(12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(19) World Intellectual Property Organization
International Bureau

(43) International Publication Date
10 January 2008 (10.01.2008)

PCT

(10) International Publication Number
WO 2008/004711 A2

(51) International Patent Classification:
A01N 53/00 (2006.01) *A01P 7/04* (2006.01)
A01N 25/34 (2006.01)

(21) International Application Number:
PCT/JP2007/063872

(22) International Filing Date: 5 July 2007 (05.07.2007)

(25) Filing Language: English

(26) Publication Language: English

(30) Priority Data:
2006-187606 7 July 2006 (07.07.2006) JP

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(81) Designated States *(unless otherwise indicated, for every kind of national protection available)*: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BH, BR, BW, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DO, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, GT, HN, HR, HU, ID, IL, IN, IS, KE, KG, KM, KN, KP, KR, KZ, LA, LC, LK, LR, LS, LT, LU, LY, MA, MD, ME, MG, MK, MN, MW, MX, MY, MZ, NA, NG, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RS, RU, SC, SD, SE, SG, SK, SL, SM, SV, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, ZA, ZM, ZW.

(84) Designated States *(unless otherwise indicated, for every kind of regional protection available)*: ARIPO (BW, GH, GM, KE, LS, MW, MZ, NA, SD, SL, SZ, TZ, UG, ZM, ZW), Eurasian (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European (AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IS, IT, LT, LU, LV, MC, MT, NL, PL, PT, RO, SE, SI, SK, TR), OAPI (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG).

Published:
— *without international search report and to be republished upon receipt of that report*

For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.

WO 2008/004711 A2

(54) Title: INSECT CONTROLLING MATERIAL

(57) Abstract: The object is the present invention is to provide an insect controlling material having excellent performance. The present invention relates to a net-like insect controlling material with excellent performance having substantially uniform meshes, made of filaments each of which contains a thermoplastic resin and an insect controlling component which shows a vapor pressure of 1 X 10-6 mmHg or lower at 25 C, wherein the content of the insect controlling component in the insect controlling material is from 0.1 to 10% by weight; the amount of the insect controlling component on the surface of the insect controlling material is from 0.03 to 3 g per 1 kg of the insect controlling material; the void area of each of the meshes is from 2 to 36 mm_2; and the bleed coefficient of the insect controlling component found by an acetone cleansing method is from 0.3 to 2.0.

DESCRIPTION

INSECT CONTROLLING MATERIAL

TECHNICAL FIELD

The present application claims priority based on Japanese Patent Application No. 2006-187606. The contents of that application are incorporated herein by reference thereto in its entirety.

The present invention relates to an insect-controlling material, and particularly to a net-like insect-controlling material.

Background Art

Net-like insect-controlling materials, typically mosquito nets, hitherto have been widely used as a method for protecting persons and cattle from sanitary inset pests such as mosquitoes and flies, particularly mosquitoes. The net-like insect-controlling materials, typically mosquito nets, are net-like woven materials made of natural fibers such as hemp and cotton, and these net-like materials have mesh sizes so small enough as to prevent mosquitoes, etc. from passing through the meshes of the net-like materials to thereby inhibit the mosquitoes from entering the meshes, in order that persons can be prevented and protected from

contacting the mosquitoes.

However, such a mosquito net has a problem in that, since the mesh size thereof should be such that mosquitoes can not pass through the meshes thereof in order to prevent physical contact between persons and mosquitoes, it is unavoidable to reduce the areas of such meshes, which is likely to lead to insufficient ventilation of air.

As a method for solving this problem, there is known a mosquito net made of resin filaments which are obtained by spinning a resin composition having an insect controlling component kneaded therein, and such a mosquito net therefore has not only physical but also chemical insect-controlling performance such as insect proof and insecticidal actions (cf. JP-A-6-322612).

Mosquito nets, etc. are used over a long period of time and thus are needed to be repeatedly washed because of stains thereon due to the long period of use. Therefore, there is a demand for an insect-controlling material capable of exhibiting sufficient insect-controlling performance under such conditions for use.

Disclosure of Invention

An object of the present invention is to provide an insect-controlling material, particularly a net-like insect-controlling material, having excellent performance.

As a result of the present inventor's intensive researches for solving the above problems, it has been found that the following insect-controlling material shows excellent performance. The present invention is accomplished based on such a finding.

That is, the present invention provides the following insect-controlling materials 1 to 9.

1. An insect controlling material which is a net-like material having substantially uniform meshes, made of filaments each containing a thermoplastic resin and an insect controlling component which shows a vapor pressure of 1 X 10^{-6} mmHg or lower at 25°C, characterized in that

the content of the insect controlling component in the insect controlling material is from 0.1 to 10% by weight,

the amount of the insect controlling component on the surface of the insect controlling material is from 0.03 to 3 g per 1 kg of the insect controlling material,

the void area of each of the meshes is from 2 to 36 mm^2, and

the bleed coefficient of the insect controlling component found by the acetone cleansing method is from 0.3 to 2.0.

2. An insect controlling material as defined in the item 1, wherein the insect controlling component which shows a vapor pressure of 1 X 10^{-6} mmHg or lower at 25°C is a

pyrethroid compound which shows a vapor pressure of 1 X 10^{-6} mmHg or lower at 25°C.

3. An insect controlling material as defined in the item 1, wherein the insect controlling component which shows a vapor pressure of 1 X 10^{-6} mmHg or lower at 25°C is permethrin.

4. An insect controlling material as defined in any one of the items 1 to 3, wherein the thermoplastic resin is an olefin-based resin.

5. An insect controlling material as defined in any one of the items 1 to 3, wherein the thermoplastic resin is an ethylene-based resin.

6. An insect controlling material as defined in any one of the items 1 to 3, wherein the thermoplastic resin is a resin composition which contains a low density polyethylene and a high density polyethylene.

7. An insect controlling material as defined in any one of the items 1 to 6, wherein the void area of each of the meshes is from 8 to 25 mm^2.

8. An insect controlling material as defined in any one of the items 1 to 7, wherein 0.01 to 10% by weight of a powdery carrier with a particle diameter of 0.01 to 40 μm is contained based on the weight of the insect controlling material.

9. An insect controlling material as defined in any one

of the items 1 to 8, wherein the filament is a monofilament with a thickness of 130 to 230 deniers.

According to the present invention, insects such as mosquitoes, etc. can be controlled.

Best Mode for Carrying Out the Invention

An insect-controlling material of the present invention is a net-like knitted or woven fabric having substantially uniform meshes, made of filaments comprising a thermoplastic resin and an insect controlling component which shows a vapor pressure of 1 X 10^{-6} mmHg or lower at 25°C (hereinafter optionally referred to as "the present resin filaments"). The net-like knitted or woven material has a texture of a conventionally known knitted or woven fabric, and can be made by weaving or knitting (for example, Raschel-knitting) the present resin filaments according to any of known methods.

The resin to be used in the insect-controlling material of the present invention contains a thermoplastic resin as a component, or may comprise a mixture of a thermoplastic resin, a thermoplastic elastomer, etc. As such a thermoplastic resin, there can be used olefin-based resins, polyvinyl chloride, polyvinyl alcohol, polycarbonate, polyester, polymethyl methacrylate, etc. There may be used a mixture of two or more appropriately

selected from these thermoplastic resins and thermoplastic elastomers in accordance with the kind and content of an insect controlling component to be used.

Specific examples of the olefin-based resin include polyethylenes (low density polyethylene (including linear low density polyethylene (LLDPE)), ultra-low density polyethylene, medium density polyehtylene and high density polyethylene), polyethylene resins such as a copolymer of ethylene with α-olefin having 3 or more carbon atoms, propylene homopolymer, random copolymer or block copolymer of propylene with α-olefin having 4 or more carbon atoms, and copolymers of ethylene with carboxyl derivatives having ehtylenically unsaturated bonds (e.g. ethylene-methyl methacrylate copolymer, ethylene-vinyl acetate copolymer, ethylene-acrylic acid copolymer, etc.). Each of these olefin-based resins may used alone or as a mixture with other selected from the above.

Examples of the thermoplastic elastomer include olefin-based thermoplastic elastomers, styrene-based thermoplastic elastomers, etc. Specifically, examples of the olefin-based thermoplastic elastomers are elastomers each comprising ethylene or propylene as a main component, and examples of the styrene-based thermoplastic elastomers are block copolymers of polystyrene block with polyisoprene block and/or polybutadiene block, and hydrogenated products

thereof.

The present resin filament contains an insect controlling component which shows a vapor pressure of 1 X 10^{-6} mmHg or lower at 25°C (hereinafter optionally referred to as "the present insect controlling component) in addition to the thermoplastic resin. The present resin filament can be readily manufactured by a known method, for example, by melt-spinning a thermoplastic resin composition (hereinafter optionally referred to as "the present resin composition") which comprises the thermoplastic resin and the present insect controlling component.

As the method for preparing the present resin composition, the following methods can be employed: for example, a predetermined amount of the thermoplastic resin and a predetermined amount of the present insect controlling component are mixed at once by melt-kneading, to obtain the resin composition; or a part of a predetermined amount of the thermoplastic resin and a predetermined amount of the present insect controlling component are previously melt-kneaded to prepare a master batch, and then, the master batch and the rest of the thermoplastic resin are melt-kneaded to obtain the resin composition.

In the method of using the master batch, out of the above methods for preparing the present resin compositions,

the thermoplastic resin used in the step of preparing the master batch may be the same as or different from the thermoplastic resin used in the step of obtaining the present resin composition from the master batch. In concrete, a linear low density polyethylene (LLDPE) is used in the step of preparing the master batch, and then, a high density polyethylene is used in the step of obtaining the present resin composition from the master batch.

The present resin composition may contain a powdery carrier in addition to the thermoplastic resin and the present insect controlling component. As the powdery carrier, for example, talc, kaolin, clay and silica-based fine particles, and fine powdery carriers of carbon, dextrin, etc.

When the powdery carrier is contained in the present resin composition, the average particle diameter of the powdery carrier to be used is generally from 0.01 to 40 μm, preferably from 0.03 to 20 μm. The content of the powdery carrier in the insect controlling material of the present invention is determined in consideration of appropriate bleeding property of the present insect controlling component and its insect controlling effect, etc. The content of the powdery carrier is generally from 0.01 to 10% by weight, preferably from 0.5 to 5% by weight.

To contain the powdery carrier in the present resin

composition, the powdery carrier is admixed when the present insect controlling component and the thermoplastic resin are melt-kneaded in each of the steps of the foregoing methods for preparing the present resin compositions. Alternatively, the present insect controlling component and the powdery carrier are previously mixed to prepare a carrier having the present insect controlling component carried thereon, which is then melt-mixed into the thermoplastic resin. In more detail, for example, the carrier having the present insect controlling component carried thereon, obtained by previously mixing the present insect controlling component with the powdery carrier, is melt-mixed with a part of the predetermined amount of the thermoplastic resin to prepare a master batch, which is then melt-kneaded with the rest of the thermoplastic resin.

To the present resin filament, common ingredients such as an antioxidant, pigment, lubricant, etc., which are generally added to thermoplastic resins, may be compounded, as well as the powdery carrier.

According to the insect-controlling material of the present invention, an insect-controlling effect is exhibited when the present insect controlling component bleeds to the surface of the insect-controlling material of the present invention and contacts insects at the surface

thereof.

As the present insect controlling component, there is used a relatively low evapotranspirating substance which shows a vapor pressure of 1 X 10^{-6} mmHg or lower at 25°C. Therefore, the present insect controlling component contained in the insect-controlling material of the present invention does not rapidly evaporate off after bleeding to the surface of the material, but is retained there to inhibit further bleeding of the present insect controlling component. Therefore, the insect-controlling material of the present invention can sustain its insect controlling performance over several years.

There is no particular limit in selection of the present insect controlling component, in so far as the component shows a vapor pressure of 1 X 10^{-6} mmHg or lower at 25°C. Examples thereof include pyrethroid compounds such as permethrin, cypermethrin, cyphenothrin, d-phenothrin, resmethrin, fenvalerate, esfenvalerate, fenpropathrin, etofenprox, tralomethrin, deltamethrin, silafluofen, bifenthrin, etc. and other insect controlling components.

The content of the present insect controlling component changes depending on the type thereof, usage, a period of time to be used, etc. However, the content of the present insect controlling component is such that its

insect-controlling effect can be exhibited in use, specifically 0.1 to 10% by weight, preferably 0.5 to 5% by weight, based on the weight of the insect-controlling material of the present invention.

The present insect controlling component contained in the insect-controlling material of the present invention exhibits its effect when bleeding to the surface of the insect-controlling material. In use, the amount of the present insect controlling component present on the surface of the insect-controlling material of the present invention is generally from about 0.03 to about 3 g per 1 kg of the insect-controlling material of the present invention. The amount of the present insect controlling component present on the surface of the insect-controlling material of the present invention can be easily determined by measuring the amount of the present insect controlling component which is eluted into an organic solvent such as acetone, xylene or the like, after the insect-controlling material of the present invention is cleansed with the organic solvent.

It is also needed that the insect controlling material of the present invention has a bleed coefficient of 0.3 to 2.0, preferably 0.5 to 1.2. The bleed coefficient is an index which indicates the degree of ease (or difficulty) for the present insect controlling component to bleed to the surface of the insect controlling material of the

present invention. The bleed coefficient is expressed as follows:

Bleed coefficient = (the amount of component B)/
(the amount of component A).

That is, the present insect controlling component which bleeds to the surface of the insect controlling material of the present invention is cleansed with an organic solvent such as acetone, and then, the present insect controlling component is allowed to sufficiently bleed out by heating the insect controlling material under predetermined conditions, and is then cleansed with an organic solvent. This organic solvent is recovered to measure the amount of the present insect controlling component contained therein (hereinafter referred to as the amount of component A). In the meantime, the insect controlling material of the present invention used in the above-described operation is again heated under predetermined conditions, to thereby allow the present insect controlling component to sufficiently bleed, and is then cleansed with an organic solvent. This organic solvent is recovered to measure the amount of the present insect controlling component contained therein (hereinafter referred to as the amount of component B). Thus, the bleed coefficient is expressed by the ratio of the amount of component B to the amount of component A.

As described above, the insect controlling material of the present invention is a net-like knitted or woven fabric having substantially uniform meshes, made of the present resin filaments. Insect pests are not permitted to pass through the meshes of the insect controlling material of the present invention without contacting the surface of the insect controlling material. In the meantime, the void area per one mesh is needed to be from 2 to 36 mm^2, preferably from 8 to 25 mm^2, so as to ensure the air ventilation of the insect controlling material.

The present resin filament is generally used as a monofilament for use in a net-like knitted or woven fabric having uniform meshes. Therefore, preferably, the present resin filament has a strength sufficient to be used for a conventional mosquito net, and thus, the thickness thereof is generally from 100 to 350 deniers, preferably from 130 to 230 deniers.

The insect controlling material of the present invention should satisfy all the requirements described above. The insect controlling material of the present invention which meets these requirements can be manufactured by way of proper selection and combination of conditions so as to satisfy the respective requirements defined in the present invention. That is, it is needed to appropriately select and combine the conditions, such as

the kind of the thermoplastic resin for use in the manufacturing of the present resin filament and a mixing ratio of resins, when the resin is a mixture of at least two resins; the kind and amount of the present insect controlling component; the kinds and amounts of compounding agents, if used in the present resin filament, such as powdery carrier, antioxidant, pigment, lubricant, etc.; and spinning conditions such as a spinning temperature, multiplying factor for drawing, drawing rate, cooling temperature and the thickness of filaments, when the present resin filament is spun, for example by the extrusion drawing method.

The insect controlling material of the present invention may be directly used as a net-like knitted or woven fabric as it is, or may be sewn or worked into, for example, a mosquito net or the like for use.

Next, the present invention will be described in detail by way of Examples thereof, which should not be construed as limiting the scope of the present invention in any way.

In Examples, parts are parts by weight, unless otherwise specified.

First, the production example of an insect controlling material according to the present invention will be described.

Production Example 1

Step 1

Fine silica powder (porous silica particles having an average particle diameter of 12 μm) (48.5 parts), permethrin (50 parts) and Sumilizer BHT (an antioxidant manufactured by Sumitomo Chemical Company, Limited) (1.5 parts) were mixed and stirred to prepare silica capsules each containing an insect controlling component.

Step 2

The silica capsules (31 parts) obtained in the step 1, linear low density polyethylene (density: 0.912 kg/m^3, Sumicasen L GA807 manufactured by Sumitomo Chemical Company, Limited) (59.5 parts), zinc stearate (5 parts) and a pigment (4.5 parts) were melt-kneaded at 150°C, and the resulting knead mixture was extruded from an extruder and then was cut into resin pellets (diameter: 3 mm, and length: 3 mm) each containing the insect controlling component.

Step 3

The resin pellets (14 parts) obtained in the step 2, a high density polyethylene (density: 0.950 kg/m^3, Hizex 5000S manufactured by MITSUI CHEMICALS INC.) (85.4 parts) and zinc stearate (0.6 parts) were melt-kneaded at a temperature of 220 to 240°C to obtain a resin composition containing the insect controlling component.

The composition ratio of the respective components in the resin composition were as follows:

Fine silica powder	2.0%
Permethrin	2.0%
Linear low density polyethylene	7.2%
High density polyethylene	87.23%
Zinc stearate	1.3%
BHT	0.065%
Pigment	0.21%

Step 4

The resin composition obtained in the step 3, as a raw material, was melt-spun by the extrusion method under the following conditions, to make filaments each containing the insect controlling component.

The spinning conditions were as follows:

Spinning temperature	
Cylinder temperature:	130 to 210°C
Head temperature:	190 to 200°C
Dies temperature:	150°C
Screw revolution rate:	51 rpm
Cylinder diameter (D):	50 mm
Cylinder length (L):	1,500 mm
Ratio of L/D:	30
Number of dies:	150
Take-up rate at the time of spinning:	13.7 m/sec.

Water tank temperature for drawing: 93°C or higher

Multiplying factor for drawing: 8

Filaments obtained: monofilaments

Step 5

The filaments obtained in the step 4 were woven by the Raschel method to make a net-like insect controlling material (hereinafter referred to as the present insect controlling material (1)) having a mesh size of 3 X 3 mm (void area per mesh: 9 mm^2).

The permethrin content in the present insect controlling material (1) was 2.0% by weight.

In this regard, the filaments each containing the insect controlling component obtained in the step (4) were woven into the present insect controlling material (1) after one week had passed since the manufacturing of the filaments.

(Bleed Coefficient Determining Method)

1. Weighing of Sample

The insect controlling material was optionally cut to obtain a small piece thereof as a sample, which was then weighed.

2. Preparation of Solution

(1) Dioctyl adipate (an internal standard substance, hereinafter referred to as DOA) (about 150 mg) was correctly weighed, and acetone was added thereto to prepare

an acetone solution having a DOA concentration of 3,000 ppm (hereinafter referred to as Solution A).

(2) Solution A (5 ml) was diluted with acetone to prepare 50 ml of a diluted acetone solution having a DOA concentration of 300 ppm (hereinafter referred to as Solution B) (for use in extraction).

(3) Solution A (5 ml) was diluted with acetone to prepare 50 ml of a diluted acetone solution having a DOA concentration of 300 ppm (hereinafter referred to as Solution C) (for use in analysis).

(4) Permethrin (about 150 mg) was correctly weighed, and acetone was added thereto to prepare an acetone solution having a permethrin concentration of 3,000 ppm (hereinafter referred to as Solution D).

(5) Solution D (5 ml) was diluted with acetone to prepare 50 ml of a diluted acetone solution having a permethrin concentration of 300 ppm (hereinafter referred to as Solution E) (for use in analysis).

(6) Solution C and Solution E (each 5 ml) were added to a screw tube (No. 5, 20 ml) and were mixed therein to form a standard solution.

3. Extraction Treatment

(1) The sample and Solution B (10 ml) were added to a screw tube, and were sufficiently agitated for 1 minute for extraction, and then, the sample was removed (the first

extraction treatment).

(2) The extract left to remain in the screw tube after the removal of the sample was transferred to an eggplant-type flask. The screw tube was cleansed with acetone (1 ml), and this acetone cleansing liquid was added to the above eggplant-type flask to be combined with the extract to thereby obtain a solution mixture (hereinafter referred to as Extract F).

(3) Extract F in the eggplant-type flask was concentrated under a reduced pressure, and acetone (2 ml) was added to the concentrated residue to prepare a sample for gas chromatography. By analyzing the amount of permethrin in Extract F, the amount of the insect controlling component on the surface of the insect controlling material (a surface bleed amount) was measured.

(4) The sample removed in the operation of the above step (1) was air-dried at 23°C for 20 minutes, and was then put in a new screw tube, and was left to stand alone in a thermostat at 70°C for 2 hours.

(5) After the 2-hour standing, the sample was subjected to the operations of the above steps (1) to (4) so as to measure the surface bleed amount in the second extraction (the second extraction treatment).

(6) After the second 2-hour standing, the sample obtained by the operation of the above step (5) was again subjected

to the operations of the above steps (1) to (4) so as to measure the surface bleed amount in the third extraction (the third extraction treatment).

4. Analytic Conditions for Gas Chromatography

Form:	FID
Air:	50 kPa
Hydrogen gas:	55 kPa
Column:	SE-30 (5%, 1.1 m)
Column temperature:	220°C (INJ, DEC: 250°C)
Carrier gas:	nitrogen gas (50 ml/cm^2)

5. Bleed coefficient

The following equation was used to calculate a bleed coefficient from the surface bleed amounts determined by the steps (5) and (6) of the above item 3:

Bleed coefficient = (W_B/W_A)

wherein W_A is a surface bleed amount found from the second extraction treatment, and W_B is a surface bleed amount found from the third extraction treatment.

6. Surface Amount Measuring Method

The amount of the insect controlling component on the surface of the insect controlling material per 1 kg of the same material can be determined from the surface bleed amount of the sample determined by the operations up to the extraction treatment (3) of the above bleed coefficient determining method 3.

Surface Bleed Amount:

The amount of the insect controlling component (or the amount of permethrin) on the surface of the present insect controlling material (1) was measured according to "the surface amount measuring method" as above. As a result, it was 0.800 g per 1 kg of the present insect controlling material (1).

The bleed coefficient of the present insect controlling material (1) was measured according to "the bleed coefficient determining method". As a result, the surface bleed amount (W_A) found in the second extraction and the surface bleed amount (W_B) found in the third extraction were, respectively:

W_A = 0.620 [g/kg], and

W_B = 0.434 [g/kg],

and the bleed coefficient was 0.70.

7. Denier Evaluation Method

(1) A spun filament wound onto a bobbin was unwound (for relaxation of the winding stress).

(2) The filament unwound by the operation of the above step (1) was cut to obtain 5 or more pieces thereof with lengths of about 1.5 m, which were then left to stand alone at room temperature for 24 hours.

(3) Five pieces of the filament obtained by the operation of the step (2) were correctly cut with lengths of 1.000 m

(having an error of smaller than ± 1 mm, by using a linear scale manufactured by SUS (a first-class product according to JIS)), and the resulting pieces of the filament were correctly weighed to the order of 0.1 mg, with a precision balance.

(4) The denier (g/9,000 m) of each piece of the filament was calculated from the weight per 1 m of the weighed piece of the filament.

Denier = (the weight of the filament per one meter thereof) X 9,000.

(5) The denier of the filament used as the sample was determined from an average value of the deniers of the five pieces of the filament (the average value was obtained by counting fractions over 1/2 as one and disregarding the rest at one place of decimals).

The thickness of the filament obtained in the step 4 of Production Example 1 was measured according to the denier evaluation method. As a result, the thickness thereof was 188 [deniers].

Test Example

A piece with the shape of square (7 X 7 cm), cut out of the present insect controlling material (1), was put in a Petri dish with a diameter of 35 mm, together with 10 insects (Aedes aegypti) as specimens, and was allowed to contact the insects for 3 minutes. After that, the piece

of the present insect controlling material (1) was removed from the Petri dish, and the insects were released into a nylon cage (30 cm in length, 30 cm in width and 30 cm in height) so as to observe the life or death and knocked-down state of the insects with time, to thereby find a fifty per cent lethal time (KT_{50}) and a fifty per cent knocked-down time (KD_{50}), and also to find a percentage of dead insects from the number of the dead insects after 24 hours had passed since the start of the test.

The results are shown below.

KT_{50}:	6.5 minutes
KD_{50}:	2.4 minutes
Percentage of dead insects after 24 hours:	100%

CLAIMS

1. An insect controlling material which is a net-like material having substantially uniform meshes, made of filaments each containing a thermoplastic resin and an insect controlling component which shows a vapor pressure of 1 X 10^{-6} mmHg or lower at 25°C, characterized in that

the content of the insect controlling component in the insect controlling material is from 0.1 to 10% by weight,

the amount of the insect controlling component on the surface of the insect controlling material is from 0.03 to 3 g per 1 kg of the insect controlling material,

the void area of each of the meshes is from 2 to 36 mm^2, and

the bleed coefficient of the insect controlling component found by an acetone cleansing method is from 0.3 to 2.0.

2. The insect controlling material of claim 1, wherein the insect controlling component which shows a vapor pressure of 1 X 10^{-6} mmHg or lower at 25°C is a pyrethroid compound which shows a vapor pressure of 1 X 10^{-6} mmHg or lower at 25°C.

3. The insect controlling material of claim 1, wherein

the insect controlling component which shows a vapor pressure of 1 X 10^{-6} mmHg or lower at 25°C is permethrin.

4. The insect controlling material of any one of claims 1 to 3, wherein the thermoplastic resin is an olefin-based resin.

5. The insect controlling material of any one of claims 1 to 3, wherein the thermoplastic resin is an ethylene-based resin.

6. The insect controlling material of any one of claims 1 to 3, wherein the thermoplastic resin is a resin composition which contains a low density polyethylene and a high density polyethylene.

7. The insect controlling material of any one of claims 1 to 3, wherein the void area of each of the meshes is from 8 to 25 mm^2.

8. The insect controlling material of any one of claims 1 to 3, wherein 0.01 to 10% by weight of a powdery carrier with a particle diameter of 0.01 to 40 μm is contained based on the weight of the insect controlling material.

9. The insect controlling material of any one of claims 1 to 3, wherein the filament is a monofilament with a thickness of 130 to 230 deniers.