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Susceptibility of Forest Insect Pests to Sulfuryl Fluoride

1. Wood Borers and Bark Beetles

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Abstract: All life stages of 7 species of forest insect pests, cryptomeria bark borer, *Semanotus japonicus*, small cedar longicorn beetle, *Callidiellum rufipenne*, Japanese pine sawyer, *Monochamus alternatus*, small pine bark beetle, *Cryphalus fulvus*, larch ips, *Ips cembrae*, thuja bark beetle, *Phloeosinus perlatus*, *Sirahoshizo sp.* were fumigated with sulfuryl fluoride at 5-7 doses for 24 hours (for larval, pupal and adult stages) and 48 hours (for egg stages) at 15 ℃ to evaluate the susceptibility to sulfuryl fluoride. The Probit analysis data showed that egg stages were more resistant than larval, pupal and adult stages in all species and *C. fulvus* egg was the most resistant stage (LD_{50} : 52.0 g/m^3 ; LD_{95} : 86.5 g/m^3) in egg stages of all species, while *Shirahoshizo sp.* larvae were the most resistant stage (LD_{50} : 6.9 g/m^3 ; LD_{95} : 24.7 g/m^3) of all larval, pupal and adult stages. It was estimated that a practical dose of sulfuryl fluoride for attaining 100 % mortality of *C. fulvus* egg, which was the most resistant stage, would be required for as much as 130 g/m^3 or above with considerations of additional doses for gas absorption ratio, gas leakage and quarantine security when the fumigation was conducted for 48 hours at 15 ℃.

Key words: quarantine pests, *Semanotus japonicus*, *Callidiellum rufipenne*, *Monochamus alternatus*, *Cryphalus fulvus*, *Ips cembrae*, *Phloeosinus perlatus*, *Sirahoshizo sp.*, quarantine treatment, fumigation, sulfuryl fluoride, susceptibility

Introduction

Methyl bromide was listed as an ozone depleting substance in the Fourth Meeting of Montreal Protocol in November 1992. It was determined that the domestic production and consumption from 1995 was freezed at 1991 levels with the exception for the plant quarantine and preshipment. It was further requested to make every effort to reduce emission as much as possible in plant quarantine (TATEYA, 1993).

On the other hand, Japan imports more than 70 % of its domestic wood consumtion from foreign countries. Foreign wood is named after the producing areas and is generally called either Asian tropical wood, Siberian wood, American wood or African wood. The amount of the import reached to 22,651,000m^3 in 1994 made up American wood (7,580,000m^3; 33.5 %), Asian tropical wood (7,056,000m^3; 31.2 %), Siberian wood (4,852,000m^3; 21.4 %), African wood (746,000m^3; 3.3 %) and others (2,417,000m^3; 10.7 %). In plant quarantine inspection at the port of entry, forest insect pests of quarantine importance such as *Scolytidae, Cerambycidae, Platypodidae, Curculionidae, Bostrychidae, Lyctidae, Anthribidae, Brentidae* are often intercepted and they are mainly subjected to methyl bromide at timber yard and ship's hold covered with a tent. Approximately 55 % of methyl bromide is used for imported wood in Japanese quarantine (Yokohama Plant Protection Station, MAFF, 1995). It is, therefore, an

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urgent matter for Japanese quarantine to find out a new fumigant.

Sulfuryl fluoride has been used as structural fumigants since the 1950s (STEWART, 1957). This fumigant is easy to apply, nonflammable, and noncorrosive and it also offers high diffusion for rapid penetration and aeration (KENAGA, 1957; MEIKLE & STEWART, 1962). This fumigant was registered in the United States for use against various wood-infesting and household pests (USDA ; 1985 ; Anonymous, 1992, 1993; THOMS & SCHEFFRAHN, 1994).

Our objective was to study the susceptibility of representing 7 species of forest insect pests to sulfuryl fluoride fumigation, and to discuss the potential use for imported wood as a fumigant.

Materials and Methods

Test Insects

Cryptomeria bark borer, *Semanotus japonicus* (LACORDAIRE)

Adults were collected with band traps attached to living Japanese cedars at Nishimurayama-Gun, Ya-magata Prefecture in April 1994. The insect was reared on cedar logs using the method described by MAKIHARA (1991), i.e. a pair of adults was put in each plastic container (15 cm diameter) in which previously barked cedars (5 to 8 cm diameter) was stood on a filter paper. The male adults were removed after one day. The female adults were left in the container following 2 to 4 days for allowing to oviposit between the cedar and the filter paper. After removing the cedar and damaged eggs on the paper, the remaining intact eggs were counted and provided for the test. Larvae were prepared by rearing 6 to 8 of hatched larvae on the cedar (15 cm diameter, 1 m height) for approximately 40 days and then picked out from the cedars to provide for the test.

Small cedar longicorn beetle, *Callidiellum rufipenne* (MOTSUCHULSKY)

Adults were collected from Japanese cedar and Japanese cypress logs in the forest near Kobe, Hyogo Prefecture in April to May 1994. Egg stage was obtained by the same rearing method as *S. japonicus*. Mated females were put on Japanese cedar and Japanese cypress logs (5 to 10 cm diameter) and allowed to oviposit for 40 days to prepare larvae under the bark, and for 3 to 5 months to prepare larvae, pupae and adults in the xylem. These infested cedar and cypress logs were provided for the test.

Japanese pine sawyer, *Monochamus alternatus* HOPE

Mature larvae were obtained from infested red pine logs in Fukuoka Prefecture in February and March 1995. They were put in well ventilated glass bins with pine sawdusts. One larva per bin was prepared for the test to avoid cannibalism.

Small Pine bark beetle, *Cryphalus fulvus* NIIJIMA

Adults were collected from infested red pines in Ishinomaki, Iwate Prefecture in June to August 1994. They were put on new red pine logs (4 cm diameter) and allowed to oviposit. The eggs oviposited under the bark were collected and provided for the test with pieces of bark. The remaining eggs were stored until desired larvae, pupae and adults were obtained and then infested logs were provided for the test.

Larch ips, *Ips cembrae* (HEER)

Adults were collected from infested Japanese larches in Sapporo, Hokkaido in May 1994. They were put on new Japanese larch logs (15 cm diameter) and allowed to oviposit for a week and then stored until desired larvae, pupae and adults were obtained. These stages were picked out from the bark and then provided for the test with pieces of bark.

Thuja bark beetle, *Phloeosinus perlatus* CHAPUIS

Infested Japanese cypress logs (2 to 5 cm diameter) were collected at Kakomo, in Gifu Prefecture in May and August 1994. They were stored until desired larvae and pupae were obtained and then provided for the test.

Sirahoshizo sp.

Infested pine logs (10 to 15 cm diameter) were collected in Aichi Prefecture in June 1995. These logs infesting with larvae and adults were provided for the test.

Fumigation

Fumigation apparatus and gas collecting system are shown in Fig. 1. Fumigation was performed in 30 litre fiber-glass chambers equipped with a gas applicator, an exhauster, ports for a gas collector, a manometer and a temperature prove. Sulfuryl fluoride (purity; 99 % or more) was dispensed from a commercial cylinder through a regulator on which the outlet has been reduced to a teflon tube and gas flows to a collecting line. A desired dose was collected in a syringe by controlling a gas pressure regulating valve by hand and then introduced into the fumigation chamber.

Egg stages were fumigated for 48 hours at 15 ℃ on the basis of data, which Coloptera egg was more resistant stage than other stages to sulfuryl fluoride (KENAGA, 1957; DOTY & WHITNEY, 1967), while larval, pupal and adult stages were fumigated for shorter time of 24 hours at 15 ℃. Fumigation temperature of 15 ℃ was introduced to the test for being applied widely to imported wood fumigation with the

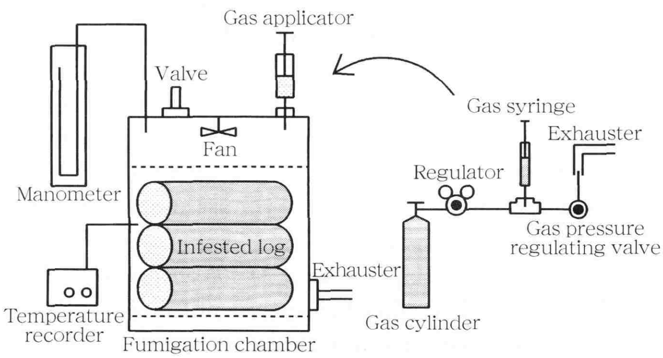

Fig.1. Fumgation apparatus and gas collecting system used for sulfuryl fluoride fumgation.

exception of winter season.

Air circulation fan was kept on for 30 minutes after gas introduction. Gas concentrations during fumigation were monitored with a gas chromatograph (TCD, Shimazu) using the method described by AKAGAWA & SOMA (1995) at time intervals of 1, 24 and 48 hours after commencement of the fumigation. Temperature was also monitored with an automatic temperature recorder (Hybrid recorder AH, Chino).

After fumigation, the air-fumigant mixture was exhausted for one hour by the exhauster.

Evaluation of Mortality

Fumigated insects were removed from the container and stored at 22 to 25 ℃, 70 % R.H. The mortality was evaluated by hatching for 1 to 14 days for eggs, and reaction by stimulating with a small paint blush under microscope at 3 to 5 days after for *C. fulvus*, *I. cembrae*, *P. perlatus* and *Sirahoshizo sp.* larvae, pupae and adults. *M. alternatus* larvae were evaluated by observing their pupation for 10 to 60 days. *C. rufipenne* larvae and adults were observed by barking or chopping the logs at 3 to 11 days after and the pupal stages were evaluated by observing their emergence by chopping the logs at 17 days after.

Data Analysis

Dose-mortality data were analyzed by the Probit procedure using FINNEY's formula (FINNEY, 1971). Linearity regression lines obtained from the statistical analysis were tested by the Chi-square test and fiducial limits were calculated using FIELLER's formula (FINNEY, 1971). The LD_{50}'s and LD_{95}'s were considered to be significant difference when their 95 % fiducial limits did not overlap. The Probit calculation was made by using a computer program (LeOra Software, 1987).

Results and Discussion

Susceptibility to Sulfuryl Fluoride

The results of the Probit analysis for egg stages of *S. japonicus*, *C. rufipenne*, *C. fulvus* and *P. perlatus* are shown in Table 1.

The bark beetle eggs were more resistant than the longicorn beetle eggs. *C. fulvus* egg was the most resistant stage (LD_{50} : 52.0 g/m^3, LD_{95} : 86.5 g/m^3) of all stages tested. The Probit analysis for 0-4 day-old *S. japonicus* eggs and 1-5 and 6-10 day-old *C. rufipenne* eggs were not calculated because of high mortality ratios at relatively low doses used and shortage of appropriate data.

Table 2 shows results of larval, pupal and adult stages of 7 species fumigated at 15 ℃ for 24 hours. *C. fulvus* larvae, pupae and adults, *I. cembrae* larvae, pupae and adults and *Shirahoshizo sp.* adults were not calculated by the Probit analysis because of high mortality ratios at low doses used. *Sirahoshizo sp.* larvae under the bark were the most resistant stage (LD_{50} : 6.9 g/m^3, LD_{95} : 24.7 g/m^3) of all test insects. No significant differences in the susceptibility were observed on *Shirahoshizo sp.*, *C. rufipenne* and *M. alternatus* larvae when evaluated on the basis of the LD_{50}'s. However, different larval conditions of *Sirahoshizo sp.* (larvae under the bark), *C. rufipenne* (larvae in the xylem) and *M. alternatus* (larvae picked out from logs) were used in the susceptibility tests. If *M. alternatus* larvae in the xylem were tested, the larvae may be more resistant stage than other two species of larvae. Further tests, therefore, are necessary with larvae in the xylem because same test conditions were considered with the influence of gas penetration property, which is one of important fumigation elements (MONRO, 1969).

From these data, it could be clearly said that *C. fulvus* eggs were the most resistant stage in all stages of 7 species of forest insect pests to sulfuryl fluoride at 15 ℃.

Gas Sorption by Fumigation Items

Fig. 2 shows changes of progressive gas concentration ratios (gas concentration during fumigation / initial dose x 100) at initial doses of 5, 30 and 60 g/m^3 at 15 ℃ for 48 hours with loading of 10 to 15 %. Approximately 25 % of initial dose of 5 g/m^3, 4 % that of 60 g/m^3 and 10 % that of 30 g/m^3 were absorbed in the 48 hours, respectively. The gas sorption ratio to logs may be slightly affect at 5 g/m^3, and almost not affect at doses of 30 and 60 g/m^3.

Table 1. Estimated LD_{50} and LD_{95} values for egg stages of 4 species of forest insect pests fumigated with sulfuryl fluoride for 48 hours at 15 ℃.

Species & Stages	LD_{50}(95 % FL) (g /m³)	LD_{95}(95 % FL) (g /m³)
*S. japonicus**		
0- 4 day-old egg	—	—
5 - 8	18.7(17.3-21.1)	39.6(36.0- 44.3)
*C. rufipenne**		
1- 5 day-old egg	—	—
6 -10	—	—
*C. fulvus***		
1- 7 day - old egg	52.0(47.6-57.6)	86.5(76.9-102.0)
*P. perlatus***		
1-10 day-old egg	41.6(—)	61.3(—)

* : Eggs oviposited on the paper were used.
**: Eggs with pieces of bark were used.
\- : Probit analysis could not be calculated because of high mortality ratios at low doses and shortage of appropriate data.

Table 2. Estimated LD_{50} and LD_{95} values for larval, pupal and adult stages of 7 species of forest insect pests fumigated with sulfuryl fluoride for 24 hours at 15 ℃.

Species & Stages	LD_{50}(95 % FL) (g /m³)	LD_{95}(95 % FL) (g /m³)
S. japonicus		
larva*	1.7 (1.5- 2.0)	4.5 (3.6 - 6.4)
C. rufipenne		
immature larva under bark	2.0 (1.6- 2.6)	4.2 (3.1 - 9.0)
mature larva in xylem	6.0 (3.3- 8.0)	21.1 (14.7 -53.8)
pupa in xylem	3.3 (1.7- 4.3)	8.4 (6.5 -15.8)
adult in xylem	3.9 (2.7- 5.3)	7.5 (6.0 -12.6)
M. alternatus		
mature larva*	4.3 (2.7- 5.1)	7.7 (7.0 - 9.6)
C. fulvus		
larva under bark	—	—
pupa under bark	—	—
adult under bark	—	—
I. cembrae		
larva*	—	—
pupa*	—	—
adult*	1.1 (1.0- 1.3)	2.2 (2.0 - 2.7)
P. perlatus		
larva under bark	1.2 (1.0- 1.4)	2.9 (2.4 - 4.0)
pupa under bark	—	—
Shirahoshizo sp.		
larva under bark	6.9 (3.9- 8.9)	24.7 (20.3 -37.0)
adult**	—	—

* : Stages picked out from the logs were used.
**: Stages on the logs were used.
\- : Probit analysis could not be calculated because of high mortality ratios at low dose of 5 g /m³ or below and shortage of appropriate data.

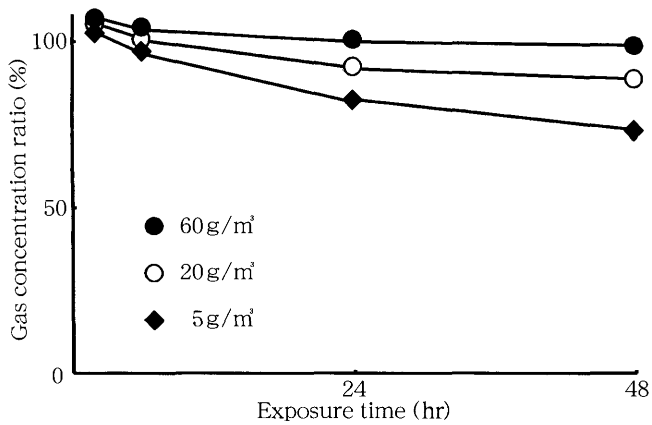

Fig.2. Progressive residual gas concentration ratios (gas concentration during fumgation / initial dose × 100) under fumgation with sulfuryl fluoride at doses of 5, 30 and 60 g/ m³ for 48 hours at 15 ℃ with looding (V/V) of 10 % to 15 %.

Doses for 100 % Mortality

Applied doses for attaining 100 % mortality against larval, pupal and adult stages of *I. cembrae*, *P. perlatus*, *M. alternatus*, *C. rufipenne*, *Shirahoshizo sp.* and *S. japonicus* were killed completely at the range of 5-40 g/m³ for 24 hours at 15 ℃, while the egg stages of *S. japonicus*, *C. fulvus* and *P. perlatus* were not killed completely at 60 g/m³ for 24 hours at 15 ℃. It was estimated that a practical dose of sulfuryl fluoride for attaining 100 % mortality against *C. fulvus* eggs, which was the most resistant stage (LD_{95} : 86.5 g/m³) would be necessary as much as 130 g/m³ or above for 24 hours at 15 ℃ when considered fumigation elements, such as gas absorption ratios to logs, gas leakage from fumigation tents and a guarantee of quarantine security. However, such high doses use would be difficult in the practical quarantine fumigation. Further tests must be conducted with other methods of higher temperatures or mixture gas with other fumigants for obtaining more sufficient efficacy against forest insect pests.

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和文摘要

フッ化スルフリルに対する木材害虫の感受性

1. カミキリムシ及びバークビートル

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1．検疫くん蒸剤として，フッ化スルフリルの導入を検討するため，カミキリ類3種，キクイムシ（バークビートル）類3種，ゾウムシ類1種を用い，15℃，1～60 g/m³ で24時間（幼虫，蛹，成虫）または48時間（卵）くん蒸した。
2．スギカミキリの卵及び若齢幼虫，ヒメスギカミキリの卵，マツノマダラカミキリの老熟幼虫，キイロコキクイムシの卵，カラマツヤツバキクイムシの幼虫，蛹及び成虫，ヒバノキクイムシの卵，シラホシゾウムシ属の成虫は裸虫で，ヒメスギカミキリの若齢幼虫，キイロコキクイムシの幼虫，蛹及び成虫，ヒバノキクイムシの幼虫及び蛹，シラホシゾウムシ属の幼虫は樹皮下に寄生した状態で，ヒメスギカミキリの老熟幼虫，蛹及び成虫は木部内に寄生した状態で供試した。
3．卵は幼虫，蛹及び成虫よりも感受性が低かった。卵の中では，キイロコキクイムシがヒバノキクイムシ，スギカミキリ及びヒメスギカミキリの卵よりも感受性が低く，キイロコキクイムシの卵の LD_{95} 値は48時間くん蒸で86.5 g/m³ であった。
4．スギカミキリの若齢幼虫，ヒメスギカミキリの若齢幼虫，老熟幼虫，蛹及び成虫，マツノマダラカミキリの老熟幼虫，シラホシゾウムシの幼虫及び成虫の中では，ヒメスギカミキリの老熟幼虫及びシラホシゾウムシの幼虫の感受性が低く，24時間くん蒸における LD_{95} 値はヒメスギカミキリが21.1 g/m³，シラホシゾウムシが24.7 g/m³ であった。両害虫は40 g/m³ でいずれも完全殺虫された。
5．バークビートル（キイロコキクイムシ，カラマツヤツバキクイムシ及びヒバノキクイムシ）の幼虫，蛹及び成虫は極めて感受性が高く，24時間，5 g/m³ のくん蒸で完全殺虫された。
6．以上の結果から，15℃のくん蒸温度においては，卵以外の態は5～40 g/m³，24時間のくん蒸で完全殺虫することが可能と考えるが，バークビートルの卵を殺虫するためには48時間で130 g/m³ 以上の薬量が必要であり，フッ化スルフリルの検疫への導入は困難であると考える。

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