



Insecticidal incense and its production method and applications

Field of the art

The present invention relates to an insecticidal incense, a method for producing
5 such an insecticidal incense, and applications of such an insecticidal incense.

Background of the art

It is well known that pyrethroid compounds can be used for preventing and
controlling sanitary pests. On the other hand, appearance of flies around fishing
10 villages, aquatic product processing factories, waste treatment plants, livestock
houses, poultry houses, and the like, has always been very annoying. As a strategy
to prevent flies from appearing, in addition to using insecticides such as insecticidal
emulsion, oil, powder, and so on, ordinary families generally use an insecticidal
spray which however has the defect of so-called "one-off" and lacks sustaining
15 effect. Hence, it is desired to develop a fly-repellent incense, which, as a space
treatment agent, not only can consistently function for hours but also has a great
diffusibility. Therefore it can be used for repelling flies even in an open space.

At present, the commercial mosquito-repellent stick incense comprising
Es-allothrin and transfluthrin as its major active ingredients is convenient for use
20 and can keep a long-term space treatment from being ignited to burned out. It has
stable volatilization and efficacy, and is recognized as a very reasonable manner for
killing pests. This type of mosquito-repellent incense, however, generally has a poor
effect on flies and thus is not suitable for being used as a fly-repellent incense, even
with an increased concentration of the active ingredients in the stick incense.

25 Therefore, selection of the most suitable active ingredients for a fly-repellent
incense is an important project. Japanese Patent No. JP10-203905 discloses a
fly-repellent incense comprising
2-allyl-3-methyl-cyclopent-2-ene-1-oxo-4-yl-2,2,3,3-tetramethylcyclopropane
carboxylate as its active ingredient, but its effect is not satisfying. Chinese patent No.
30 CN200810132507.7 discloses a fly-repellent incense comprising chlorempenthrin



as its active ingredient, but it has disadvantages such as strong irritating smell, etc., despite its improvement in fly-repellent effect.

An insecticidal incense is produced in the present invention with 2,3,5,6-tetrafluoro-4-methoxymethylbenzyl-1R-3-(2,2-dichlorovinyl)-2,2-dimethylcyclopropane carboxylate as its active ingredient. This insecticidal incense has great effects on preventing and controlling mosquitoes and flies, and also overcomes the disadvantage of irritating smell of the previous products. Therefore, this insecticidal incense is suitable for use in different occasions such as outdoors, indoors, etc..

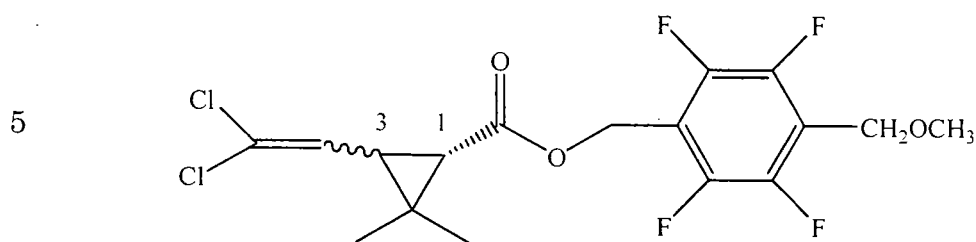
10 Disclosure of the invention

The present invention is intended to provide an insecticidal incense, which has great effects on preventing and controlling sanitary pests such as mosquitoes, flies, and the like.

The present invention also provides a method for producing this new insecticidal incense and its applications.

The objects of the present invention are achieved through the following technical embodiments:

An insecticidal incense is provided, which contains 0.002%~5.0% by weight of 2,3,5,6-tetrafluoro-4-methoxymethylbenzyl-1R-3-(2,2-dichlorovinyl)-2,2-dimethylcyclopropane carboxylate, i.e. meperfluthrin with a structure represented by the following formula (X), as the insecticidal active ingredient A.



(X)

The compound with the structure represented by the formula (X) has two individual isomers, i.e.

10 2,3,5,6-tetrafluoro-4-methoxymethylbenzyl-1R,trans-3-(2,2-dichlorovinyl)-2,2-dimethylcyclopropane carboxylate and

2,3,5,6-tetrafluoro-4-methoxymethylbenzyl-1R,cis-3-(2,2-dichlorovinyl)-2,2-dimethylcyclopropane carboxylate. Thus, the active ingredient A may be any one or a

15 mixture at any ratio of the above two.

The insecticidal incense may also further contain C8-C14 calcium alkylbenzene sulfonate which accounts for 0.002%~0.2% of the total weight of the insecticidal incense, and C8-C14 polyoxyethylene alkyl ether which accounts for 0.002%~0.2% of the total weight of the insecticidal incense. C8-C14 calcium

20 alkylbenzene sulfonate and C8-C14 polyoxyethylene alkyl ether are conventionally selected surfactants, with which the insecticide can be produced in an emulsion form.

The insecticidal incense may also further contain a di-*tert*-butylphenol-type stabilizer, which accounts for 0.01%~1.0% of the total weight of the insecticidal

25 incense.

The di-*tert*-butylphenol-type stabilizer can be one or a mixture of two or more selected from 2,6-di-*tert*-butyl-4-methylphenol (BHT),

2,2-methylene-di-(4-methyl-6-*t*-butylphenol),

2,2-methylene-di-(4-ethyl-6-*t*-butylphenol),

30 4,4-butyldiene-di-(3-methyl-6-*t*-butylphenol) or

4,4-thio-di-(3-methyl-6-*t*-butylphenol).

A method for producing the insecticidal incense is provided in the present invention, in which the active ingredient A and the optionally added di-*tert*-butylphenol-type stabilizer are dissolved in a solvent to form a solution with a certain concentration, and then directly sprayed onto the substrate of the insecticidal incense which has been dried to form a shape in advance. Then the resultant product is dried naturally to form an insecticidal incense which contains 0.002%~5.0% by weight of the active ingredient A.

The solvent may be a saturated C10-C20 alkane solvent, which may be selected from the group consisting of decane, undecane, dodecane, tridecane, tetradecane, pentadecane, hexadecane, heptadecane, octadecane, nonadecane, and alkane mixtures including D80(Exxon Mobil Corporation, boiling point: 206°C~243°C), D100(Exxon Mobil Corporation, boiling point: 223°C~254°C), D110(Exxon Mobil Corporation, boiling point: 249°C~267°C), D130(Exxon Mobil Corporation, boiling point: 279°C~313°C), Isopar-L(Exxon Mobil Corporation, boiling point: 186°C~201°C), Isopar-M(Exxon Mobil Corporation, boiling point: 223°C~253°C), and so on.

The substrate of the insecticidal incense may be a conventional substrate, which usually consists of a combustion adjuvant (organic filling) and an adhesive.

The examples of the combustion adjuvant include dried vegetal coarse powder or powder such as wood powder, pyrethrum extraction powder, orange skin powder, palm oil powder, coconut shell powder or walnut shell powder, or carbon powder selected from charcoal powder, activated carbon powder, or coal powder, or the mixtures thereof. The adhesive may be starch selected from cassava starch, corn starch, or wheat starch, casein, methylcellulose, carboxymethylcellulose or polyvinyl alcohol and the polymers of the mixtures thereof. They can be used separately or in combination of above two or more.

The invention also provides another method for producing the insecticidal incense, which comprises the following steps:

1) mixing the active ingredient A with C8-C14 calcium alkylbenzene sulfonate,

C8-C14 polyoxyethylene alkyl ether and saturated C10-C20 alkane solvent to form a solution;

2) diluting the solution obtained from step 1) with water to form an emulsion which is then mixed evenly with substrate powder of the insecticidal incense;

5 3) shaping and drying the mixture obtained from step 2) to finally form the insecticidal incense which contains 0.002%~5.0% by weight of the active ingredient A.

A di-*tert*-butylphenol-type stabilizer may be further added to the emulsion in step 2).

10 The proportion of the water amount used for dilution in step 2) is preferably 1 weight part of the solution to 10~1000 weight parts of water.

In step 2), the mixing ratio of the diluted emulsion and the substrate powder is preferably 0.1~1.2 weight parts of the diluted emulsion to 1 weight part of the substrate of the insecticidal incense.

15 If needed, in the shaping process in step 3), water may be further added. The resultant mixture, after kneaded, is shaped into an appropriate form such as strip, coiled disk, and so on, using a mould such as a compacting machine, an extruder, an embossing machine, a stamping machine, and the like. Then the shaped mixture is dried to form the insecticidal incense of the present invention.

20 If needed, the insecticidal incense may further contain 1% or less by weight of colorant and/or preservative, and so on. The examples of the colorant include organic dyes such as malachite green and the like. The examples of the preservative include acids such as benzoic acid, sorbic acid, dehydroacetic acid, *p*-hydroxybenzoic acid, *o*-phenylphenol, etc., and the salts and esters thereof.

25 One preferable embodiment of the finally obtained insecticidal incense is shown as follow (the total weight of the insecticidal incense is represented by 100%, and the contents are shown in weight percentages): the insecticidal incense contains 0.002~5.0 of meperfluthrin (the active ingredient A), 0.002~0.2 of C8-C14 calcium alkylbenzene sulfonate, 0.002~0.2 of C8-C14 polyoxyethylene alkyl ether,
30 0.01~1.0 of di-*tert*-butylphenol-type stabilizer, 0.01~1.0 of other insecticidal active

ingredients, 1.0 or less of colorant and/or preservative, and the rest is the substrate of the insecticidal incense.

The use of the insecticidal incense in the aspect of preventing and controlling mosquitoes and flies is also provided in the present invention.

5 The insecticidal incense of the present invention can be used according to normal procedures. In other words, the insecticidal incense of the present invention may be kept alight continuously in a breeding ground for pests (such as mosquitoes, flies, and the like) or in a room, etc. As the insecticidal incense is alight, the active ingredient is volatilized and diffused, so as to control the pests.

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Detailed embodiments

The embodiments and effects of the present invention are explained in detail by Examples as follows, but the present invention is not limited to the following Examples. In the following Examples, all the proportions and 'parts' are by weight, unless indicated otherwise.

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Production Example 1

2,3,5,6-tetrafluoro-4-methoxymethylbenzyl-1R-3-(2,2-dichlorovinyl)-2,2-dimethylcyclopropane carboxylate (1R,cis : 1R,trans=1:1) (0.0007g) was dissolved in 0.1g D100 solvent oil (Exxon Mobil Corporation, boiling point: 223°C~254°C). The resultant solution was sprayed onto 35g blank incense base and dried naturally for 48 hours to form the insecticidal incense I of the present invention, which contains 0.002w/w% of the active ingredient A.

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Production Example 2

2,3,5,6-tetrafluoro-4-methoxymethylbenzyl-1R,trans-3-(2,2-dichlorovinyl)-2,2-dimethylcyclopropane carboxylate (0.014g) was dissolved in 0.5g D100 solvent oil (Exxon Mobil Corporation, boiling point: 223°C~254°C). The resultant solution was sprayed onto 35g blank incense base and dried naturally for 48 hours to form the insecticidal incense II of the present invention, which contains 0.04w/w% of the

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active ingredient A.

Production Example 3

2,3,5,6-tetrafluoro-4-methoxymethylbenzyl-1R,cis-3-(2,2-dichlorovinyl)-2,2-dimethylcyclopropane carboxylate (0.28g) and 0.08g BHT were dissolved in 1g D100 solvent oil (Exxon Mobil Corporation, boiling point: 223°C~254°C). The resultant solution was sprayed onto 35g blank incense base and dried naturally for 48 hours to form the insecticidal incense III of the present invention, which contains 0.8w/w% of the active ingredient A.

Production Example 4

2,3,5,6-tetrafluoro-4-methoxymethylbenzyl-1R-3-(2,2-dichlorovinyl)-2,2-dimethylcyclopropane carboxylate (1R,cis:1R,trans=5:95) (0.71g) was dissolved in 2g D100 solvent oil (Exxon Mobil Corporation, boiling point: 223°C~254°C). The resultant solution was sprayed onto 35g blank incense base and dried naturally for 48 hours to form the insecticidal incense IV, which contains 2w/w% of the active ingredient A.

Production Example 5

2,3,5,6-tetrafluoro-4-methoxymethylbenzyl-1R-3-(2,2-dichlorovinyl)-2,2-dimethylcyclopropane carboxylate (1R,cis:1R,trans=70:30) (1.44g) was dissolved in 4g D100 solvent oil (Exxon Mobil Corporation, boiling point: 223°C~254°C). The resultant solution was sprayed onto 35g blank incense base and dried naturally for 48 hours to form the insecticidal incense V, which contains 4w/w% of the active ingredient A.

Production Example 6

Five parts of 2,3,5,6-tetrafluoro-4-methoxymethylbenzyl-1R,trans-3-(2,2-dichlorovinyl)-2,2-dimethylcyclopropane carboxylate, 4.5 parts of calcium dodecylbenzene sulfonate, 4.5

parts of polyoxyethylene lauryl ether, and 86 parts of D100 solvent oil were mixed to form solution R1.

Ten parts of
2,3,5,6-tetrafluoro-4-methoxymethylbenzyl-1R-3-(2,2-dichlorovinyl)-2,2-dimethylcyclopropane carboxylate (1R,cis:1R,trans=30:70) , 4 parts of calcium alkylbenzene sulfonate, 4 parts of polyoxyalkylene alkyl ether, 1 part of BHT and 81 parts of D100 solvent oil were mixed to form solution R2.

Solution R1 (0.4 parts) and 49.6 parts of water were mixed to form a diluted emulsion for producing the insecticidal incense. The above diluted emulsion was mixed with 20 parts of pyrethrum extraction powder, 30 parts of wood powder, and 10 parts of water. The resultant mixture was substantially kneaded and then shaped and dried(40°C, 6 hours) to form the desired insecticidal incense VI of the present invention, which contains 0.04w/w% of the active ingredient A.

Solution R2 (0.4 parts) and 49.6 parts of water were mixed to form a diluted emulsion for producing the insecticidal incense, into which 0.02 parts of malachite green and 0.02 parts of sodium benzoate were further added. The above diluted emulsion was mixed with 20 parts of pyrethrum extraction powder, 30 parts of wood powder, and 10 parts of water. The resultant mixture was substantially kneaded and then shaped and dried(40°C, 6 hours) to form the desired insecticidal incense VII of the present invention, which contains 0.08w/w% of the active ingredient A.

Control Example 1

Allethrin (0.014g) was dissolved in 0.5g D100 solvent oil(Exxon Mobil Corporation, boiling point: 223°C~254°C). The resultant solution was sprayed onto 35g blank incense base and dried naturally for 48 hours to form the control insecticidal incense I , which contains 0.04w/w% of allethrin.

Control Example 2

Allethrin (0.28g) was dissolved in 1g D100 solvent oil(Exxon Mobil Corporation, boiling point: 223°C~254°C). The resultant solution was sprayed onto 35g blank

incense base and dried naturally for 48 hours to form the control insecticidal incense II, which contains 0.8w/w% of allethrin.

Control Example 3

5 Dimefluthrin (0.28 g) and 0.08g BHT were dissolved in 1g D100 solvent oil(Exxon Mobil Corporation, boiling point: 223°C~254°C). The resultant solution was sprayed onto 35g blank incense base and dried naturally for 48 hours to form the control insecticidal incense III, which contains 0.8w/w% of dimefluthrin.

10 Testing Example 1

The insecticidal incense II, VI of the present invention and the control insecticidal incense I, II were applied to a test for comparing the mosquito-killing efficacy in accordance with GB13917.4-92, wherein the subject insects were female *Culex pipiens pallens* which were emerged 2~3 days ago and were not fed with blood. The detailed procedure is shown as follows: 20 subject mosquitoes were collected by a mosquito absorbing tube and placed into an airtight drum-shape testing device; a portion of the insecticidal incense to be tested was picked discretionarily and positioned on an incense shelf; time started to be counted after the insecticidal incense was ignited, and the insecticidal incense was removed after 15 1 min; the number of the subject mosquitoes being knocked out was recorded every a period of time, and the value of KT50 was calculated. The result of this test is shown in Table 1.

Table 1. Comparison of the mosquito-killing efficacy between the insecticidal incenses of the present invention and the control insecticidal incenses

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Insecticidal incenses	Active ingredients	Concentration (w/w%)	KT50(min)
Insecticidal incense II of the present invention	Active ingredient A	0.04	3.4
Insecticidal incense VI of the present invention	Active ingredient A	0.04	3.5
Control insecticidal incense I	Allethrin	0.04	> 30
Control insecticidal incense II	Allethrin	0.8	4.1

Testing Example 2

The insecticidal incense III of the present invention and the control insecticidal incense III were applied to a test for their indoor efficacy of killing *Musca domestica* in accordance with GB13917.4-92. The subject insects were *Musca domestica*, which were the fourth-day adults after emergence, with half male and half female. An airtight drum-shape device was utilized. The detailed procedure is shown as follows: the subject insects were placed into the drum-shape device; a portion of the insecticidal incense to be tested was picked discretionarily and positioned on an incense shelf, after the subject insects resumed their normal activities; time started to be counted after the insecticidal incense was ignited, and the insecticidal incense was removed after 1 min; the number of the subject insects being knocked out was recorded every a period of time. The result of this test is shown in Table 2.

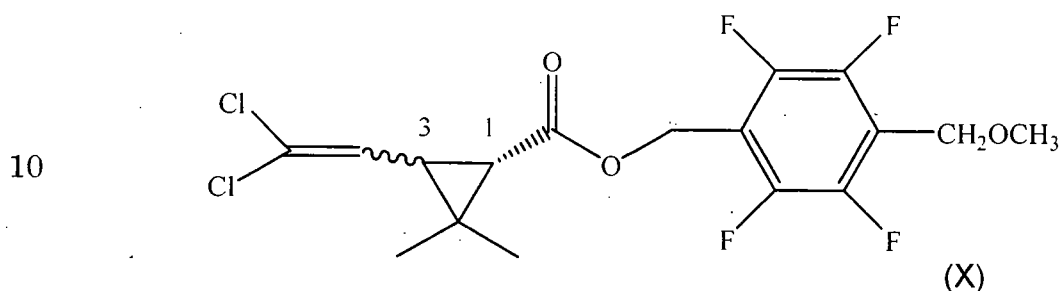
Table 2. Comparison of the fly-killing efficacy between the insecticidal incense of the present invention and the control insecticidal incense

Insecticidal incenses	Active ingredients	Concentration (w/w%)	KT50(min)
Insecticidal incense III of the present invention	Active ingredient A	0.8	3.6
Control insecticidal incense III	Dimefluthrin	0.8	13.2

Through the above tests, it is known that the insecticidal incenses of the present invention possess more notable pest-killing efficacy than any existing insecticidal incenses with other active ingredients.

Claims

1. An insecticidal incense, characterized in that the insecticidal incense comprises 0.002%~5.0% by weight of 2,3,5,6-tetrafluoro-4-methoxymethylbenzyl-1R-3-(2,2-dichlorovinyl)-2,2-dimethylcyclopropane carboxylate, i.e. meperfluthrin with a structure represented by the following formula (X), as the insecticidal active ingredient A.



15 2. The insecticidal incense of Claims 1, characterized in that the active ingredient A is one selected from 2,3,5,6-tetrafluoro-4-methoxymethylbenzyl-1R,trans-3-(2,2-dichlorovinyl)-2,2-dimethylcyclopropane carboxylate or 2,3,5,6-tetrafluoro-4-methoxymethylbenzyl-1R,cis-3-(2,2-dichlorovinyl)-2,2-dimethylcyclopropane carboxylate, which are represented by the formula (X), or a mixture of
20 thereof.

3. The insecticidal incense of Claims 1, characterized in further comprising C8-C14 calcium alkylbenzene sulfonate which accounts for 0.002%~0.2% of the total weight of the insecticidal incense, and C8-C14 polyoxyethylene alkyl ether
25 which accounts for 0.002%~0.2% of the total weight of the insecticidal incense.

4. The insecticidal incense of Claims 1, characterized in further comprising a di-*tert*-butylphenol-type stabilizer, which accounts for 0.1%~1.0% of the total weight of the insecticidal incense.

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5. The insecticidal incense of Claims 4, characterized in that the

di-*tert*-butylphenol-type stabilizer is one or a mixture of two or more selected from 2,6-di-*tert*-butyl-4-methylphenol, 2,2-methylene-di-(4-methyl-6-*t*-butylphenol), 2,2-methylene-di-(4-ethyl-6-*t*-butylphenol), 4,4-butyldiene-di-(3-methyl-6-*t*-butylphenol) or 4,4-thio-di-(3-methyl-6-*t*-butylphenol).

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6. A method for producing the insecticidal incense of Claims 1, wherein the active ingredient A and an optionally added di-*tert*-butylphenol-type stabilizer are dissolved in a saturated C10-C20 alkane solvent to form a solution with a certain concentration, and then directly sprayed onto a substrate of the insecticidal incense which has been dried to form a shape in advance; then the resultant product is dried naturally to form the insecticidal incense which contains 0.002%~5.0% by weight of the active ingredient A.

7. A method for producing the insecticidal incense of Claims 1, comprising the following steps:

1) mixing the active ingredient A with C8-C14 calcium alkylbenzene sulfonate, C8-C14 polyoxyethylene alkyl ether and a saturated C10-C20 alkane solvent to form a solution;

2) diluting the solution obtained from step 1) with water to form an emulsion which is then mixed evenly with substrate powder of the insecticidal incense;

3) shaping and drying the mixture obtained from step 2) to finally form the insecticidal incense which contains 0.002%~5.0% by weight of the active ingredient A..

8. The method of Claims 7, characterized in that a di-*tert*-butylphenol-type stabilizer is further added to the emulsion in step 2).

9. Use of the insecticidal incense of Claims 1 for preventing and controlling mosquitoes and flies.