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Residual Content of Synthetic Pyrethroid Transfluthrin on Bed Sheets, Pillowcases, Bedroom Floors, and Development of Analytical Methods Using GC-MS

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Abstrak

Dengue Hemorrhagic Fever (DHF) is an infectious disease with high morbidity and mortality rates worldwide. Two-fifths of the population lives in areas prone to contracting the dengue virus in tropical and subtropical countries. In Indonesia, dengue disease is an endemic problem that spreads in 33 provinces. One way to prevent the transmission of DHF disease is to kill mosquitoes using mosquito repellent spray, electric or mosquito coils. Regular use of household aerosols for mosquito control is widespread, especially during outbreaks of dengue fever, chikungunya, and Zika. In this study, it will be proven whether, in the use of mosquito repellent made from active transfluthrin, the remaining residue attached to indoor items is still within safe limits if exposed to the skin or inhaled by humans. The purpose of this study was to determine the content of transfluthrin mosquito repellent pesticide residues on the surface of objects in the room still below the maximum limit required using the Gas Chromatography-Mass Spectrometry (GC-MS) tool as well as the development of measurement analysis methods for Lienarity, Recovery, Precision/Specivity, Quantitative Limit, and Detection Limit. The study's results on measuring Transfluthrin residues using GCMS with a data collection height of 100-200 cm obtained a concentration range of 22-149 mg/kg. Validation test of the analysis method obtained LOD: 0.005 (µg/ml), LOQ: 0.016 (µg/ml), RSD system conformance test: 0.118%, Linear Regression Linearity (r2): 0.99946, %Recovery: 102.00%, Placebo specificity: does not show a peak at Transfluthrin retention time.

Keywords: *GC-MS, insect repellent, pyrethroid, transfluthrin, validation*

1 Introduction

Dengue Hemorrhagic Fever (DHF) is an infectious disease with high morbidity and mortality rates worldwide. Two-fifths of the population live in areas prone to dengue virus infection in tropical and subtropical countries [1]. DHF is an endemic problem in Indonesia that spreads across 33 provinces [2]. One way to prevent the transmission of DHF is to kill mosquitoes using mosquito spray, electric or mosquito coils. The routine use of household aerosols for mosquito control is very common, especially during outbreaks of dengue, chikungunya, and Zika [3]. From the research results in the Magelang area, 49.35% of households chose spray-type insecticides [4]. The type of insect repellent that is widely used is the pyrethroid group. Pyrethroids are synthetic analogs of pyrethrins developed as pesticides from dry extracts and powder of Chrysanthemum cineraria folium flower [5]. Examples of pyrethroids that are widely used as active ingredients in mosquito repellents are Dimefluthrin, Prallethrin, Transfluthrin, Cypermethrin, and Cyfluthrin.

One example of a pyrethroid insecticide that is widely used as an active ingredient in mosquito repellents is transfluthrin. Transfluthrin household pesticide is a fast-acting insecticide. It is used in household and hygiene products, especially

against flying insects, such as mosquitoes and flies, but also against material pests, such as moths. Transfluthrin can be classified as a synthetic pyrethroid based on its chemical source and an insecticide based on the type of target organism. Based on the results of WHO research in 2002, transfluthrin has low acute toxicity in rats, with an LD50 >5000 mg/kg bb through each route of administration and with an acute and dermal NOEL of 100 mg/kg bw/day. LC50 in 4 hours was >513 mg/m3 of air for male and female rats. The only sign noted during the 14-day observation period was a slight tremor in the female within 5 minutes of dosing. Transfluthrin is not a skin or eye irritant or a skin sensitizer.

Regarding application methods, pyrethroids are mainly used in indoor spraying, outdoor spraying, and using nets and clothing [6]. From the results of studies that have been conducted, the concentration of transfluthrin in the measured air ranged from below the limit of detection to 1.32 ng/L [7]. Based on Minister of Agriculture Decree No. 369 of 2020, the tolerance limits for the results of quality tests for formulations and technical (processed) pesticides. For synthetic pesticides, the maximum content allowed is a maximum of 500 g/kg with a tolerance of ± 25 g/kg or g/L, while for pesticides made from biologically active is required at least 1 x 106 cfu/mL in solution or 1 x 106 Cfu/g in solid according to the Indonesian National Standard (SNI).

2 Method

2.1 Sampling Method

The area of the sampling room is close to 12 m2. Filter paper is prepared and then cut into 10x10 cm sizes and placed on pillows, bed sheets, and the floor at the side of the bed. Then two types of Aerosol mosquito repellent were prepared, type A and Type B containing the active substance transfluthrin. Spray mosquito repellent into the room within 45 cm of the bed. Sample spraying was carried out several times based on the spray position and sampling time, according to Table 1. Residue measurements are taken based on the height of the spray position and the time the sample is taken after the Aerosol is sprayed in the air. Prepare a plastic clip, insert filter paper into the plastic, and then seal it. Then put it in the sample container and close it tightly.

Table 1. Sampling Method

No	Height	Quantity Filter Paper	Position			Sampling Time		
			Pillow	Bed	Floor			
1	200 cm	6 pcs	2 pcs	2 pcs	2 pcs	20 min	30 min	60 min
2	150 cm	6 pcs	2 pcs	2 pcs	2 pcs	20 min	30 min	60 min
3	100 cm	6 pcs	2 pcs	2 pcs	2 pcs	20 min	30 min	60 min

2.2 Sample Preparation

In this study, the reference standard used was Transfluthrin Tagros Chemical India Pvt., ltd, (CAS no 118712-89-3) with a purity of 98.5% and Internal standard Ethyl stearate Kanto chemical (005B2262). Acetonitrile p.a (CAS #: 75-05-8) from Merck was used as a solvent, and a comparison of Ethyl acetate (CAS #: 141-78-6) and Cyclohexane (CAS #: 110-82-7) as a reagent.

Weigh as much as 0.75 grams of filter paper sample cut into a 50 ml test tube. Add 150 µL Internal Ethyl stearate standard from 1000 mg/L Internal standard stock solution. Add 15 mL Acetonitrile. Vortex for 2 minutes, followed by Rotary Extraction for 30 minutes. Centrifuge 7000 rpm for 5 minutes, pipette 500 ul of the filtrate into a 10 mL tube, and evaporate with nitrogen gas in a turbovap at 60ºC for 30 minutes. Then dissolve with 100 µL ethyl acetate: cyclohexane (1:9).

2.3 General Procedures

2.3.1 Gas Chromatography-Mass Spectrometry Analysis

Transfluthrin residue content was measured using Gas Chromatography-Mass Spectrometry (GC-MS). The column used for this study was a capillary column V-5 ms film thickness 0.25 µm x 30 m, ID 25 mm (Agilent), maximum temperature 300° C. Initial temperature 70°C for 2 minutes, then increase in temperature 13°C/ min to a final temperature of 280° C for 1.5 minutes. The injection system is splitless; the column flow rate

is 2.24 ml/min, with the carrier gas being nitrogen (N_2). Compared to previous studies on developing pyrethroid residue analysis methods in fish using GC-MS [8], there are differences in the temperature program used and the carrier gas. In this study, the oven used a setting temperature with an initial temperature of 100°C held for 1 minute, then increased to 150°C with a temperature rise rate of 8.35°C/min, then increased to 280 °C with a rate of 20°C/min held for 5 minutes and finally raised to 300°C with a flow rate of 20°C/min held for 5.48 minutes, a total analysis time of 25 minutes. The carrier gas used is Helium (He).

2.3.2 Validation of Analytical Methods

Performance Validation of analytical methods based on the criteria listed in ISO 17025 Standard of Laboratory Test and Calibration and United State Pharmacopeia (USP) 43 NF 28 (2020). The parameters tested are system suitability tests. In order to confirm the system suitability of an analytical instrument, a parameter test is required based on the robustness studies of the system/instrument. Several parameters must be met in the system suitability test: resolution, reproducibility (RSD), and tailing factor. Linearity At least five standard concentrations are required for analysis. In addition to visually evaluating the analytical signal, statistical calculations are needed, such as calculating linear regression, slope, intercept, and correlation coefficient. To ensure that the tool meets the linearity requirements, the results of calculating the correlation coefficient (r2) must be at least 0.998. Calculation of the quantitation limit (QL) and detection limit (DL) where the detected value is obtained from the standard deviation multiplied by 10 divided by the slope of the calibration curve by n = 5 samples for QL and the standard deviation multiplied by 3 for DL.

Accuracy/spiked recovery is measured as proving the closeness of the value resulting from the calculation results with the value of the reference that has been set. The characteristic of this parameter is an indication of bias or deviation. For a quantitative approach, it takes at least nine pieces of data in a specific range to be determined.

3 Result and Discussion

3.1 Residual Content Test Results

Transfluthrin pyrethroid residues were taken from room objects using filter paper stored in certain positions. In this study, the position of the sample is on the pillowcase, bed sheet, and bedroom floor. This position is determined because there is more frequent contact with humans in that area. Sampling was carried out according to Table 1.

Table 2. Transfluthrin Residues by Position

Position	Height (cm)	Concentration of transfluthrin pyrethroid residue (mg/kg) at different range time			Average Concentration (mg/kg)
		20 (min)	30 (min)	60 (min)	
Pillow case	100	149.5	115.5	100.8	121.9
	150	22.1	28.2	60.8	37.0
	200	36.2	30.9	57.0	41.4
Bed Sheet	100	109.0	103.5	133.9	115.5
	150	55.8	43.6	135.8	78.4
	200	41.4	46.5	74.9	54.2
Floor	100	16.9	103.3	101.4	73.9
	150	96.7	80.0	55.0	77.2
	200	105.7	43.7	89.6	79.7

Table 2 shows that the highest transfluthrin pyrethroid residue content is at the height of 100 cm, and an average sampling time of 20 minutes is 121.9 mg/kg. Overall, the pyrethroid transfluthrin residue on room objects is 22-149 mg/kg. Based on studies of existing indoor aerosol pyrethroid concentrations, airborne pyrethroid content is affected by differences in the number of sprays used, and the duration of sampling after spraying are possible reasons for the variation in pyrethroid concentrations [9]. Specifically for aerosol sprays, the spray time is directly affected by the number of active ingredients emitted into the air and varies from 6 seconds to 20 seconds

[10–12]. Table 3 shows the difference in the results of the concentration of pyrethroids in the air during the application and the sampling time after application.

Table 3. Aerosol pyrethroid concentrations during and after application of pyrethroid anti-mosquito in several studies

Active Ingredient	Content	Volume of Test Room (m^3)	Air Concetretation (During Aplication)	Air Concetration (After Aplication)	Reference
Prallethrin	1.5% (w/w)	26.2	<0.1-13.8 mg m^{-3}		Ramesh and Vijayalakshmi [9]
Transfluthrin	13.4% (w/w)	32.3	4.9-8.5 µg m^{-3}		Vesin et al., 2013 [13]
Allethrin	0.13% (w/w)	26.2	<0.1-80 mg m^{-3}		Ramesh and Vijayalakshmi [9]
	0.15 (w/w)	36.2	170-271 ng m^{-3}	209-74.4 ng m^{-3}	Li et al. [12]
Cyflutrin	0.27% (w/w)	50	10-90 µg m^{-3}		Class and Kintrup [10]
Cypemetrin	0.15% (w/w)	36.2	21.7-36 ng m^{-3}	0.46-0.61 ng m^{-3}	Li et al. [12]
Dertametrin	0.022% (w/w)	26.2	<0.1-5.7 mg m^{-3}		Ramesh and Vijayalakshmi [9]
Tetrametrin	0.9% (w/w)	50	45-300 µg m^{-3}		Class and Kintrup [10]
Transfluthrin	0.3% (w/w)	36.2	16.5-48.3 ng m^{-3}	2.4-9.5 ng m^{-3}	Li et al. [12]

The distribution of pyrethroids in the air in the gas and particle phases is a critical factor in determining their properties and behavior after being released into the air. In this study, it can be seen that at a height of 100 cm, the residual content of pyrethroid transfluthrin has a range of 16-149 mg/kg, while at a height of 150 cm and 200 cm, the residue range is between 22-135 mg/kg. This distribution affects the reach of objects and the potential for human contamination. The farther the pyrethroid is released from an object, the smaller the residual content will be. Other factors that affect the distribution are the condition of the closed or open room and the area of the room. During indoor spraying, the active substance will experience dispersion, partitioning between the gas and particle phases, degradation, and deposition [12].

In general, pyrethroids associated with solid particles are more easily deposited on various surfaces. In contrast, pyrethroids in the form of gaseous compounds have a greater tendency to remain in the air [13,14]. Based on the case study, pyrethroid gas sprayed from an aerosol will produce droplets containing a water-oil emulsion with the active ingredient and propellant as a solvent with a diameter of 1-50 µm [15] and a diameter of 14-46 µm [11].

In this study, the residual concentrations obtained were 37-121 mg/kg. Based on the toxicology test of the transfluthrin pesticide, oral LD_{50} values were > 5000 mg/kg, LC50> 5 [12] mg/m3, and Acceptable Daily Intake (ADI): 0.0075 mg/kg bw [16]. The data is the toxicity value of pure transfluthrin. In contrast, the toxicity standard is based on the Decree of the Minister of Agriculture of the Republic of Indonesia number 399/KPTS/SR.330/M/6/2020, the requirements for acute toxicity in the formulation are LD50 Oral> 200 mg/kg (Liquid), $LC_{50} \geq 0.05$ mg/l during the 4 hour exposure period. The required Maximum Residue Limit (BMR) refers to SNI 7313 of 2008. In the regulations, it is also stated that if it is not available, it can refer to the CODEX Alimentarius Committee. If there is no data from CODEX, then it can refer to ADI. BMR data in SNI 7313:2008 is the maximum residue limit allowed for agricultural commodities. If referring to the tolerance limit for transfluthrin pyrethroid toxicity in the formulation in the Decree of the Minister of Agriculture of the Republic of Indonesia 399/KPTS/SR.330/M/6/2020, the residue in the results of this study is still below the required standard.

3.2 Methods Validation Result

The system suitability test is calculated by injecting analyte samples into the GC-MS system six times. From the calculation results, it is obtained that the %RSD is 0.118%. The standard is that if following the requirements for the number of injections 6 times, the RSD

requirement above 2.0% is still permitted. The results of reading the tool's response also obtained the magnitude of the tailing factor: 1.28. and Resolution: 25.53. The tailing factor, commonly known as the asymmetry factor, indicates an asymmetrical chromatographic peak shape. The tailing factor can also be interpreted as the occurrence of tailing in the chromatogram so that the shape of the chromatogram becomes asymmetrical (Figure 1). In the United State Pharmacopeia (USP) 43 NF 28 (2020) pharmacopeia, it is stated that in one analytical instrument, several equipment components must meet the requirements and achieve the required performance to carry out tests or determine levels. System suitability tests are interpretations of the analytical procedures used to ensure adequate performance of a chromatographic system. Factors affecting the system suitability test include retention factor (mass distribution), repeatability system, signal-to-noise, asymmetry factor, and resolution. There are no acceptance criteria for retention time and relative retention. Relative standard deviation requirements are specified in the individual monographs of the pharmacopeia. If the requirement is 2.0 or less, the calculation is based on data from five replicate injections of the analyte. If the requirement is more than 2.0%, then data from six replicate injections.

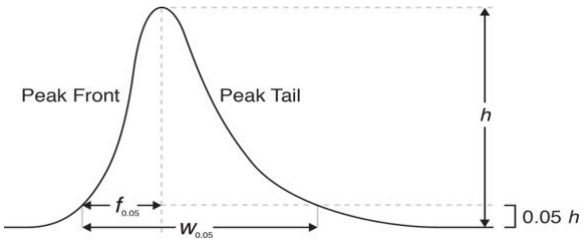

Figure 1. Tailing Factor

Linearity is one way to prove the ability of GC-MS by reading the response to 5 concentration series, which produces a linear calibration curve with a regression coefficient value (r^2) close to 1.0. A series of standards was made with a concentration range of 1.37-2.05 ppm, and a calibration curve was made; then, the straight line equation was calculated to get the value of r^2.

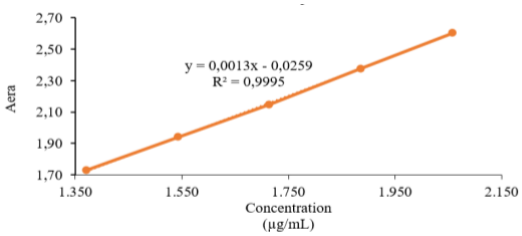

Figure 2. Linearity Curve

From the linear calibration curve, the Slope value (b) is obtained: 0.0013 and the intercept value (a): -0.0259. The value of the correlation coefficient is calculated using Equation 1.

$$r = \frac{\sum(x-\bar{x})(y-\bar{y})}{\sqrt{\sum(x-\bar{x})^2}\sqrt{\sum(y-\bar{y})^2}} \qquad \text{Eq. 1}$$

From Figure 2, the value of r^2 is 0.9995. It shows a good tool response to analytes with various types of concentrations. In previous studies, linearity measurements in the 5-75 ug/kg concentration range obtained r^2 values higher than 0.98 with a standard deviation of ≤±20% of the true concentration [8].

The calculation of DL and QL is taken from the standard calibration curve, then the value of the linear line equation is calculated: y=bx ± a, so that the slope and intercept are obtained, which are calculated using the standard deviation (sd). The calculation results obtained the DL value: 0.005 µg/ml and QL value: 0.016 µg/mL. A small DL value indicates that the tool has high sensitivity and can detect analyte samples at low concentrations.

The accuracy test is the closeness between the test results of an analytical method and the actual value, where accuracy is often expressed as a percentage of the recovery. The accuracy test is carried out to determine the presence of matrix disturbances in the test sample for the reagent or the accuracy of the method used. Three standard concentrations of transfluthrin were made with final concentrations of 1.35, 1.69, and 2.03 µg/ml, then placebo filter paper blanks were added. Three solutions were made for each concentration, then injected into the conditioned GCMS tool. The recovery test was continued by adding a positive sample, carried out by making a standard solution with a concentration of 10 µg/ml 7 times and adding a sample containing transfluthrin residue.

The results of reading the standard recovery test tool transfluthrin with three concentrations with the addition of filter paper blanks obtained an average yield of 102.0%. The repeatability test

was seven times the standard plus a positive sample of 104.5% transfluthrin.

These results still meet the standard recovery specifications of 90% -107% 18. It shows that the measurement accuracy obtained still meets the requirements with the addition of a disturbing matrix. Rawn et al. [17] used the QuEChERS method to analyze pyrethrin I and II, cypermethrin, and deltamethrin in different fish samples with GC. Recovery for sampled salmon ranged from 70 to 115%. Meanwhile, Sapozhnikova et al [19] developed a multi-class, multi-residue analytical method using GC–MS/MS, including bifenthrin, cis and trans-permethrin, and deltamethrin. Recovery values range from 71 to 116%. Chatterjee et al. [18] developed a method for analyzing multiple classes of pesticides in fatty fish using GC–MS/MS. Twelve pyrethroids were determined, and the recoveries for pyrethroids range from 65 to 119% Specificity is carried out to determine the effect of other components in the analyte that can interfere with the measurement results. Specificity was achieved by injecting transfluthrin standard, acetonitrile solvent, Nitrogen gas (N_2) as a carrier, and internal ethyl stearate standard. The results of the tool response showed a chromatogram peak at a retention time of 3.5 minutes, Internal standard of 5.4 minutes, while acetonitrile and N_2 gas did not show a chromatogram peak at a retention time of transfluthrin and ethyl stearate. The graph in the GC-MS library obtained a retention value that is quite far between transfluthrin and Internal standard (IS) as shown in Figure 3 and Figure 4 below.

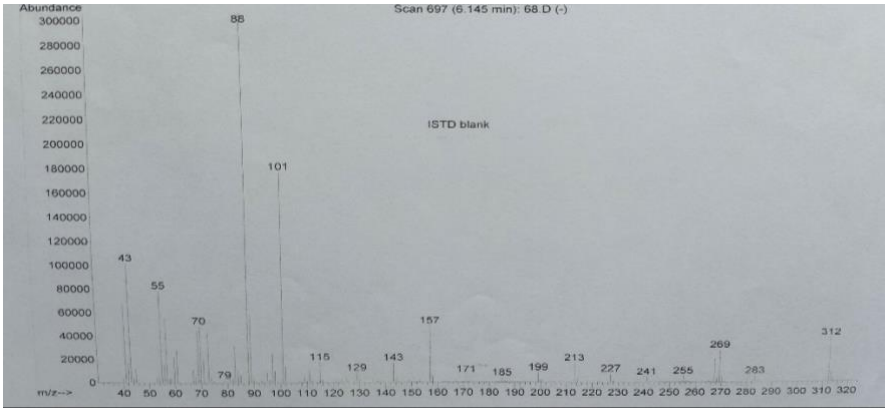

Figure 3. GC-MS Output of Internal Standard

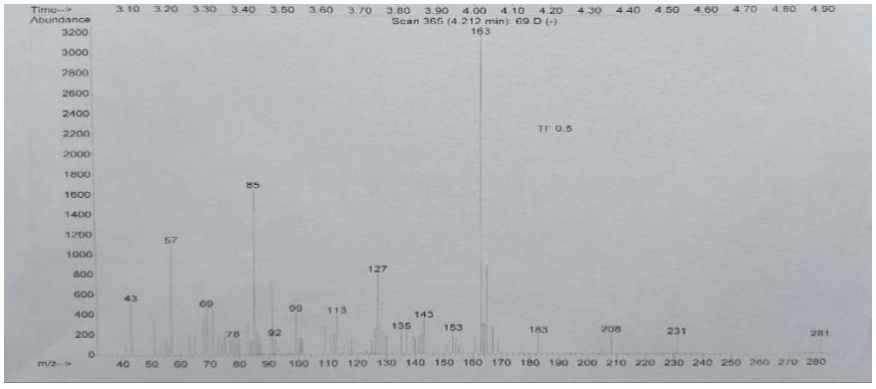

Figure 4. GC-MS Output of Transfluthrin

In the study of pyrethroid residue content in food, according to the European Commission, SANTE/11813/2017, the newly expanded matrix is a pragmatic approach for validating samples from the same commodity group based on sample composition: high water content, high sugar content, high oil content, high, dairy, meat, and seafood, etc. Scope expansion includes two fish species with different matrix compositions: tilapia and tainha. No interference peaks were observed at the same retention time of the analysis when the indigo, tainha, and reagent blank samples were analyzed. Matrix effect studies revealed increased

ion signal for all tainha and tilapia extracts analytes.

4 Conclusion

The results of transfluthrin pyrethroid residues sprayed at a height range of 100-200 cm showed different values. It is caused by aerosol particles dispersed in the air, which will produce a water-oil emulsion that will be exposed to the air for some time, falls to the ground based on droplet size, and be absorbed on filter paper. However, the results of residual measurements showed a value below the toxicity number of LD50 < 500 mg/kg bw. The validation performance of the analytical method showed satisfactory results. The results were below the established standards from the five parameters of specificity, system suitability test, linearity, Recovery, DL, and QL. This value indicates that the test method is robust and that the results can be trusted.

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