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Research article

Pyrethroids residues analysis in Indonesian commercial tea by GC-ECD

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Abstract: Monitoring of pesticides residue including pyrethroids is required since they are major contaminants in tea and have been regulated in European Union and countries such as China, Australia, Japan and America. This study aimed to analyze several pyrethroids in Indonesian commercial tea. The analysis was done by applying low volume solid-liquid extraction followed by quantification by gas chromatography-electron capture detector as a relatively cheaper alternative instrument in pesticides monitoring compared to mass spectrophotometer. The detection limits of the method were 0.006 mg/kg for deltamethrin, 0.05 mg/kg for fenvalerate, 0.10 mg/kg for cypermethrin and 0.05 mg/kg for λ-cyhalothrin. The linearity of the method was good with coefficient correlation of higher than 0.997 at 0.3 to 2.0 mg/kg. The repeatability of the method was also good with lower % relative standard deviations compared to two-thirds of Horwitz coefficient of variation and European Commission guideline. The recoveries were all in the range suggested by European Commission. Weak matrix effect was observed for deltamethrin and cypermethrin while matrix suppression effect was observed for λ-cyhalothrin and fenvalerate. Analysis of tea samples planted and produced in Indonesia showed that the pyrethroid residue in the samples were all below the maximum residue limits while the risk assessment suggested that Indonesian commercial tea is safe for consumption.

Keywords: green analytical chemistry; Indonesian tea; low volume liquid extraction; pyrethroids; risk assessment

1. Introduction

Tea, produced from the buds and leaves of *Camelia sinensis*, is the most consumed beverage in the world. It was believed as a health drink since long ago and research has proven its benefits to human [1–3]. Food and Agriculture Organization of the United Nations [4] reported that the world

tea consumption was increasing and in 2013 the number has reached almost 5 million tonnes. From this number, around 78% was contributed from black tea while 20% from green tea [2].

However, tea may pose a risk associated with the contaminants present in the tea itself. Pesticide residues, including pyrethroids, are one of the major class contaminants [5], apart from mycotoxins [5], heavy metals [5], and polycyclic aromatic hydrocarbons [6].

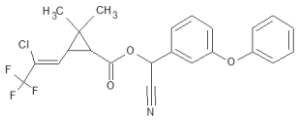
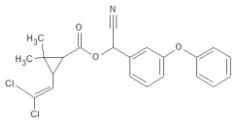
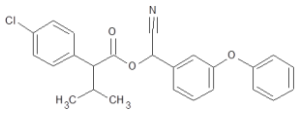
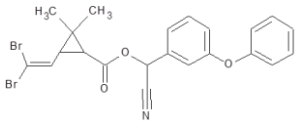
Pyrethroids had been known as relatively safe pesticides. They were used to replace the more toxic class of pesticide such as organochlorins. International Agency for Research on Cancer (IARC), United State of Environmental Protection Agency (USEPA), and World Health Organization (WHO) have not listed them as carcinogenic agents (Table 1). However, recently studies show the dangerous effects of pyrethroids. Pyrethroids were proven to be very toxic to fish and invertebrate showing with low values of LC_{50} [7]. In addition, deltamethrin exhibited neurotoxicity and hepatotoxicity in mammals [8–10] while fenvalerate was suggested as human sperm genotoxic agent [11].

Pyrethroids residues in tea were sourced from the application of pesticides during tea plantation and tea processing including cultivation, storage, and tea production [12,13]. The short time interval in between pesticides application and tea harvest also contributed to the pyrethroids residues in tea. Furthermore, the pesticides were applied during the storage time directly to the tea leaves to prevent pests and longer the storage therefore increases the pesticides residue level in tea. The maximum residue limits (MRLs) for pyrethroids in several countries are given in Table 1. Pyrethroids contamination in tea has been reported previously [6,14,] but to our knowledge, there is no report on tea sources from Indonesia. Previously, pyrethroids analysis involved liquid-liquid or solid-liquid extraction which used a big volume of samples and solvents. Further sample preparation was developed to lower the volume of samples and solvent and utilized some combinations of several extraction techniques such as vortex, ultrasonication, centrifugation, and rotary evaporation [15–17]. Some of the sample preparation utilized dichloromethane as a solvent during extraction [16], a solvent which has listed in Group 2A (probably carcinogenic to human) in IARC classification [18]. Sample preparation by QuEChERS has been known in pyrethroids analysis tea [17,19–21]. Moreover, advances in nanoparticles has successfully utilized magnetic nanoparticles for pyrethroid determination in some food matrices [22–24] in combination with high performance liquid chromatography (HPLC)—photodiode array detector. These methods result low LODs and are relatively simple. However, the drawback of these methods is the utilization of magnetic nanoparticles that are needed to be developed.

Like other simultaneous pesticide residue analysis, gas chromatography (GC) or liquid chromatography (LC) are usually coupled with mass spectrophotometer (MS) or tandem mass spectrophotometer (MS/MS) for low level detection. However, for moderately equipped laboratories, MS detector is not usually available due to its relatively high cost and maintenance. Therefore, alternative detectors with relatively lower cost compare to MS, such as ECD, are needed to be utilized.

This paper was aimed to analyze pyrethroids residue, namely λ-cyhalothrin, cypermethrin, fenvalerate, and deltamethrin (properties in Table 1), in Indonesian tea samples. The analysis applied green analytical chemistry where low volume solid-liquid extraction was utilized to minimize chemical use and dispersive clean-up was applied to remove interferences. The quantification of the pyrethroids was then performed on GC-ECD.

Table 1. Carcinogenicity classification, maximum residue limits (MRLs) in tea, and properties of pyrethroids.

Pyrethroids	Carcinogenicity and MRL				Chemical and physical properties	
	Carcinogenicity		MRL (mg/kg)			
λ-cyhalothrin	IARC : NA	Codex	:	NA	Formula	$C_{23}H_{19}ClF_3NO_3$
		EU	:	1†	Octanol/water	log K_{ow} = 6.80
	USEPA : D	China	:	15‡	Coefficient	
		Australia	:	NA	Structure	
	WHO : II	Japan	:	20†		
		Indonesia	:	NA		
Cypermethrin	IARC : NA	Codex	:	10†	Formula	$C_{22}H_{19}Cl_2NO_3$
		EU	:	NA	Octanol/water	log K_{ow} = 6.60
	USEPA : C	China	:	NA	Coefficient	
		Australia	:	NA	Structure	
	WHO : II	Japan	:	20†		
		Indonesia	:	NA		
Fenvalerate	IARC : 3	Codex	:	NA	Formula	$C_{25}H_{22}ClNO_3$
		EU	:	NA	Octanol/water	log K_{ow} = 6.20
	USEPA : E	China	:	0.1‡	Coefficient	
		Australia	:	NA	Structure	
	WHO : II	Japan	:	NA		
		Indonesia	:	NA		
Deltamethrin	IARC : 3	Codex	:	5†	Formula	$C_{22}H_{19}Br_2NO_3$
		EU	:	5⁑	Octanol/water	log K_{ow} = 6.20
	USEPA : *	China	:	10‡	Coefficient	
		Australia	:	5†	Structure	
	WHO : II	Japan	:	NA		
		Indonesia	:	10⁂		

*Not likely to be carcinogenic; †FAO [25]; ‡FAO [26]; ⁑EU [27]; ⁂Komisi Pestisida [28]; Chemical and physical properties are taken from HSDB [29–32].

2. Materials and methods

2.1. Chemicals and reagents

All pesticides used in this study were sourced from Chem Service, West Chester, USA. The purity of the standard based on the certificate of analysis are: λ-cyhalothrin (99.5%); cypermethrin (99.2%); fenvalerate (99.5%); deltamethrin (99.3%). Stock solutions were prepared individually for each pesticide by weighing and dissolving the pesticide in n-hexane at 100 mg/L. A mix stock solution was prepared from the individual stock solutions at 5 ppm for each pyrethroid and the standard mix solutions were prepared by diluting the mix stock solution at different concentrations ranging from 1 to 1000 ppb. Primary secondary ammine (PSA) and graphitized carbon black (GCB) were sourced from Agilent, Folsom, USA. Florisil was sourced from Merck, Darmstadt, Germany, and activated at 600 ℃ before use. Other chemicals and solvents used in this study were all sourced

from Merck unless otherwise stated.

Tea samples used in this study were commercial tea that purchased from local supermarkets in the package of tea bags or loose tea. The samples are accepted as indicated in their labels. All tea samples are planted and produced in Indonesia. More details of the tea samples are given on Table 3.

2.2. Apparatus

The GC-ECD analysis was carried out in an Agilent 7890B GC coupled with a micro ECD from Agilent. The separation was performed in a HP-5 Agilent column (30 m × 0.320 mm × 0.25 μm). The separation was optimized in these conditions [33]: carrier gas flow (He) was 2 mL/min; make-up gas (N_2) was 30 mL/min; injection mode: automatic liquid autosampler (1 μL) at a ratio of 1:1; injector temperature was 250 ℃; detector temperature was 350 ℃; oven program: start at 200 ℃ (1 minute hold); ramping up 20 ℃/min to 280 ℃ (8 minutes hold).

2.3. Analytical procedures and analysis of tea samples

A spike-experiment was conducted to analyze the tea samples and the result was compared to the control (no-spike-experiment). In the spike-experiment, a quantity of target pesticides was spiked to the tea samples before the extraction. The spiked tea was then vortexed and sit for 1 hour to let the target pesticides interact with the samples.

For the extraction, a low volume of solid-liquid extraction was proposed. A 15 mL of n-hexane was used for each 1.5 g tea and the extraction was performed for 10 minutes on a rotary agitator followed by 5 minutes centrifugation at 4000 rpm. The n-hexane phase was then filtered by a 0.45 μm syringe filter and the filtrate then underwent clean-up. In the clean-up, a mixture of 20 mg of PSA and 20 mg of GCB was applied to 1 mL of the n-hexane aliquot. The mixture was then vortexed and centrifugated for 2 minutes at 4000 rpm. The n-hexane phase was then filtered by a 0.45 μm syringe filter and the filtrate was injected to GC-ECD.

3. Results and discussion

3.1. Method evaluation

In our preliminary work [33], n-hexane was able to extract pyrethroids in the infusion tea that has been spiked with targeted pyrethroids at concentration of 0.67 μg/L (proportional to 0.02 mg/kg in tea). In this study, ethyl acetate, n-hexane and 20% acetone in n-hexane were used as solvents for the extraction. However, the difference was relatively not significant and n-hexane was chosen in preference for the extraction. PSA, GCB, and florisil were used as dispersive adsorbent for the clean-up step and the result showed that the sample treated by the mixture of PSA and GCD resulted a relative smoother baseline thus PSA and GCB were then applied during the clean-up for the analysis. The chromatogram of the spiked sample is given in Figure 1 while the evaluation of the proposed method is given in Table 2.

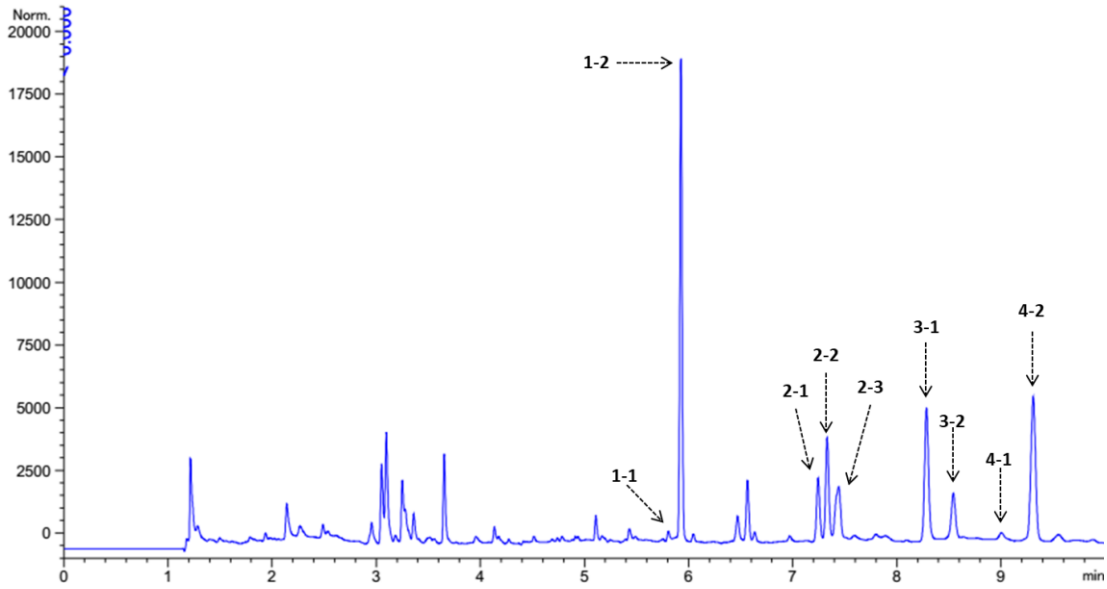


Figure 1. Chromatogram of spiked sample. Peak 1-1 and 1-2 for λ-cyhalothrin, 2-1, 2-2 and 2-3 for cypermethrin, 3-1 and 3-2 for fenvalerate, and 4-1 and 4-2 for deltamethrin.

Table 2. Evaluation of the method.

Pyrethroids	Linear equation (R) Standard solutions	Method	Matrix effect (%)	RSD* ($\frac{2}{3}\mathrm{CV}_{Horwitz}$)	LOD (mg/kg)	LOQ (mg/kg)
λ-cyhalothrin	y = 180.8x − 721.8 (0.9998)	y = 13325.8x + 316.5 (0.9996)	−26.29	3.98 (15.76)	0.05	0.16
Cypermethrin	y = 103.8x + 236.3 (0.9997)	y = 8955.2x − 151.8 (0.9975)	−13.71	6.08 (15.40)	0.1	0.32
Fenvalerate	y = 96.1x − 332.3 (0.9998)	y = 6393.1x + 1154.9 (0.9975)	−33.46	13.85 (18.00)	0.05	0.16
Deltamethrin	y = 97.7x − 400.9 (0.9998)	y = 8198.5x − 259.9 (0.9992)	−16.06	2.45 (15.67)	0.006	0.02

*Measured at 0.5 mg/kg.

Linearity of the calibration curve was established in the range of 1 to 1000 μg/L with very good correlation coefficient (r > 0.999) as showed in Figure 2. Similarly, good linearity (r > 0,997) was also observed for the proposed method for spike concentration of 0.3 to 2 mg/kg range as given in Figure 2. This implies that the responses (area) given by the spike-samples that extracted and cleaned-up by the proposed method are linear to the spike concentration (0.3–2 mg/kg).

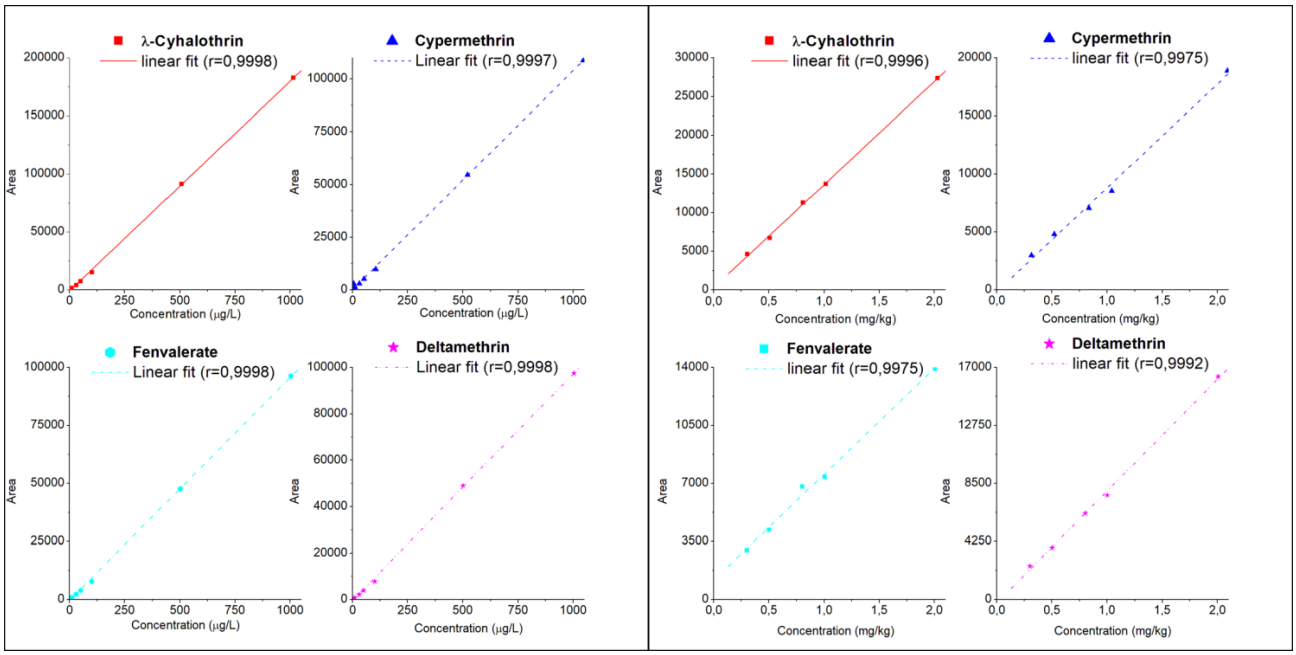


Figure 2. Linearity of standard solutions at range of 1 to 1000 µg/L (left box) and the method at 0.3 to 2 ppm (right box).

The limit of detection (LOD) and limit of quantification (LOQ) were estimated from the signal-to-noise ratio of the blank samples, 3 times and 10 times respectively for LOD and LOQ. The LOD values are different for each pyrethroid and reach as low as 0.01 mg/kg for deltamethrin to 0.1 mg/kg for cypermethrin. The LOQ of the method lies from 0.02 mg/kg for deltamethrin to 0.32 mg/kg for cypermethrin. The values of LOD and LOQ for other pyrethroids are given in Table 2. As comparison, method analysis of deltamethrin in tea (quantification by GC-ECD) as an individual analyte but with a higher volume of solvent and extracted with a relative more complicated method gave a LOD and LOQ of 0.015 and 0.05 mg/kg respectively [14] which is higher than in this study. However, compare to other methods which utilize GC-MS or GC-MS/MS, the LOD and LOQ obtained in this method was higher as predicted.

The precision of the proposed method was evaluated by spiking 5 tea samples at 0.5 mg/kg and extracted by the proposed method. The percent relative standard deviation (RSD%) values obtained in this study are all below the two third of Horwitz coefficient variation ($CV_{Horwitz}$) and the guideline of European Commision [34]. This implies that the repeatability of the method is acceptable.

Meanwhile the recoveries of the spike-experiment at fortified level of 0.3 to 2 mg/kg levels (72.55 to 102.97%) were in the range suggested by European Commission [34] which is 70 to 110%. Therefore the proposed method can be applied to analyse pyrethroids residue in tea.

The matrix effect was also investigated in this study. The matrix effect (ME in %) was calculated as $\frac{(\text{slope of calibration curve in method})-(\text{slope of calibration curve in standard solution})}{\text{slope of calibration curve in standard solution}} \times 100$ [21]. The slopes of linear equations for standard solutions in Table 2, which were derived from Figure 2, were used. Meanwhile for the method, the spike concentrations were changed to the equivalent µg/L units and new curves were built to obtain the slope of linear equation for the method. Weak or soft matrix effect (< ± 20%) was observed for deltamethrin and cypermethrin. Meanwhile, medium matrix

effect (> ± 20% and < ± 50%) was observed for λ-cyhalothrin and fenvalerate. Variation in matrix effect is normal for analysis in tea matrices [21,35] and may due to the interaction of the functional groups of pesticides and the GC instrument such as liner and glass wools [36]. However, to our knowledge, there is no regulation on permitted value of % ME. Therefore, the % ME values in this study is only an information on how the matrix affect the measurement.

To sum up, this method is able to measure pyrethroids in tea with LODs and LOQs that are lower than the MRLs. In addition, the linearity, repeatability, and recovery of this method are acceptable thus this method can be applied to measure pyrethroids in commercial tea. However, this method cannot achieve LODs or LOQs as lower as in LC or GC that is combined with MS or tandem MS [21,37–39] or when nanoparticles are used as adsorbent [22–24]. Nevertheless, the LOD or LOQ values of this method is still comparable to other method that utilized GC-ECD but with more complicated sample preparation steps [14,40]. Moreover, to obtain lower LOD or LOQ values, further investigation can be approached for pyrethroid analysis using GC-ECD where nanoparticles are utilized as absorbent [22–24].

3.2. Analysis of tea samples

The validated method was then used to analyse commercial tea products planted and produced in Indonesia. The result of the analysis is given in Table 3.

Table 3 shows the concentrations of pyrethroids in the tea samples are all below the lowest MRL values therefore safe for consumption. λ-cyhalothrin was not detected (<LOD) in all tea samples. Cypermethrin, fenvalerate, and deltamethrin were all detected in tea samples as high as 0.67; 0.078; and 0.035 mg/kg respectively indicating that these pyrethroids were used during tea plantation and or storage and persisted during tea production. In addition, cypermethrin, fenvalerate, and deltamethrin are generally found in samples from area A to E. For undetected cases of cypermethrin in area D and those three pyrethroids in area F are possibly due to limited number of samples from that area of plantation.

To assess the risk of consuming tea with residue concentrations found in this study, a risk assessment was conducted. The risk was assessed by comparing the theoretical maximum residue contributions (TMRC) and maximum permissible intake (MPI). The TMRC for each pyrethroid was calculated based on the highest concentrations detected in this study, except for λ-cyhalothrin which was based on the LOQ of this study. The MPI was calculated for an adult with a body weight of 60 kg. The TMRC and MPI comparison (Table 4) shows that TMRC of the four pyrethroids are all below the MPI. This suggests that consuming tea with the amount of pyrethroids detected in this study every day for the whole life is unlikely to cause health concerns.

Table 3. Concentration of pyrethroids in Indonesian tea samples.

No	Sample Type	Planted Area	Concentration of pyrethroids in mg/kg (standard deviation in mg/kg)			
			λ-Cyhalothrin	Cypermethrin	Fenvalerate	Deltamethrin
1	Black tea, tea bag	Area A	<LOD	<LOD	<LOD	<LOD
2	Black tea with vanilla flavor, tea bag	Area A	<LOD	<LOD	<LOD	<LOD
3	Black tea, tea bag	Area B	<LOD	<LOD	0.0505 (0.0043)	<LOD
4	Black tea with vanilla flavor, tea bag	Area C	<LOD	<LOD	0.0587 (0.0070)	0.0232 (0.0014)
5	Black tea, tea bag	Area D	<LOD	<LOD	0.0519 (0.0083)	0.0114 (0.0011)
6	Jasmine green tea, tea bag	Area E	<LOD	0.3223 (0.0044)	0.0714 (0.0260)	0.0354 (0.0033)
7	Jasmine green tea, tea bag	Area B	<LOD	0.6746 (0.0330)	0.0787 (0.0158)	<LOD
8	Jasmine green tea, tea bag	Area A	<LOD	0.2591 (0.0149)	0.0539 (0.0102)	0.0088 (0.0001)
9	Jasmine tea, tea bag	Area C	<LOD	0.3791 (0.0555)	<LOD	<LOD
10	Jasmine green tea, loose tea	Area B	<LOD	0.2625 (0.0920)	<LOD	<LOD
11	Green tea, tea bag	Area B	<LOD	<LOD	<LOD	0.0102 (0.0013)
12	Jasmine green tea, loose tea	Area A	<LOD	<LOD	<LOD	0.0115 (0.0003)
13	Jasmine green tea, loose tea	Area A	<LOD	0.2680 (0.1386)	<LOD	<LOD
14	Green tea, loose tea	Area B	<LOD	0.5192 (0.0220)	<LOD	<LOD
15	Jasmine green tea, loose tea	Area F	<LOD	<LOD	<LOD	<LOD
16	Jasmine green tea, loose tea	Area A	<LOD	<LOD	0.0640 (0.0257)	<LOD
17	Jasmine green tea, loose tea	Area E	<LOD	0.205 (0.0216)	<LOD	<LOD

Table 4. Risk assessment of pyrethroids in Indonesian commercial tea.

Pyrethroid	λ-Cyhalothrin	Cypermethrin	Fenvalerate	Deltamethrin
Maximum residue level (mg/kg)*	0.16	0.67	0.078	0.035
Average daily consumption of tea (g)**	1.0411	1.0411	1.0411	1.0411
Theoretical maximum residue contribution (TMRC ; mg per person per day)	1.67×10^{-4}	6.98×10^{-4}	8.12×10^{-5}	3.64×10^{-5}
Acceptable daily intake (ADI, mg/kg)***	0.02	0.05	0.02	0.01
Body weight (kg)	60	60	60	60
Maximum permissible intake (MPI, mg)	1.2	3.0	1.2	0.6
Note	TMRC < MPI	TMRC < MPI	TMRC < MPI	TMRC < MPI

*The highest concentration (or LOQ) of pyrethroids in samples; **for Indonesia [41]; ***based on HSDB [29–32]; TMRC = maximum residue level × average daily consumption of tea; MPI = ADI × body weight.

4. Conclusion

The low volume solid-liquid extraction followed by clean-up with PSA and GCG and quantification by GC-ECD was validated to analyse pyrethroids in tea samples. The LODs and LOQs are all below the MRLs, while the linearity, repeatability, and recoveries are in acceptable values. The concentration of pyrethroids of tea samples planted and produced in Indonesia were all below the MRLs. The conducted risk assessment showed that all tea samples are safe for consumption and unlikely to cause health concerns even for daily consumption for the whole life.

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Conflict of interest

There is no conflict of interest in this article.

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