packs were stripped at the corresponding time and in the plot to correct the number of worm packs. The last survey needs to record the victim Number of branches. At the same time, observe the growth of apples at 3, 7, and 10 days after the drug, and visually check whether the test agent has caused any harm to the apple.

Calculation method:

In the formula: Pt ----- number of insects in the treatment area; CK ----- number of insects in the blank control area.

Table 7 Control results of apple leaf curl moth

As can be seen from the table above, 5% Methofenazide + 5% Ethylmethrin OF, 9% Methofenazide + 3% Bifenthrin ME, 15% Methofenazide + 5% highly efficient chlorine Cyfluthrin OF has better control effect on the apple leaf curler moth at medium and high test doses, and its fast-acting and holding period is better than that of the control single agent, which can effectively reduce the amount of each active ingredient and has a significant synergistic effect. Apple security.

Biological example 4 field efficacy test of controlling cauliflower beet armyworm

For the control of Cauliflower beet armyworm, please refer to the "Field Test Guidelines (I) GB / T 17980.13-2000 Insecticide for Lepidoptera Larvae of Cruciferous Vegetables". Arranged, the plot area is 30m², 4 repetitions, the insect base number is investigated before the application, and the control effect is investigated before the application and 3 or 10 days after the application. Five surveys were randomly taken from each plot, two strains were surveyed at each spot, and the total number of live larvae at different ages on each plant was surveyed.

Effectiveness calculation method:

In the formula: CK0 and CK1 are the number of worms before and after application in the blank control area, respectively;

PT0 and PT1 are the number of insects before and after application in the drug treatment area, respectively.

Table 8 Test results for controlling Cauliflower beet armyworm

Table 4 Field tests show that the combination of methotrexate with permethrin, bifenthrin, or lambda-cyhalothrin pyrethroid insecticides significantly improves the fast-acting properties of Cauliflower beet armyworm and The holding period reduces the dosage and cost, and is safe for the growth of cauliflower.

Biological example 5 field efficacy test of cotton bollworm control

For cotton cotton bollworm control, please refer to the "Field Test Guideline (I) GB / T 17980.5-2000 Insecticide for Cotton Bollworm Control". The plots of test agents, control agents and blank controls are arranged in random blocks, with a plot area of 50m². 5-6 rows of cotton plants, 4 repetitions. The middle rows of each plot were randomly sampled and fixed at 5 points. Each point fixed 5 cotton plants with cotton bollworm eggs and larvae, a total of 25. The number of larvae and eggs was recorded before the treatment. Record the number of larvae after the medicine. At least 50 eggs were collected from cotton plants other than the surveyed plants and brought back to the room for moisturizing culture at room temperature to determine the egg hatching rate at different times. Or the rate of egg hatching in the blank control area of the field shall prevail. In the last survey after the application, the number of damage to the number of buds and the effect of keeping buds need to be counted. If there are fewer buds, it is necessary to count the top bud damage rate and top preservation effect, that is, the total number of buds and bolls on the fixed plant, the total number of plants and the number of top buds. Cardinal number survey. The number of larvae and eggs on the fixed plant were investigated before application. One day after the first investigation, three days after the second investigation, and seven days after the third investigation, the damage rate of Lei Ling or top buds was also investigated.

Calculation method of pharmacodynamics:

Egg hatching rate (%) = (number of eggs hatched / total eggs) x 100%

In the formula: worm population decline rate in the PT agent treatment area; CK blank control area population decline rate

Damage rate of flower buds (%) = cumulative number of flower buds killed × 100 / total number of flower buds surveyed

Bud-preserving effect (%) = (blank damage rate of blank control area-buds damage rate of chemical treatment area) × 100 / blank damage rate of blank control area

Top bud injury rate (%) = number of injured plants at the growing point × 100 / total number of plants surveyed

Top-protection effect (%) = (top bud damage rate in blank control area-top bud damage rate in chemical treatment area) × 100 / top bud damage rate in blank control area

Table 9 Test results for controlling cotton bollworm

Table 5 Field tests show that the combination of methotrexate with ethethrin, bifenthrin, or lambda-cyhalothrin pyrethroid insecticides significantly improves the effectiveness and retention of cotton bollworm The effective period reduces the dosage and cost, and is safe for cotton growth.

To sum up, the composition of the present invention uses a two active ingredient compounding scheme, and its activity and insecticidal effect are not a simple superposition of the activities of the components. Compared with the existing single preparation, , Trilobata, Oryza sativa, Cotton bollworm, Apple leaf roller moth, Vegetable beet armyworm, Lepidoptera pests have significant insecticidal effects, and also have significant synergistic effects, which can slow the generation of resistance, The crop has good safety, low cost of medicine, and meets the safety requirements of pesticide preparations.



SUPERINTENDENCIA DE INDUSTRIA Y COMERCIO

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JUAN CARLOS CUESTA QUINTERO
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Detalle del pago

Id.	Rentístico	Concepto y Servicio	Cant.	Val. Unit.	Subtotal
1	50005-01-03	Modificaciones / Correcciones al registro Patente PCT (DESCUENTO INTERNET)	1	\$ 214,000	\$ 214,000
TOTAL					\$ 214,000