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Risk Assessment of the Exposure of Insecticide Operators to Fenvalerate during Treatment in Apple Orchards

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ABSTRACT: Dermal and inhalation exposure of the applicator to the insecticide fenavalerate in an apple orchard was measured for risk assessment during treatment. Emulsifiable concentrate (EC) and wettable powder (WP) formulations were sprayed using a speed sprayer (SS) or power sprayer (PS). Dermal patches, gloves, socks, and masks were used to monitor potential dermal exposure to fenavalerate, while personal air samplers with XAD-2 resins were used to monitor potential inhalation exposure. Validation of analytical methods was performed for the instruments' limit of detection, limit of quantitation, reproducibility, linearity of calibration curve, and recovery of fenvelerate from various exposure matrices. The results were encouraging and reasonable for an exposure study. Applicability of XAD-2 resin was evaluated with a trapping efficiency and breakthrough test. During mixing/loading, the amount of dermal exposure ranged from 262.8 μg (EC/SS) to 1652.6 μg (WP/PS) of fenvalerate, corresponding to ~0.0011−0.0066% of the total prepared quantity. In the case of WP, the amount of dermal exposure was 2032.3 μg (0.0081% of the total applied amount) for SS and 1087.9 μg (0.0145%) for PS after application. In the case of EC, the amount of dermal exposure was 3804.6 μg (0.0152%) for SS and 4055.0 μg (0.0541%) for PS after application. The primary body parts subject to exposure were thigh and upper arm for SS, and thigh and hand for PS. The amount of inhalation exposure with WP was 2.2 μg (8.65×10^{-6}% of the total applied amount) for SS and 1.3 g (1.67×10^{-5}%) for PS. The amount of inhalation exposure with EC was 2.5 μg (9.81×10^{-6}%) for SS and 3.7 μg (4.97×10^{-5}%) for PS. The absorbable quantity of exposure and margin of safety (MOS) were calculated for risk assessment. The MOS for all 4 cases was much greater than 1, indicating a low possibility of risk.

KEYWORDS: *fenvalerate, exposure, apple, risk assessment, sprayer, MOS*

INTRODUCTION

Humans may be exposed to pesticides through their occupation, as well as through environmental contamination, since pesticides are widely used to control insects, crop diseases, and weeds in agricultural fields or homes. In agricultural fields, exposure of the operator to pesticides generally occurs during mixing/loading, spraying, harvesting, and during other postapplication activities. Such direct contact with pesticides by operators who handle and apply these agents can lead to harmful effects on the individual, depending on their toxicities. The most common routes of human exposure are dermal deposition and inhalation, with the greatest potential exposure being through dermal absorption during spray and harvest operations, and their importance has been confirmed by many studies.[1−5] Therefore, field monitoring under practical working conditions is important for the assessment of worker exposure to pesticides. Such surveys provide essential data for risk assessment and are a practical alternative to the extrapolation of human safety data from animal exposure studies.[5−8]

Fenvalerate (Figure 1) is a pyrethroid insecticide, which acts on the nervous system of insects and disturbs the function of neurons by interacting with sodium channels. It has been used for the control of a wide range of insects that are chewing, sucking, and boring kinds infesting various types of crop land, forestry, and noncrop land. Additionally, fenvalerate has been used in the control of flying and crawling insects in public health situations and in animal houses.[9] For example, it has been used regionally for controlling apple leaf miner, orange leaf miner, and many types of moths and aphids in Korea. Mammalian toxicity to fenvalerate is relatively low, showing an acute oral LD_{50} in rats of 451 mg/kg, an acute percutaneous LD_{50} in rabbits of ~100−3200 mg/kg, and an inhalation LC_{50} for rats >101 mg/m^3. The value of the 2-year no observable effects limit (NOEL) for rats is 250 mg/kg.[9]

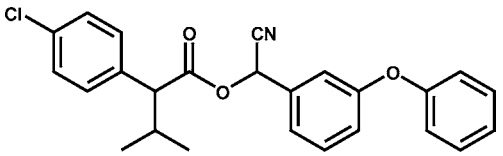

Figure 1. Structure of fenvalerate.

Several recent reports have indicated that pyrethroids are linked to endocrine disruptions, subsequently leading to reproductive dysfunction. Fenvalerate induced a significant decrease in testis weight, epididymal sperm counts, motility of sperms, and testicular enzyme markers for testosterone biosynthesis.[10] Moreover, occupational exposure to fenvalerate may affect the semen quality of workers.[11]

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In Korea, apple is the fruit that is cultivated the most and consumed the most.[12] When farmers treat pesticides in orchards, they typically wear long-sleeved shirts and long trousers instead of the more cumbersome protective garments because of their greater convenience.[13] To the best of our knowledge, no previous studies on exposure and risk assessment of fenvalerate in apple fields have been conducted. The present study was conducted to assess the exposure of operators to fenvalerate when fenvalerate wettable powder (WP) and emulsifiable concentrate (EC) were applied in an apple orchard. A power sprayer (PS) and speed sprayer (SS) were employed for insecticide application. Dermal and inhalation exposure patterns according to the types of formulation and application methods were compared, and the exposed body parts were identified. On the basis of the results obtained, risk assessment was conducted by calculating the margin of safety (MOS) to ascertain the existing state of exposure of operators to fenvalerate.

MATERIALS AND METHODS

Chemicals. Analytical standard of fenvalerate (99.6%) was purchased from Riedel-de-Haën (Seelze, Germany). The fenvalerate formulation was 5% WP and 5% EC (Dongbang Agro, South Korea), available commercially. All of the solvents were of HPLC grade and were purchased from Fisher Scientific Korea Ltd. (South Korea).

Dermal Patches, Gloves, Socks, and Masks. Dermal patches to conduct dermal exposure measurements were constructed by placing cellulose thin-layer chromatography (TLC) paper (1 mm thickness) in the patch pocket (10 × 10 cm) possessing a circular exposure part (50 cm^2). The back of the TLC paper was covered with aluminum foil to prevent contamination of the pocket. Safety pins were used to attach patches on the protective garment (SP protective, KleenGuard; Yuhan-Kimberly, South Korea). Hand exposure was monitored using cotton gloves, and foot exposure was measured using cotton socks. Facial exposure was evaluated using a cotton mask (200 cm^2).[8]

Personal Air Monitor and U-Shaped Glass Tube. Inhalation exposure was measured using a personal air monitor equipped with an air pump (Gillian Model 224-PCXR7, USA), a solid sorbent (ORBO 609 Amberlite XAD-2 400/200 mg, Supelco, USA), and a glass fiber filter (SKC, USA). The U-shaped glass tube for trapping efficiency test was manufactured by Daejung Chemical (Daejon, Korea).

Mixing/Loading and Application of Spray Mixture. The exposure measurement was conducted at an apple orchard at the Apple Research Institute (ARI). Workers prepared the spray mixture by mixing WP (500 g for SS and 150 g for PS) or EC (500 mL for SS and 150 mL for PS) with 500 L (SS) or 150 L (PS) of water in a mixing tank, respectively. The operator sprayed the WP or EC mixture using PS (manual spraying with spray lance) or SS (SS machine without canopy) in an apple field for 20 min for each operation. Application with PS was performed by stepping backward with an up-and-down motion of the lance. Every experiment was repeated 3 times.

Exposure Monitoring. For measuring the amount of dermal exposure during the mixing/loading procedure, two dermal patches (on the forearms) and gloves (on the hands) were used, where most of the exposure was expected. During spraying, 13 dermal patches were placed on the outside of the protective garment, including forehead (1), front of the neck (1), back of the neck (1), chest (1), back (1), upper arms (2), forearms (2), thighs (2), and shins (2). Workers wore cotton gloves, cotton socks, and masks. After the application, exposed samples were removed for analyzing the pesticide content. For measuring inhalation exposure, a glass fiber filter cassette and a XAD-2 resin tube were attached to the breathing zone with a clip, and an air pump was fastened onto the waist belt set at a flow rate of 1.6 L/min. After spraying, the XAD-2 resin and filter were removed and analyzed for pesticide content.

Validation of Analytical Methods. Aliquots (1 μL) of standard solutions from 0.01 to 1 ppm were analyzed to determine the limit of detection (LOD) and limit of quantitation (LOQ). To validate instrumental reproducibility, 3 levels of standard solution (0.5, 5, and 15 ppm) were analyzed 5 times, and the coefficient of variation (C.V.) was calculated. Various standard solutions (LOD ∼15 ppm) were analyzed to construct a calibration curve, and the linearity of the curve was investigated again after 1 and 3 days of storage.

For the recovery test, 3 levels of standard solution (2× LOQ, 10× LOQ, and 50× LOQ) were spiked in patches, gloves, mask, socks, and XAD-2 resin by shaking, prior to the extraction with the appropriate volume of acetone by shaking. For the field recovery test, the standard solution (5× LOQ) was spiked on the same exposure matrices in the field and exposed to the outdoors for a period of time equivalent to the duration of a spray application in order to simulate field study conditions. Recovery and field recovery tests were repeated 3 times.

The trapping efficiency test was conducted by spiking the standard solution (100× LOQ) on the bottom of U-shaped glass tube and by passing air through the system at 1.6 L/min for 4 h. To assist in vaporizing the pesticide, the U-shaped glass tube was heated to 70 °C. The residue in the U-shaped glass tube and the quantity of insecticide trapped in the XAD-2 resin were analyzed, and the mass balance was calculated. The test was repeated 3 times.

A breakthrough test was repeated 3 times by adding the standard solution (10× LOQ) in the first-resin part of the sorbent tube and passing air through the tube at 1.6 L/min for 4 h. The first and second parts of the resin were analyzed separately.

Analysis of Pesticides. For the analysis of fenvalerate in various exposure matrices, an Agilent 6890 gas chromatograph (GC; Santa Clara, CA, USA) with an electron capture detector was used with a DB-1 column (60 m × 0.25 mm, 0.25 μm; J&W Scientific, Folsom, CA, USA). Sample injections (1 μL) were performed in split mode (20:1) at 240 °C. The column temperature was maintained at 220 °C for 1 min, elevated at a rate of 10 °C/min to a final temperature of 300 °C, and maintained at this temperature for 10 min. Helium was pumped as carrier gas at 1 mL/min.

Calculations of Exposure. Dermal exposure amount per body part was calculated by measuring the surface area (cm^2) of the appropriate body region[14,15] and by applying the corresponding dermal exposure intensity (μg/cm^2). The inhalation exposure amount (ng) was determined using the respiration rate (light work, 1740 L/h)[14] and the inhalation exposure rate (ng/h).

Risk Assessment. The potential dermal exposure (PDE) and potential inhalation exposure (PIE) values were obtained by extrapolating the corresponding exposure rate (μg/h) to 4 h per day. The external dermal exposure (EDE) was calculated based on assumptions of 10% cloth penetration for dermal exposure.[16] For internal dermal exposure (IDE), 10% of penetration of EDE through the skin was assumed.[17] For PIE, 100% absorption was assumed.[14] The absorbable quantity of exposure (AQE) value was obtained by summing up the IDE and PIE values. The MOS values were calculated by an adaptation of Severn's formula:[18]

$$\mathrm{MOS} = (\mathrm{NOEL} \times \mathrm{BW})/(\mathrm{AQE} \times \mathrm{SF}) \quad (1)$$

where NOEL is 100 mg/kg/day (dermal) or 3.5 mg/kg/day (oral), average adult male body weight (BW) is 70 kg, and safety factor (SF) is 100.[19]

RESULTS AND DISCUSSION

Fenvalerate is a suspected endocrine disruptor (ED). An apple orchard was selected as the site for the operational part of the study because the quantity and rate of pesticide application is relatively higher there than in other agriculture fields, and both WP and EC were used. Regarding the application methods, Hong et al.[13] reported that the primary application instruments were a Speed Sprayer (SS, 64.9%) and a Power Sprayer (PS, 33.9%) in South Korea.

Method Validation. The LOD (S/N > 3) was set at 0.05 ng, and the LOQ was defined as 4× LOD (0.2 ng). These values were sufficiently low to detect trace levels of fenvalerate

in exposure matrices. The reproducibility of analysis (C.V. < 5%) was good (Table 1), and the linearity of the calibration curves was consistent for 3 days (Table 2), suggesting that the analytical instrument is stable and capable of generating reliable results.

Table 1. Reproducibility of Analysis

pesticide	concn (ppm)	average (area)	C.V. (%)
fenvalerate	0.5	112.4	1.2
	1	232.1	4.2
	5	1136.2	2.9

Table 2. Linearity of the Calibration Curve

	equation of calibration curve and R^2		
pesticide	day of preparation	after 1 day	after 3 day
fenvalerate	$y = 199.04x + 13.05$	$y = 199.58x + 0.34$	$y = 193.29x + 4.66$
	$R^2 = 0.9999$	$R^2 = 0.9974$	$R^2 = 0.9970$

Recovery rates of fenvalerate from various matrices were in the range of ~88.5−117.0% with low C.V. (Table 3), providing

Table 3. Extraction Efficiency of Fenvalerate from Patches, Gloves, Socks, Masks, and XAD-2 Resin

		recovery (%)	C.V. (%)
patch	2 LOQ	103.7	1.8
	10 LOQ	109.3	6.9
	50 LOQ	107.9	4.9
glove	2 LOQ	88.5	6.4
	10 LOQ	99.3	2.1
	50 LOQ	94.9	3.4
sock	2 LOQ	105.6	3.6
	10 LOQ	114.4	2.8
	50 LOQ	103.7	5.5
mask	LOQ	103.1	2.2
	5 LOQ	111.0	2.4
	50 LOQ	104.1	1.7
XAD-2	LOQ	97.2	4.6
	5 LOQ	117.0	2.0
	50 LOQ	113.6	3.2

reasonable extraction efficiencies. Field recovery tests were performed because the apple field was located at a great distance from our laboratory, and pesticides on various exposure matrices may degrade substantially when exposed to sunlight for significant periods or during storage and transport. Field recovery values were over 92.7% with low C.V. (Table 4), demonstrating that any losses of pesticide due to transfer, storage, transit conditions, and exposure to light were not significant.

When measuring the trapping efficiency of XAD-2 resin, good mass balance (Table 5) demonstrated that XAD-2 resin

Table 4. Field Recovery of Fenvalerate

pesticide	matrices	treated level	recovery (%)	C.V. (%)
fenvalerate	patch	5 LOQ	109.5	1.1
	glove	5 LOQ	92.7	5.2
	sock	5 LOQ	115.3	2.1
	mask	5 LOQ	105.3	6.4
	XAD-2	5 LOQ	103.8	3.5

Table 5. Trapping Efficiency of XAD-2 Resin

		recovery (%)			
pesticide	treated level	residue	trapped	total	C.V. (%)
fenvalerate	10 LOQ	10.5	76.3	86.8	3.8

was useful for trapping fenvalerate in air. Fenvalerate was retained at a rate of 96.7% on the first part of the XAD-2 resin in the breakthrough test, indicating that the first resin part possessed a good holding capacity for fenvalerate (Table 6). According to the above validation results, the analytical method proved itself adequate for measuring the exposure of fenvalerate.

Table 6. Breakthrough of XAD-2 Resin

		recovery (%)		
pesticide	treated level	first resin	second resin	C.V. (%)
fenvalerate	10 LOQ	96.7	0	8.1

Dermal Exposure during Mixing/Loading. In the preparation of the spray mixture, the amount of dermal exposure ranged from 262.8 μg (EC/SS) to 1652.6 μg (WP/PS), corresponding to ~0.0011−0.0066% of the total prepared amount (Table 7). As expected, hands were exposed (~55.8−

Table 7. Exposure Amount and Ratios during Mixing/Loading and Application of Fenvalerate in an Apple Orchard[a]

formulation	WP		EC	
spray methods	SS	PS	SS	PS
mixing/applied amount (g)	25	7.5	25	7.5
	Dermal Exposure during Mixing/Loading			
total exposure amount (μg)	751.5	1652.6	262.8	373.4
ratio to prepared amount (%)	0.0030%	0.0066%	0.0011%	0.0015%
	Dermal Exposure during Application			
total exposure amount (μg)	2032.3	1087.9	3804.6	4055.0
ratio to applied amount (%)	0.008	0.015	0.015	0.054
	Inhalation Exposure during Application			
total exposure amount (ng)	2162.4	1252.1	2452.6	3729.3
ratio to applied amount (%)	8.65×10^{-6}	1.67×10^{-5}	9.81×10^{-6}	4.97×10^{-5}
ratio to dermal exposure (%)	0.106	0.115	0.065	0.092

[a]WP, wettable powder; EC, emulsifiable concentrate; SS, speed sprayer; PS, power sprayer.

99.3% of total exposure) to a far greater extent than forearms were (Figure 2). It is well established that hand exposure is higher than that of other parts of the body, especially during the mixing and loading steps.[20] Krieger reported that hands receive 1-to-3 orders of magnitude greater exposure per unit area than other regions of the body.[21] Additionally, hands typically are exposed to several-fold higher dose density than other portions of the body.[22] Therefore, it is extremely important that some form of hand protection be used during insecticide application. Between the 2 formulations examined, WP showed greater exposure than EC due to the operator's direct contact with the

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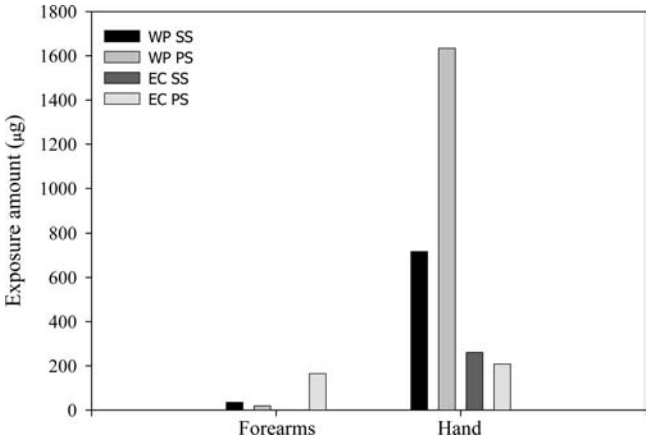

Figure 2. Distribution of dermal exposure amount during mixing/loading.

WP when tearing the pouch and pouring out the WP into the mixing reservoir to generate a suspension. Within the same WP formulation, greater exposure was observed for PS than SS because the mixing reservoir for PS was taller than that of SS, resulting in greater drift or spilling of powder.

Dermal Exposure during Application. The amount of dermal exposure to WP during the application of the pesticide was 2032.3 μg for SS and 1087.9 μg for PS, whereas that of EC was 3804.6 μg for SS and 4055.0 μg for PS (Figure 3). The

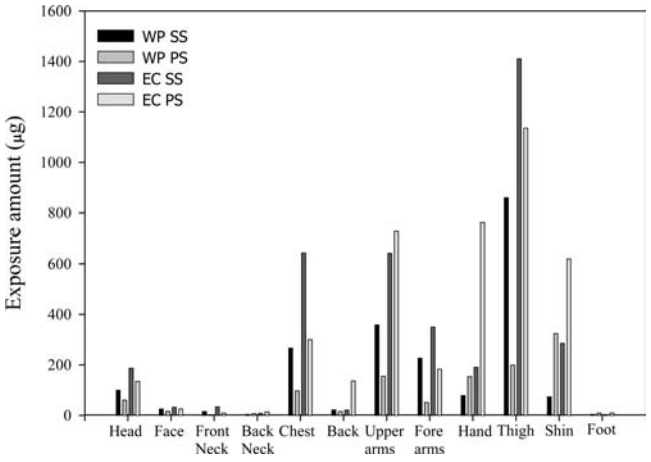

Figure 3. Distribution of dermal exposure amount during application.

exposure ratio to total spraying amount was within the range of ~0.008−0.054% (Table 7), being similar to a prior report (0.01−0.04%) with methomyl.[25] By contrast, a greater value was observed in a mandarin field with cypermethrin, with a ratio of ~0.18−0.34%.[26] When EC and WP were compared using the same spraying technique (SS or PS), the amount of dermal exposure for the EC formulation was ~1.9−3.7 times greater than the WP formulation used in this study, suggesting a difference in the exposure pattern of the 2 different types of formulations. A similar trend was observed for EC versus WP during methomyl and cypermethrin exposure.[25,26] Regardless of the types of formulation, greater exposure resulted from PS than SS due to manual spraying in the case of PS, consistent with results reported in other studies.[25,26]

In this study, every part of the body was exposed to the pesticide, but the primary parts of exposure were the thigh, upper arms, and chest for both SS and PS. Other studies have reported similar results in apple orchards[25,26] and mandarin fields.[26] In addition to these body parts, hands and shins were also primary parts of exposure for PS, while forearms were the primary parts of exposure for SS (Figure 3).

Hands were the primary parts of exposure during application using PS because the operator sprayed the apples by lifting the spray lance overhead in an up-and-down motion, similar to that observed in the methomyl study.[25] In the case of spraying with SS, the primary exposure part was the thigh because the fine spray might be deposited on the thigh as the operator drove the SS machine in a seated position. The head showed significant levels of exposure when considering the relatively small surface area, suggesting that protection of the head is also critically important.

Inhalation Exposure during Spraying. In the present study, inhalation exposure was observed in all cases at low microgram levels (Table 7), and the ratio-to-applied amount values were within the range of ~8.65×10^{-6}−4.97×10^{-5}%, while the ratio-to-dermal exposure was approximately 0.1%. Similar to the dermal case, EC demonstrated greater inhalation exposure than WP, and PS demonstrated ~2−5-fold greater inhalation exposure than SS within the same formulation type because the sprayer with PS spent a greater amount of time spraying the same area than with SS.[25]

Risk Assessment. The PDE values were obtained by extrapolating a dermal exposure of 20 min to 4 h of effective exposure (hours per day) in South Korea.[13]

For mixing/loading, 12 events were used for a one-day exposure calculation since the spraying of one tank (500 L) by SS typically takes about 20 min. The actual dose, AQE, was calculated by summing up IDE and PIE, thereby determining MOS. In the determination of MOS, dermal toxicity value was also used as a toxicological end point, in addition to the lowest value of NOEL,[23] because groups of rabbits who were administered fenvalerate dermally developed symptoms of severe weight loss, clinical poisoning, and gross dermal defects.[23] In this study, the MOS value was found to be higher than 1 (Table 8), indicating that both application methods (SS and PS) are relatively safe.[24] Since MOS > 1, exposure control need (ECN, in %) and safe work time (SWT, in h)[8] were not estimated.

Table 8. IDE, PIE, AQE, and MOS Values When Fenvalerate Treatment Was Applied in an Apple Orchard[a]

formulation	instrument	IDE (mixing/loading) (μg/day)	IDE (application) (μg/day)	PIE (μg/day)	AQE (mg/day)	MOS	MOS
WP	SS	90.18	244.12	25.96	0.36	194	6.8
	PS	198.30	130.66	15.04	0.34	206	7.2
EC	SS	31.54	457.0	29.46	0.52	130	4.7
	PS	44.80	476.88	44.80	0.54	125	4.4

[a]IDE: internal dermal exposure. PIE: potential inhalation exposure. AQE: absorbable quantity of exposure. MOS: margin of safety using dermal toxicity value. MOS: margin of safety using oral NOEL value.

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Notes

The authors declare no competing financial interest.

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