Pesticide residue monitoring and risk assessment in the herbal fruits *Schisandra chinensis*, *Lycium chinense*, and *Cornus officinalis* in Korea

Junheon Kim¹ · Jihye Shin² · Chung Gyoo Park³ · Sang-Hyun Lee¹

Received: 24 March 2020 / Revised: 13 September 2020 / Accepted: 14 October 2020 / Published online: 4 November 2020
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Abstract *Schisandra chinensis*, *Lycium chinense*, and *Cornus officinalis* are cultivated in South Korea, China and Japan. Because of their beneficial biological effectiveness, the consumption of these herbs is increasing. The objective of this study was to analyze the pesticide residue levels on these herbal fruits produced in Korea. A short- and long-term risk assessment of the pesticides in herbal fruits was conducted. Pesticides were detected in most samples, and all samples with multiple residues were also noted. The average numbers of pesticides detected in the *S. chinensis*, *L. chinense*, and *C. officinalis* samples were 1.84, 10.28, and 5.87, respectively. The short-term and highest long-term risks, and the cumulative risk (cHI) were 0.633%, 1.14%, and 7.56% (*S. chinensis*), $1.0 \times 10^{-1}\%$, $1.1 \times 10^{-5}\%$, and $4.0 \times 10^{-5}\%$ (*L. chinense*), and

$2.2 \times 10^{-5}\%$, $1.6 \times 10^{-5}\%$, and $8.9 \times 10^{-5}\%$ (*C. officinalis*). No significant health risk for consumers via herbal fruit consumption was detected.

Keywords *Schisandra chinensis* · *Lycium chinense* · *Cornus officinalis* · Pesticide residue · Risk assessment

Introduction

The increased use of pesticides for crop protection is prospectively based on the increasing world population and the need for a larger food supply. In spite of their protection of plants, many pesticides are toxic and pose many side effects, including environmental and human health problems, if they are not used properly. Currently, pesticide residues in agricultural commodities are monitored worldwide (Akoto et al., 2013; Diop et al., 2016; Golge et al., 2018; Kumari and John, 2019; Park et al., 2014) to protect consumer health, promote the management of agricultural resources and reduce economic losses.

Most countries use maximum residue limits (MRLs) to regulate pesticides. MRLs are defined as the upper legal concentration of pesticide residues (mg/kg or ppm) in or on food or feed based on good agricultural practices (GAP) and are used to ensure the lowest possible consumer exposure (EFSA, 2011). In Korea, to reinforce the safety management of agricultural commodities, a positive list system (PLS) was introduced in 2016 and has been applied to all agricultural commodities since 2019 (MFDS, 2003). The PLS defines the specific established MRL for registered pesticides; however, for unregistered pesticides, a MRL of 0.01 ppm is applied (MFDS, 2019a).

The fruits of magnolia berry (*Schisandra chinensis*), goji berry (*Lycium chinense*), and Japanese cornelian cherry

Electronic supplementary material The online version of this article (<https://doi.org/10.1007/s10068-020-00840-5>) contains supplementary material, which is available to authorized users.

✉ Junheon Kim
junheonkim@korea.kr

Jihye Shin
ssjh@jj.ac.kr

Chung Gyoo Park
parkcg@gnu.ac.kr

Sang-Hyun Lee
shlee4@korea.kr

¹ Forest Insect Pests and Diseases Division, National Institute of Forest Research, Seoul 02455, Republic of Korea

² Research Center for Agro-Bio-EM & Environmental Resources, Jeonju University, Jeonju 55069, Republic of Korea

³ Institute of Life Science (BK21+ Program), Gyeongsang National University, Jinju 52828, Republic of Korea

(*Cornus officinalis*) have been used in traditional herbal medicine for centuries in Korea and China (Atsumi et al., 2019; Ju et al., 2015; MFDS, 2014). The herbal fruits of *S. chinensis* and *L. chinense* are known to have biological functions, such as longevity promoting, immunity enhancing, antiaging, antitumor, and antioxidant activities (Chan, 2011; Guo et al., 2008; Nzeuwa et al., 2019; Panossian and Wikman, 2008; Skenderidis et al., 2019). As medicinal herbs, *S. chinensis*, *L. chinense*, and *C. officinalis* are import forest products. Moreover, the income from *S. chinensis* represented 43.9% of the total income from medicinal herbs from forests (KFS, 2019). The wide range of beneficial biological effects attributed to these plants has contributed to an increase in their popularity, and thus, the demand for these foods has increased enough for them to be commercialized as beverages.

Although the safety management of agricultural commodities was reinforced by PLS, the pesticide residue-related safety of these herbal fruits has not been investigated since 2012 (Yu et al., 2012). Therefore, the present study was undertaken to monitor pesticide residues in these three herbal fruits grown in Korea to determine the impact of patterns of use of these chemicals on the observed contamination. An attempt was also made to predict the health risk associated with the presence of these pesticide residues. This information also supports the legal use of agrochemicals and the establishment of MRLs to reduce or prevent human health risks due to harmful pesticide residues in primary and derived agricultural products.

Materials and methods

Chemicals and reagents

Pesticide standards were purchased from AccuStandard, Inc. (New Haven, CT, USA) and had a purity > 95%. Individual stock solutions (1000 µg/mL) of each pesticide standard were prepared in acetonitrile (MeCN) or acetone, etc. Quick easy cheap effective rugged and safe (QuEChERS) kits were obtained from BEKOlut (Hauptstuhl, Germany). Extraction kits contained 4 g of anhydrous magnesium sulfate, 1 g of sodium chloride, 1 g of sodium citrate tribasic dihydrate, and 0.5 g of sodium citrate dibasic sesquihydrate. Dispersive SPE kits consisting of 150 mg of primary secondary amine (PSA; average particle size 40 µm) and 900 mg of magnesium sulfate were used to clean 5 mL sample extract. HPLC-grade acetonitrile (99.9%), methanol (99.9%) and water were purchased from J.T. Baker (Philipsburg, NJ, USA). Ammonium formate was purchased from Junsei (Tokyo, Japan). Formic acid (99.0%) was purchased from Daejun (Hwasung, Korea). The mixed analyte protectants (APs) used for

experiments consisted of 3-ethoxy-1,2-propanediol (≥ 98%), D-(+)-gluconic acid-δ-lactone (≥ 99%), D-sorbitol (≥ 98%), and shikimic acid (≥ 99%), which were obtained from Sigma-Aldrich (St. Louis, MO, USA). APs were used after mixing 3:1 with methyl nitrile (MeCN) containing 0.5% formic acid. Triphenyl phosphate (TPP; ≥ 99% purity) was obtained from Sigma-Aldrich and used as an internal standard (IS) in gas chromatography-mass spectrometry (GC-MS/MS) analysis. The buffer solution used to dilute samples for liquid chromatography-tandem mass spectrometry (LC-MS/MS) analysis was 100 mM ammonium formate in water (pH 4–4.5).

Sample preparation (QuEChERS)

The samples were prepared according to the citrate-buffered QuEChERS kit. Thus, 5 g of the dried sample was weighed into a 50 mL polypropylene centrifuge tube, and 10 mL of water was added. After 1 h, 10 mL of MeCN containing 0.1 µg/mL of TPP (IS) was added, and the solution was mixed using a Vortex mixer for 1 min. Then, Bekolut QuEChERS extract kits were transferred into the extracts and mixed immediately by manually shaking and holding in icy water for 3–5 min until the heat dissipated. The tubes were mixed using a Vortex mixer for 2 min and centrifuged for 5 min at 3000 rpm. The upper layer (1 mL) was filtered by an NY syringe filter (0.22 µm, 17 mm) and used as a LC-MS/MS sample after diluting with buffer solution. Cleanup was performed by transferring 5 mL of the upper layer to a 15-mL polypropylene centrifuge tube containing Bekolut QuEChERS dispersive SPE. The extract was vortexed for 2 min and then centrifuged for 5 min at 3000 rpm. After filtering with an NY syringe filter (0.22 µm, 17 mm) and adding APs, the sample was used for GC-MS/MS.

Solutions and standards

Working standard solutions were made at the concentrations of 2 and 4 µg/mL by mixing the individual stock solutions (1000 µg/mL) of each pesticide standard for LC-MS/MS and GC-MS/MS analysis, respectively, and the solutions were diluted with the appropriate solvent before use. The solutions were stored at − 20 °C. A 0.1 µg/mL concentration of TPP in acetonitrile was used as a quality control (QC) standard for the GC-MS/MS analysis.

Pesticide residue analysis

GC-MS/MS analysis

The analyses were performed on an Agilent 7000C triple quad mass spectrometer equipped with an Agilent 7890B

GC system (Agilent Technologies, Santa Clara, CA, USA). Chromatographic separations of pesticides were done by using a DB-5MS UI capillary column (30 m × 0.25 mm × 0.25 µm; Agilent Technologies, Santa Clara, CA, USA). Helium (99.999%) was used as the carrier at a constant flow of 1 mL/min. The temperature of a split/splitless injector was held at 250 °C, and the injection volume was 1 µL in splitless mode. The column oven temperature program was as follows: 90 °C for 3 min, increased to 200 °C at a rate of 20 °C per min, and increased to 300 °C at a rate of 8 °C per min (held for 3 min). The total runtime was 30 min. After each injection, the syringe was washed with a 1:1 mixture of methanol and water followed by acetone. The MS/MS detector used electron ionization mode at 70 eV. The MS data were obtained in the multiple reaction monitoring (MRM) mode, and the collision energy of each pesticide in MRM was used as predetermined. The temperatures of the transfer line and ion source were set at 280 °C and 300 °C, respectively. Nitrogen was used as the collision gas, and the solvent delay was 3.2 min.

LC–MS/MS analysis

LC–MS/MS analysis was performed using a Shimadzu LC-8040 instrument (a liquid chromatograph with a triple quadrupole mass detector; Shimadzu, Kyoto, Japan), and the separation of pesticides was performed using an Agilent Pursuit XRs Ultra C18 column (100 mm × 2.0 mm, 2.8 µm particle size; Agilent Technologies, Santa Clara, CA, USA). Deionized water with 5 mM ammonium formate and 0.1% formic acid (mobile phase A) and methanol with 5 mM ammonium formate and 0.1% formic acid (mobile phase B) were used for the gradient program, which started with 10% B for 1 min and was linearly increased to 98% B over 15 min. The column was reconditioned for 4 min back to 10% B. The column temperature was kept at 40 °C, and the injection volume was 20 µL with a constant flow rate of 0.3 mL/min. The ESI source was used in the positive and negative mode, the nebulizing gas flow was 3 L/min, the drying gas flow was 15 L/min, the DL temperature was 240 °C, and the heat block temperature was 400 °C. The MS data were obtained in MRM mode.

Quality control and quantification

Quantitative analysis was based on matrix-matched calibration. The first qualitative analysis of the herbal fruits was performed using a screening calibration curve and a grape blank, a representative matrix. Second, samples with components detected above the MRL were reanalyzed. The matrix-matched calibration curve for reanalysis was prepared using a pesticide-free blank matrix of each sample extracted by the same method. The standard concentrations

of the matrix-matched calibration curves were 0.02, 0.01, 0.005, 0.0025, 0.00125, and 0.00063 (µg/mL) for LC–MS/MS analysis and 0.2, 0.1, 0.05, 0.025, 0.0125, and 0.0063 (µg/mL) for GC–MS/MS analysis. The correlation coefficient of the calibration curve (r^2) was ≥ 0.995 . The standard deviation (σ) was obtained by measuring the concentration of LOQ seven times, and LOD (3σ) and LOQ (10σ) were calculated with a statistical method. Both the LC–MS/MS 207 component and the GC–MS/MS 113 component were measured at levels below 5 ppb. Recovery experiments were carried out three times using rice, cabbage and apples at the concentrations of 10, 50, and 100 ppb, respectively. To verify the validity of the experiment, the precision and accuracy were confirmed. Precision was within 15% with relative standard deviation (% RSD), and accuracy was in the 70–120% range, which fulfilled the criterion for quantitative methods (MFDS, 2019b).

Sample collection

Each dried herbal fruit was collected from 15 different cities or counties, including the main production area in Korea (Figure S1). All samples were obtained from local markets and harvested at maturity in 2018. The collected samples were all domestic and 15 samples of each fruit were analyzed.

Health risk assessment

The acute/short-term consumer health risk (aHI) was calculated based on the estimated short-term intake (ESTI) and the acute reference dose (ARfD). The chronic/long-term consumer health risk (hazard quotient, HQ) was calculated based on the estimated daily intake (EDI) and the acceptable daily intake (ADI). The relevant formulas are as follows. For a precise evaluation, the ARfD and ADI are expressed as a percentage of daily intake for a 60 kg person. A HQ value higher than 100% indicates an unacceptable risk, and a higher aHI/HQ value represents a higher risk.

$$\text{ESTI} = \frac{\text{the highest residue level}}{\text{food consumption/body weight}} \quad (1)$$

$$\text{aHI} = \text{ESTI}/\text{ARfD} \times 100\% \quad (2)$$

$$\text{EDI} = \frac{\text{mean residue level}}{\text{food consumption/body weight}} \quad (3)$$

$$\text{HQ} = \text{EDI}/\text{ADI} \times 100\% \quad (4)$$

The cumulative risk index (cHI) was calculated by the sum of HQs from the multiple pesticide residues that the consumer is exposed to, which is calculated by summing the HQs for each pesticide in the sample: $\text{cHI} = \sum \text{HQ}$ (5).

Table 1 Occurrence of detected pesticides and their residue levels in dry *S. chinensis*

Pesticide	Occurrence	Registration in Korea ^a	Maximum concentration of residue (mg/kg)	Mean concentration of residue (mg/kg)	Korean MRL (mg/kg)	Mode of action ^b
<i>Insecticide</i>						
Acetamiprid	2	O	0.012	0.011 ± 0.001	2.0	4a
Bifenthrin	2	O	0.138	0.086 ± 0.052	0.01*	3a
Buprofezin	6	O	0.068	0.038 ± 0.010	3.0	16
Clothianidin	5	O	0.065	0.042 ± 0.010	0.01*	4a
Deltamethrin	3	O	0.158	0.094 ± 0.033	0.01*	3a
Dinotefuran	7	–	1.343	0.546 ± 0.231 [†]	0.01*	4a
Etofenprox	8	O	0.104	0.055 ± 0.014	0.01*	3a
Fenitrothion	3	O	0.188	0.074 ± 0.057	5.0	1b
Fenvalerate	1	–	0.303	0.303	0.01*	3a
Imidacloprid	1	O	0.010	0.010	0.01*	4a
Methoxyfenozide	6	O	0.016	0.013 ± 0.002	1.0	18
Novaluron	2	O	1.275	0.701 ± 0.575	0.01*	15
Tebuconazole	1	O	0.005	0.005	0.01*	18
Thiamethoxam	2	–	0.033	0.023 ± 0.011	0.01*	4a
<i>Fungicide</i>						
Azoxystrobin	3	O	0.147	0.064 ± 0.042	2.0	C3
Boscalid	5	–	0.055	0.032 ± 0.008	0.01*	C2
Carbendazim	3	–	0.172	0.071 ± 0.051	0.01*	B2
Fenbuconazole	1	O	0.13	0.013	3.0	G1
Fluopyram	7	–	0.573	0.149 ± 0.080	0.01*	C2
Iprodione	6	–	2.516	0.652 ± 0.386	0.01*	E3
Penthiopyrad	2	–	0.034	0.026 ± 0.009	0.01*	C2
Pyraclostrobin	10	O	0.132	0.067 ± 0.012	5.0	C3
Tetraconazole	2	–	0.024	0.020 ± 0.004 [†]	0.01*	G1
Trifloxystrobin	6	O	0.123	0.040 ± 0.017	7.0	C3

*MRL of 0.01 ppm was applied for unregistered pesticides

^aO: registered, –: not registered. ^bThe mode of action code is from the IRAC and FRAC (FRAC, 2019; IRAC, 2019). [†]Mean concentration of residue exceeded Korean MRL

Results and discussion

Occurrence of pesticides in *S. chinensis*

Pesticides were detected in 13 out of the 15 total *S. chinensis* samples (Table 1). The number of pesticides detected in each sample ranged from 2 to 13. In total, 24 kinds of pesticides were detected in 13 samples, representing 14 insecticides and 10 fungicides, and the average was 1.84 kinds per sample. The most commonly detected pesticide was pyraclostrobin (76.9% of 13 samples), followed by etofenprox (61.5%), dinotefuran, fluopyram (53.8%), buprofezin, methoxyfenozide, iprodione, and trifloxystrobin (46.2%). The maximum residue concentration of insecticides ranged between 0.010 and 1.343 mg/kg and that of fungicides was 0.024 – 2.516 mg/kg. When classified according to mode of action, among the detected insecticides, nicotinic acetylcholine receptor (nAChR)

competitive modulator, 4a (neonicotinoids) was the predominant type, and sodium channel modulators, 3a (pyrethroid) followed. Among the fungicides, C3 and C2, which are photosynthesis II (PSII) inhibitors, were abundant.

Occurrence of pesticides in *L. chinense*

Pesticides were detected in 14 out of the 15 total *L. chinense* samples (Table 2). The number of pesticides detected in each sample ranged from 2 to 21 kinds with an average of 10.28 kinds per sample. In total, 43 kinds of pesticides were detected in 14 samples, including 27 insecticides and 15 fungicides. The most commonly detected pesticide was pyraclostrobin and tebuconazole (64.3% of 14 samples), followed by azoxystrobin, etofenprox (50.0%), bifenthrin, dinotefuran, imidacloprid, indoxacarb, spiromesifen, and difenoconazole (42.8%). The maximum residue concentration of boscalid was 3.14 mg/kg, which was the highest

Table 2 Results of pesticide residue analysis of dry *L. chinense*

Pesticide	Occurrence	Registration in Korea ^a	Maximum concentration of residue (mg/kg)	Mean concentration of residue (mg/kg)	Korean MRL (mg/kg)	Mode of action ^b
<i>Insecticide</i>						
Acetamiprid	3	—	0.172	0.082 ± 0.045 [†]	0.01*	4a
Bifenthrin	6	—	0.056	0.024 ± 0.007 [†]	0.01*	3a
Chlorantraniliprole	5	—	0.369	0.160 ± 0.057 [†]	0.01*	28
Chlorfenapyr	5	O	0.130	0.040 ± 0.023	2.0	13
Chlorpyrifos	2	—	0.020	0.014 ± 0.006 [†]	0.01*	1b
Clothianidin	4	O	0.090	0.062 ± 0.0107	1.0	4a
Cyhalothrin	3	O	0.117	0.053 ± 0.032	2.0	3a
Cypermethrin	4	O	0.144	0.060 ± 0.028	5.0	3a
Deltamethrin	3	O	0.026	0.030 ± 0.009	2.0	3a
Dinotefuran	6	O	0.234	0.177 ± 0.037	0.01*	4a
Etofenprox	7	O	0.138	0.056 ± 0.020	3.0	3a
Flonicamid-	5	O	0.017	0.116 ± 0.085	5.0	29
Fenitrothion	1	O	0.011	0.011	3.0	1b
Fenpropathrin	1	—	0.009	0.009	0.01*	3a
Flubendiamide	3	—	0.098	0.050 ± 0.025 [†]	0.01*	28
Imidacloprid	6	O	0.100	0.063 ± 0.015	5.0	4a
Indoxacarb	6	O	0.083	0.039 ± 0.012	10.0	22a
Lufenuron	2	O	0.025	0.021 ± 0.004	0.01*	15
Methomyl	1	—	0.092	0.092	0.01*	1a
Methoxyfenozide	1	—	0.053	0.053 [†]	0.01*	18
Novaluron	3	O	0.092	0.038 ± 0.027	5.0	15
Pyridaben	1	—	0.023	0.023 [†]	0.01*	UN
Pyridalyl	3	O	0.063	0.052 ± 0.006	20.0	21a
Spiromesifen	6	O	0.118	0.047 ± 0.017	3.0	23
Sulfoxaflor	1	O	0.026	0.026	0.01*	4c
Tebufozide	1	—	0.037	0.037 [†]	0.01*	18
Thiamethoxam	1	O	0.009	0.009	0.01*	4a
<i>Fungicide</i>						
Azoxystrobin	7	O	0.710	0.217 ± 0.087	10.0	C3
Bitertanol	1	—	0.006	0.006	0.01*	G1
Boscalid	3	—	3.149	1.642 ± 0.906	0.01*	C2
Carbendazim	4	—	0.083	0.030 ± 0.018	0.01*	B1
Cyazofamid	4	—	0.183	0.252 ± 0.093 [†]	0.01*	C4
Difenoconazole	6	O	0.501	0.206 ± 0.077	7.0	G1
Fluopicolide	4	—	0.772	0.351 ± 0.141 [†]	0.01*	B5
Hexaconazole	1	—	0.007	0.007	0.01*	G1
Iprodione	1	—	0.165	0.165 [†]	0.01*	E3
Prochloraz	1	—	0.014	0.014 [†]	0.01*	G1
Procymidone	1	—	0.008	0.008	0.01*	E3
Propamocarb	2	—	0.442	0.272 ± 0.072 [†]	0.01*	F4
Pyraclostrobin	9	O	0.700	0.285 ± 0.072	5.0	C3
Tebuconazole	9	O	0.203	0.071 ± 0.023	10.0	G1
Trifloxystrobin	1	—	0.007	0.007	0.01	C3

*MRL of 0.01 ppm was applied for unregistered pesticides

^aO: registered, —: not registered. ^bThe mode of action code is from the IRAC and FRAC (FRAC, 2019; IRAC, 2019). [†]Mean concentration of residue exceeded Korean MRL

Table 3 Results of pesticide residue analysis of dry *C. officinalis*

Pesticide	Occurrence	Registration in Korea ^a	Maximum concentration of residue (mg/kg)	Mean concentration of residue (mg/kg)	Korean MRL (mg/kg)	Mode of action ^b
<i>Insecticide</i>						
Buprofezin	1	–	0.014	0.014	0.01*	16
Chlorfenapyr	1	–	0.015	0.015	0.01*	13
Chlorpyrifos	3	–	0.013	0.009 ± 0.002	0.01*	1b
Cyhalothrin	7	–	0.065	0.023 ± 0.007	0.01*	3a
Cypermethrin	6	–	0.680	0.283 ± 0.099	0.01*	3a
Deltamethrin	4	–	0.077	0.031 ± 0.016	0.01*	3a
Dinotefuran	11	–	0.727	0.101 ± 0.064	0.01*	4a
Etofenprox	8	O	0.292	0.100 ± 0.031	0.01*	3a
Fenitrothion	6	–	0.246	0.060 ± 0.037	0.01*	1b
Phosphamidon	6	–	0.021	0.013 ± 0.002	0.01*	1b
Pyridalyl	1	–	0.020	0.022	0.01*	UN
<i>Fungicide</i>						
Carbendazim	9	O	0.115	0.062 ± 0.014	0.01*	B1
Difenoconazole	3	O	0.038	0.026 ± 0.007	0.01*	G1
Iprodione	3	–	0.050	0.031 ± 0.011	0.01*	E3
Metconazole	2	–	0.015	0.014 ± 0.001	0.01*	G1
Propamocarb	1	–	0.010	0.010	0.01*	F4
Pyraclostrobin	3	–	0.015	0.011 ± 0.003	0.01*	C3
Tebuconazole	4	–	0.016	0.011 ± 0.003	0.01*	G1
Trifloxystrobin	2	–	0.032	0.027 ± 0.006	0.01*	C3
<i>Herbicide</i>						
Pendimethalin	1	–	0.011	0.011	0.01*	K1

*MRL of 0.01 ppm was applied for unregistered pesticides

^aO: registered, -: not registered. ^bThe mode of action code is from the IRAC, FRAC and HRAC (FRAC, 2019; HRAC, 2019; IRAC, 2019). [†] Mean concentration of residue exceeded Korean MRL

value among the detected pesticides. The maximum residue concentration of insecticides ranged between 0.009 and 0.369 mg/kg and that of fungicides was 0.007–0.710 mg/kg, except boscalid. When classified according to mode of action, among the detected insecticides, 4a (neonicotinoids) was the most predominant type, followed by 3a (pyrethroids). Among the fungicides, G1, a demethylation inhibitor, was the most predominant, followed by C3.

Occurrence of pesticides in *C. officinalis*

Pesticides were detected in 14 out of the 15 total *C. officinalis* samples (Table 3). The number of pesticides detected in each sample ranged from 2 to 12 kinds, with an average of 5.87 kinds per sample. In total, 20 kinds of pesticides were detected in 14 samples, including 11 insecticides, 8 fungicides and 1 herbicide. The most commonly detected pesticide was dinotefuran (78.6% of 14 samples), followed by carbendazim (64.3%), etofenprox (57.1%), cyhalothrin (50%), cypermethrin, fenitrothion, and phosphamidon (42.8%). The maximum residue

concentration of insecticides ranged between 0.013 and 0.727 mg/kg and that of fungicides ranged from 0.010 to 0.115 mg/kg. When classified according to mode of action, among the detected insecticides, 3a (pyrethroids) was the most abundant, followed by 1b (organophosphates), acetylcholinesterase (AChE) inhibitors and 4a (neonicotinoids). Among the fungicides, B1 and G1 were abundant.

In this study, we investigated the pesticide residues of three domestic herbal fruits which were produced nationally. The frequencies of occurrence and the kind of pesticides used were increased in this survey compared with previous reports. Yu et al. (2012) reported that pesticides were detected in 33.3% of 9 domestic dry *S. chinensis* samples, 91.7% of 12 *L. chinense* samples, and 27.3% of 11 *C. officinalis* samples and that 4, 10, and 4 kinds of pesticides were present in domestic and imported *S. chinensis*, *L. chinense*, and *C. officinalis*, respectively, in 2012 (Yu et al., 2012). In fresh *S. chinensis* fruit, 3 kinds of pesticides were detected (Jeong et al., 2012). From the previous two reports, endosulfan, an organochloride, was detected from *S. chinensis*, while in this survey, no

Table 4 The short-term and long-term risks due to average daily intake of pesticides through *S. chinensis* in Korea

Pesticide	Short-term risk			Long-term risk		
	ESTI (mg kg ⁻¹ day ⁻¹)	ARfD (mg /kg ⁻¹ bw ⁻¹ day ⁻¹)	aHI (%)	EDI (mg /kg ⁻¹ day ⁻¹)	ADI (mg /kg ⁻¹ bw ⁻¹ day ⁻¹)	HQ (%)
Acetamiprid	5.0×10^{-6}	0.100	0.005	4.6×10^{-6}	0.071	0.01
Bifenthrin	5.8×10^{-5}	0.010	0.576	3.6×10^{-5}	0.010	0.36
Buprofezin	2.8×10^{-5}	0.500	0.006	1.6×10^{-5}	0.009	0.18
Clothianidin	2.7×10^{-5}	0.600	0.005	1.8×10^{-5}	0.097	0.02
Deltamethrin	6.6×10^{-5}	0.050	0.132	3.9×10^{-5}	0.010	0.39
Dinotefuran	5.6×10^{-4}	1.250	0.045	2.3×10^{-4}	0.020	1.14
Etofenprox	4.3×10^{-5}	1.000	0.004	2.3×10^{-5}	0.030	0.08
Fenitrothion	7.8×10^{-5}	0.013	0.604	3.1×10^{-5}	0.005	0.62
Fenvalerate	1.3×10^{-4}	0.020	0.633	1.3×10^{-4}	0.020	0.63
Imidacloprid	4.2×10^{-6}	0.400	0.001	4.2×10^{-6}	0.060	0.01
Methoxyfenozide	6.7×10^{-6}	0.900	0.001	5.4×10^{-6}	0.100	0.01
Novaluron	5.3×10^{-4}	–	–	2.9×10^{-4}	0.010	2.93
Tebufozide	2.1×10^{-6}	0.900	2.3×10^{-4}	2.1×10^{-6}	0.020	0.01
Thiamethoxam	1.4×10^{-5}	0.350	0.004	9.6×10^{-6}	0.080	0.01
Azoxystrobin	6.1×10^{-5}	–	–	2.7×10^{-5}	0.200	0.01
Boscalid	2.3×10^{-5}	–	–	1.3×10^{-5}	0.040	0.03
Carbendazim	7.2×10^{-5}	0.500	0.014	3.0×10^{-5}	0.030	0.10
Fenbuconazole	5.4×10^{-5}	–	–	5.4×10^{-6}	0.030	0.02
Fluopyram	2.4×10^{-4}	0.050	0.478	6.2×10^{-5}	0.030	0.21
Iprodione	1.1×10^{-3}	–	–	2.7×10^{-4}	0.060	0.45
Penthiopyrad	1.4×10^{-5}	0.500	0.003	1.1×10^{-5}	0.081	0.01
Pyraclostrobin	5.5×10^{-5}	0.050	0.110	2.8×10^{-5}	0.030	0.09
Tetraconazole	1.0×10^{-5}	0.050	0.020	8.4×10^{-6}	0.004	0.21
Trifloxystrobin	5.1×10^{-5}	–	–	1.7×10^{-5}	0.040	0.04

ARfD and ADI are from the Korean Food Standard Codex (Ministry of Food and Drug Safety [MFDS], 2019b). The “–” symbol indicates that there was no authorized ARfD value and that the corresponding risk index could not be calculated. The fruit consumption values are based on data from the Guidelines for Standardization of Food Intake (NIFDS, 2019)

endosulfan was detected from *S. chinensis*. The use of endosulfan in Korea has been severely restricted since 2012, and its use is banned in New Zealand (ERMA, 2008; RDA, 2018). The Stockholm Convention's Persistent Organic Pollutants Review Committee (POPRC) agreed to phase out this pesticide worldwide (Secretariat of the Stockholm Convention, 2009).

Most of the samples contained the residues of more than two pesticides. The occurrence of multiple residues in samples may result from the application of different types of pesticides to protect a crop from different pests and diseases and has been reported in different commodities (Jallow et al., 2017; Liu et al., 2016; Mac Loughlin et al., 2018; Suntudrob et al., 2015). Farmers with limited education and lack of knowledge on pesticide handling preferred to mix high potency pesticides to make highly toxic cocktails with dosages higher than the recommended limits, resulting in strong and fast-acting pesticides. Therefore, constant farmer education regarding the application of

pesticides is needed. Among the detected insecticides, etofenprox and dinotefuran were detected frequently in the 3 herbal fruits, although their risks are very small. Etofenprox is a technical grade active ingredient pesticide unlikely to present an acute hazard in normal use (WHO, 2009). However, etofenprox negatively affected the proportion of homing flights of honeybees (Matsumoto, 2013). Dinotefuran is categorized as a neonicotinoid, and neonicotinoids are widely used, accounting for more than 25% of the pesticides used worldwide. Neonicotinoids cause a wide range of adverse sublethal effects in honeybee and bumblebee colonies (van der Sluijs et al., 2013). The EU prohibited the use of the three neonicotinoid chemicals on crops that attract bees (Butler, 2018; European Union, 2013). Therefore, the use of both pesticides should be reduced to protect bees. Various kinds of pesticides may prevent organism resistance; however, the use of pesticides with the same mode of action does not prevent resistance. To prevent resistance to agrochemicals, the use of various

Table 5 The short-term and long-term risks due to average daily intake of pesticides through *L. chinense* in Korea

Pesticide	Short-term risk			Long-term risk		
	ESTI (mg kg ⁻¹ day ⁻¹)	ARfD (mg /kg ⁻¹ bw ⁻¹ day ⁻¹)	aHI (%)	EDI (mg /kg ⁻¹ day ⁻¹)	ADI (mg /kg ⁻¹ bw ⁻¹ day ⁻¹)	HQ (%)
Acetamiprid	4.1×10^{-10}	0.100	4.1×10^{-7}	2.3×10^{-10}	0.071	3.2×10^{-7}
Bifenthrin	1.2×10^{-10}	0.010	1.2×10^{-6}	3.6×10^{-11}	0.010	3.6×10^{-7}
Chlorantraniliprole	8.0×10^{-10}	—	—	2.8×10^{-10}	2.000	1.4×10^{-8}
Chlorfenapyr	2.0×10^{-10}	0.030	6.7×10^{-7}	1.1×10^{-10}	0.026	4.4×10^{-7}
Chlorpyrifos	7.0×10^{-11}	0.030	1.0×10^{-1}	7.0×10^{-11}	0.010	7.0×10^{-7}
Clothianidin	3.1×10^{-10}	0.600	5.2×10^{-8}	8.6×10^{-11}	0.097	8.9×10^{-8}
Cyhalothrin	2.7×10^{-10}	0.020	1.3×10^{-6}	1.6×10^{-10}	0.020	8.0×10^{-7}
Cypermethrin	3.0×10^{-10}	0.040	7.5×10^{-7}	1.4×10^{-10}	0.020	7.1×10^{-7}
Deltamethrin	1.5×10^{-10}	0.050	3.0×10^{-7}	4.4×10^{-11}	0.010	4.4×10^{-7}
Dinotefuran	8.9×10^{-10}	1.250	7.1×10^{-8}	1.8×10^{-10}	0.020	9.2×10^{-7}
Etofenprox	2.8×10^{-10}	1.000	2.8×10^{-8}	1.0×10^{-10}	0.030	3.4×10^{-7}
Flonicamid	5.8×10^{-10}	0.025	2.3×10^{-6}	4.3×10^{-10}	0.025	1.7×10^{-6}
Fenitrothion	5.5×10^{-10}	0.013	4.2×10^{-6}	5.5×10^{-11}	0.005	1.1×10^{-6}
Fenpropathrin	4.5×10^{-11}	—	—	4.5×10^{-11}	0.030	1.5×10^{-7}
Flubendiamide	2.5×10^{-10}	0.200	1.3×10^{-7}	1.2×10^{-10}	0.017	7.3×10^{-7}
Imidacloprid	3.2×10^{-10}	0.400	7.9×10^{-8}	7.3×10^{-11}	0.060	1.2×10^{-7}
Indoxacarb	2.0×10^{-10}	0.100	2.0×10^{-7}	5.8×10^{-11}	0.010	5.8×10^{-7}
Lufenuron	1.1×10^{-10}	—	—	1.1×10^{-10}	0.015	7.0×10^{-7}
Methomyl	4.6×10^{-10}	0.020	2.3×10^{-6}	4.6×10^{-10}	0.020	2.3×10^{-6}
Methoxyfenozide	2.7×10^{-10}	0.900	2.9×10^{-8}	2.7×10^{-10}	0.100	2.7×10^{-7}
Novaluron	1.9×10^{-10}	—	—	1.4×10^{-10}	0.010	1.4×10^{-6}
Pyridaben	1.2×10^{-10}	0.050	2.3×10^{-7}	1.2×10^{-10}	0.005	2.3×10^{-6}
Pyridalyl	2.6×10^{-10}	—	—	2.9×10^{-11}	0.028	1.0×10^{-7}
Spiromesifen	2.4×10^{-10}	2.000	1.2×10^{-8}	8.7×10^{-11}	0.030	2.9×10^{-7}
Sulfoxaflor	1.3×10^{-10}	0.300	4.3×10^{-8}	1.3×10^{-10}	0.050	2.6×10^{-7}
Tebufozide	1.9×10^{-10}	0.900	2.1×10^{-8}	1.9×10^{-10}	0.020	9.3×10^{-7}
Thiamethoxam	4.5×10^{-11}	0.350	1.3×10^{-8}	4.5×10^{-11}	0.080	5.6×10^{-8}
Azoxystrobin	1.1×10^{-9}	—	—	4.4×10^{-10}	0.200	2.2×10^{-7}
Bitertanol	3.0×10^{-11}	—	—	3.0×10^{-11}	0.010	3.0×10^{-7}
Boscalid	8.2×10^{-9}	—	—	4.5×10^{-9}	0.040	1.1×10^{-5}
Carbendazim	1.5×10^{-10}	0.500	3.0×10^{-8}	8.9×10^{-11}	0.030	3.0×10^{-7}
Cyazofamid	1.3×10^{-9}	—	—	4.6×10^{-10}	0.170	2.7×10^{-7}
Difenoconazole	1.0×10^{-9}	0.300	3.4×10^{-7}	3.8×10^{-10}	0.010	3.8×10^{-6}
Fluopicolide	1.8×10^{-9}	0.180	9.8×10^{-7}	7.1×10^{-10}	0.079	8.9×10^{-7}
Hexaconazole	3.5×10^{-11}	0.250	1.4×10^{-8}	3.5×10^{-11}	0.005	7.0×10^{-7}
Iprodione	8.3×10^{-10}	—	—	8.3×10^{-10}	0.060	1.4×10^{-6}
Prochloraz	7.0×10^{-11}	0.100	7.0×10^{-8}	7.0×10^{-11}	0.010	7.0×10^{-7}
Procymidone	4.0×10^{-11}	0.100	4.0×10^{-8}	4.0×10^{-11}	0.100	4.0×10^{-8}
Propamocarb	1.4×10^{-9}	2.000	6.8×10^{-8}	8.5×10^{-10}	0.400	2.1×10^{-7}
Pyraclostrobin	1.4×10^{-9}	0.050	2.9×10^{-6}	3.6×10^{-10}	0.030	1.2×10^{-6}
Tebuconazole	3.6×10^{-10}	0.300	1.2×10^{-7}	1.1×10^{-10}	0.030	3.8×10^{-7}
Trifloxystrobin	3.5×10^{-11}	—	—	3.5×10^{-11}	0.040	8.8×10^{-8}

ARfD and ADI are from the Korean Food Standard Codex (MFDS, 2019b). The “—” symbol indicates that there was no authorized ARfD value and that the corresponding risk index could not be calculated. The fruit consumption values are based on data from the Guidelines for Standardization of Food Intake (NIFDS, 2019)

Table 6 The short-term and long-term risks due to average daily intake of pesticides through *C. officinalis* in Korea

Pesticide	Short-term risk			Long-term risk		
	ESTI (mg kg ⁻¹ day ⁻¹)	ARfD (mg /kg ⁻¹ bw ⁻¹ day ⁻¹)	aHI (%)	EDI (mg /kg ⁻¹ day ⁻¹)	ADI (mg /kg ⁻¹ bw ⁻¹ day ⁻¹)	HQ (%)
Buprofezin	1.63×10^{-10}	0.500	3.3×10^{-8}	1.6×10^{-10}	0.009	1.8×10^{-6}
Chlorfenapyr	1.75×10^{-10}	0.030	5.8×10^{-7}	1.8×10^{-10}	0.026	6.7×10^{-7}
Chlorpyrifos	1.52×10^{-10}	0.100	1.5×10^{-7}	1.0×10^{-10}	0.010	1.0×10^{-6}
Cyhalothrin	7.58×10^{-10}	0.020	3.8×10^{-6}	2.7×10^{-10}	0.020	1.4×10^{-6}
Cypermethrin	7.93×10^{-9}	0.040	2.0×10^{-5}	3.3×10^{-9}	0.020	1.6×10^{-5}
Deltamethrin	8.98×10^{-10}	0.050	1.8×10^{-6}	3.6×10^{-10}	0.010	3.6×10^{-6}
Dinotefuran	8.48×10^{-9}	1.250	6.8×10^{-7}	1.2×10^{-9}	0.020	5.9×10^{-6}
Etofenprox	3.41×10^{-9}	1.000	3.4×10^{-7}	1.2×10^{-9}	0.030	3.9×10^{-6}
Fenitrothion	2.87×10^{-9}	0.013	2.2×10^{-5}	7.0×10^{-10}	0.005	1.4×10^{-5}
Phosphamidon	2.45×10^{-10}	—	—	1.6×10^{-10}	0.001	3.1×10^{-5}
Pyridalyl	2.33×10^{-10}	—	—	2.6×10^{-10}	0.028	9.2×10^{-7}
Carbendazim	1.34×10^{-9}	0.500	2.7×10^{-7}	7.2×10^{-10}	0.030	2.4×10^{-6}
Difenoconazole	4.43×10^{-10}	0.300	1.5×10^{-7}	3.1×10^{-10}	0.010	3.1×10^{-6}
Iprodione	5.83×10^{-10}	—	—	3.6×10^{-10}	0.060	6.0×10^{-7}
Metconazole	1.75×10^{-10}	0.010	1.8×10^{-6}	1.6×10^{-10}	0.040	4.1×10^{-7}
Propamocarb	1.17×10^{-10}	2.000	5.8×10^{-9}	1.2×10^{-10}	0.400	2.9×10^{-8}
Pyraclostrobin	1.75×10^{-10}	0.050	3.5×10^{-7}	1.3×10^{-10}	0.030	4.3×10^{-7}
Tebuconazole	1.87×10^{-10}	0.300	6.2×10^{-8}	1.3×10^{-10}	0.030	4.4×10^{-7}
Trifloxystrobin	3.73×10^{-10}	—	—	3.1×10^{-10}	0.040	7.7×10^{-7}
Pendimethalin	1.28×10^{-10}	—	—	1.3×10^{-10}	0.130	9.9×10^{-8}

ARfD and ADI are from the Korean Food Standard Codex (MFDS, 2019b). The “—” symbol indicates that there was no authorized ARfD value and that the corresponding risk index could not be calculated. The fruit consumption values are based on data from the Guidelines for Standardization of Food Intake (NIFDS, 2019)

types of pesticides with different modes of action, is recommended. Methomyl and phosphamidon were detected in *L. chinense* and *C. officinalis*, respectively. Methomyl, a carbamate, is classified as a highly hazardous pesticide, and phosphamidon, an organophosphate, is as extremely hazardous (WHO, 2009). The use of these pesticides is severely restricted by law due to their high toxicity (RDA, 2018). The use of phosphamidon is banned or severely restricted in many countries (FAO, 1997). To protect the user of the pesticide and the health of the consumer, additional education and strong legal sanctions are needed.

Additionally, 12 out of the 14 detected insecticides and 8 out of the 10 fungicides in dry *S. chinensis*, 21 out of the 27 insecticides and 4 out of the 15 fungicides in dry *L. chinense* and 2 out of the 11 insecticides and 2 out of the 8 fungicides in *C. officinalis* are registered as legal agrochemicals in Korea (NIAS, 2019); the herbicide present in *C. officinalis* was not registered. In Korea, MRLs are established for only 4 registered insecticides and 3 fungicides in dry *S. chinensis* and for 11 registered insecticides and 4 fungicides *L. chinense*. Moreover, the MRLs for pesticides in *C. officinalis* have not yet been established in Korea. The use of pesticides with no MRL is revealed in

this study. To minimize the use of unauthorized pesticides on minor crops, the Rural Development Administration (RDA) of Korea and the Ministry of Food and Drug Safety (MFDS), who are responsible for implementing national legislation, extend the MRL to all crops. Furthermore, the Korean government authorities established an MRL of 0.01 ppm for unregistered pesticides. These regulations are would reduce the health risks associated with pesticide residues in herbal medicines to which local consumers are exposed.

Health risk assessment

To assess the risk of consumer exposure to pesticide residues in herbal fruit samples, the estimated daily intakes were expressed as percentages of the ARfD and ADI values for the detected pesticides as derived from Korean Food Standard Codex evaluations (MFDS, 2019b). The dietary exposure to pesticides (mg/kg bw day) was calculated based on consumption data, individual body weights and residue monitoring data (the highest residue and mean residue). The results of each herbal fruit are shown in Tables 4, 5 6. In the short-term risk assessment, all the

ESTI values were much lower than the ARfD values submitted by the MFDS in all three herbal fruits. In addition, the higher risk of *S. chinensis* was from fenvalerate, fenitrothion, bifenthrin, and fluopyram with aHI values of 0.633%, 0.604%, 0.576% and 0.478%, respectively. In *S. chinensis*, the HQ value of the novaluron pesticide was 2.93%. The cHI for the detected pesticides in the three herbal fruits, *S. chinensis*, *L. chinense*, and *C. officinalis*, were 7.56%, $4.0 \times 10^{-5}\%$, and $8.9 \times 10^{-5}\%$, respectively.

Compared with other reports on minor fruits, nuts and vegetables (Kumari and John, 2019; Liu et al., 2016; Yang et al., 2016), this work found that the health risk from pesticide residue in the herbal fruits was relatively small, which indirectly reflects the lower residue level of pesticides in the herbal fruits.

Humans are generally exposed to many different chemicals including pesticides. Pesticide residues in agricultural commodities might be transferred to humans via food consumption. Bioaccumulation of the pesticides may take place in body organs and cause direct and indirect adverse effects on human health (Taghizadeh et al., 2019). This study has shown that there was some degree of pesticide contamination in the herbal fruits *S. chinensis*, *L. chinense*, and *C. officinalis* from Korea. The health risk assessment revealed that low pesticide residue levels were found in herbal fruits, indicating a negligible risk for systemic toxicity to consumers.

This research has provided important information on pesticide residue contamination in three herbal medicines. These results support the registration of new pesticides and the establishment of MRLs.

Acknowledgements This research was funded by National Institute of Forest Science (FP0802-2019-02-2019).

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