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Reduction of Pesticide Residues in the Production of Red Pepper Powder

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Abstract Six organophosphorus, one organochlorine, and three synthetic pyrethroid pesticides were analyzed for their residues during washing and hot-air drying of red peppers conducted in the production of powder. The residue ratio in organophosphorus pesticides was 33% in chlorpyrifos, 31% in diazinon, 50% in methidathion, 80% in EPN, 28% in fenitrothion, and 60% in profenofos. The ratio in pyrethroids was 109% in cypermethrin, 102% in deltamethrin, and 106% in fenvalerate. That in organochlorine was 56% in α-endosulfan and 90% in β-endosulfan. The results were greatly different between organophosphorus and pyrethroid pesticides. UV irradiation along with hot-air drying brought about a remarkable reduction of the residues, up to 70% as compared with hot-air drying only. The removal effect was most remarkable in pyrethroids, which are hardly removed by hot-air drying. The color of the pepper was not changed during UV irradiation. The use of oxidizing agents such as hydrogen peroxide or chlorine dioxide during washing did not show a remarkable removal of residues. The residue ratio was not affected whether the pesticide is contaminated artificially or naturally.

Keywords: red pepper, pesticide residues, washing and drying, residue reduction, UV irradiation

Introduction

Hot pepper (*Capsicum annuum* L.) is a very important spicy seasoning in Korean diets. The planted acreage of the crop reaches 70 thousand hectares, which is the largest among vegetables in Korea. The annual production of raw hot pepper amounts to 1.2 million tons, of which 83% are used as a dried and powdered form (1). Hot pepper powder is widely used in *kimchi*, hot soybean paste, soup base, and spices in Korean dishes.

A survey on the use of pesticides indicates that 57 kinds of pesticides are used in hot pepper cultivation. Among 89 pesticides registered for use on the crop as of 1999 (2), maximum residue limits (MRLs) were set for 70 pesticides in Korea (3). These figures indicate the heavy use of pesticides in hot pepper production, leading to concern about the health risk of the residues. In particular, the residues often remain on the pepper skin, which is eaten directly (4, 5).

Pesticide residues on raw vegetables can be removed during processing or cooking, and Lee *et al.* (4) estimated that about half of the residues are removed in the case of organophosphorus pesticides. Seo (6) reported the removal of deltamethrin, methomyl, and procymidone by drying processes, and Lee (7) also studied the removal of chlorpyrifos and fenitrothion in the washing and drying processes of hot pepper. Recent studies by Chung *et al.* (2) on organophosphorus and pyrethroid pesticides showed that most important steps of residue removal from hot pepper fruits were the washing and hot-air drying. However, most of previous studies were undertaken with artificially contaminated red pepper samples, which may reveal impractical results and raise the question of data reliability.

The present experiments were, therefore, undertaken with pepper samples naturally contaminated by 10 pesticides, including 6 organophosphorus, 1 organochlorine, and 3 synthetic pyrethroids. Pesticide residues were monitored during washing with or without oxidizing agents and consequent drying under UV or without UV rays.

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Materials and Methods

Pesticides used Ten pesticides including organophosphorus (chlorpyrifos, diazinon, EPN, fenitrothion, methidathion, profenofos), organochlorine (endosulfan) and synthetic pyrethroids (cypermethrin, deltamethrin, fenvalerate) used in this study were purchased from Chem Service Inc. (West Chester, PA, USA). Formulations, MRLs and legal registration for individual pesticides are indicated in Table 1.

Table 1. Regulatory status of tested pesticides

Pesticide	Formulation	A.I.[1] (%)	MRL[2] (mg/kg)	Registration[3]
Chlorpyrifos	EC	20	0.5	Yes
Cypermethrin	EC	2	0.5	Yes
Deltamethrin	EC	1	0.2	Yes
Diazinon	EC	34	0.5	Yes
Endosulfan	EC	35	1.0	No
EPN	EC	45	0.1	No
Fenitrothion	EC	50	0.1	Yes
Fenvalerate	EC	5	1.0	Yes
Methidathion	EC	40	-	No
Profenofos	EC	45	2.0	Yes

[1]Active ingredient: effective pesticide constituent.
[2]Maximum residue limit established by Korea Food and Drug Administration.
[3]Registration status by Ministry of Agriculture and Forestry, Korea.

Preparation of contaminated red pepper samples Red-colored hot pepper plants were cultivated in the open field in order to obtain naturally contaminated samples. Ten pesticides were divided into 3 groups, mixed and applied to the test crop as follows: group 1 (chlorpyrifos, diazinon, methidathion), group 2 (EPN, fenitrothion, profenofos), and group 3 (cypermethrin, deltamethrin, endosulfan, fenvalerate). Since each group contained 3-4 kinds of pesticides, the concentrations of active ingredients were adjusted during application to become 1/4 of the recommended concentration for each pesticide. The diluted pesticide solutions were sprayed once a week 4-5 times, and the red pepper fruits were harvested on the second day after the final application.

In order to obtain artificially contaminated red pepper samples, 6 organophosphorus pesticides (chlorpyrifos, diazinon, methidathion, EPN, fenitrothion, profenofos) were diluted in 2 L of water, in which fresh red pepper fruits (7 kg) were soaked for 2 min, drained and air-dried in the hood.

Washing and drying of red pepper About 4 kg of harvested red pepper fruits were washed by dipping in 20 L of tap water by hand, stirring violently for 3 min. The water was drained off until the fruit gave similar weight before and after washing.

The washed pepper fruits weighing 1 kg were placed so as not to overlap on the shelf of a forced drying oven (Shinseng, SFC-102, Seoul, Korea) and dried at 65°C, inverting the pepper every 6 hr to reach about 12% moisture content. About 26 hr were required for complete drying. Stems of dried pepper fruits were removed, and the remaining portion was crushed to obtain red pepper powder by use of a crusher (Shinhan, HMF-500, Seoul, Korea).

Use of oxidizing agents in washing One kg of red pepper fruits was washed with 5 L of water containing 20 % of hydrogen peroxide (using 34.5% H_2O_2) or 50 mg/kg of chlorine dioxide (using Oxyzon containing 5% of ClO_2, EYANG, Seoul, Korea) for 3 min with stirring. The washed pepper was subjected to hot-air drying or UV irradiation, followed by determination of organophosphorus pesticide residues.

UV irradiation during hot-air drying Two UV lamps (254 nm wavelength, 8 W, 70 cm length) were hung in the drying oven, and red peppers were irradiated at 25 cm distance for 26 hr (total drying period) or 12 hr (about half of drying period).

Color of dried peppers was measured using a color difference meter (Color reader CR-10, Minolta, Japan) for the pulp and expressed as Hunter L, a, and b values. The determination was replicated for 50 pieces of the pepper, and the color change was tested by t-test (α=0.05) using the Statistical Analysis System (Cary, NC, USA).

Analysis of pesticide residues The pepper fruits were separated into stems and edible portions (pulp and seeds) and subjected to residue analysis according to the Korea Food Code (8). In the case of fresh samples, a 20 g portion was taken to extract with an appropriate organic solvent, and in the case of dried samples, a 4 g portion was taken, 16 mL water were added, and pesticides were extracted after 1 hr with an appropriate organic solvent.

Adsorbants for column chromatography were active carbon (Daroco®G-60, Aldrich, Milwaukee, WI, USA), cellulose (Avicel®' PH-101, Fluka, Buchs, Switzerland), and florisil (60-100 mesh, activated 12 hr at 130°C, Sigma, St. Louis, MO, USA). Instruments employed were electrical aspirator (Jeio Tech, Kimpo, Korea), homogenizer (Polytron®, Kinematica, Switzerland), rotary evaporator (Büchi, Flawil, Switzerland), and nitrogen concentrator (TurboVap® LV, Zymark, Mountain View, CA, USA). A gas chromatograph (HP 6890 plus, Agilent Tech., Apple Valley, MN, USA) equipped with an HP-5 column (30 m × 0.32 mm × 0.25 μm) was used to analyze organophosphorus pesticides by flame photometric detector and organo-chlorine and synthetic pyrethroids by electron capture detector.

Recovery test Samples for residue analysis were spiked with standard pesticides at 0.5-1.0 mg/kg and subjected to routine analysis in triplicate runs. The recovery was 88-105% in organophosphorus pesticides, 75% in organochlorins and 104-115% in pyrethroids, which are judged to be satisfactory. However, the recovery ratio was not regarded in the calculation of residue concentration. Limit of quantitation (LOQ) determined by a S/N ratio of ten was less than 0.05 mg/kg for all pesticides.

Results and Discussion

Residue removal in the conventional production of red pepper powder In order to obtain the reduction factors in the conventional production of red pepper powder, harvested pepper fruits were washed, hot-air dried and crushed, followed by residue analysis. Although UV irradiation for several minutes or magnetic cleaning is involved in current commercial practices, they were ignored due to negligible removal of residues (2). It is, therefore, assumed that the data presented below should represent the practical value applicable to commercial practices.

The residue levels of pesticides in peppers contaminated naturally were in the range of 0.1-2.0 mg/kg (Table 2). These were consistent with the range of MRLs established for tested pesticides and regarded to be reasonable. The residue ratios of organophosphorus pesticides in red pepper powder after washing and hot-air drying were 32.7 % in chlorpyrifos, 31.3% in diazinon, 80.2% in EPN, 27.7 % in fenitrothion, 49.8% in methidathion, and 59.7% in profenofos. The ratios of organochlorine pesticides were 55.8% in α-endosulfan, 90.3% in β-endosulfan, and those of synthetic pyrethroids were 109% in cypermethrin, 102% in deltamethrin, and 106% in fenvalerate.

Lee (7) reported that the residue ratios of chlorpyrifos and fenitrothion after washing and hot-air drying of artificially contaminated pepper were 64.7 and 68.4%, respectively. Chung *et al.* (2) also reported that the residue ratios of chlorpyrifos, fenitrothion, and profenofos after washing and drying of naturally contaminated pepper were 52.0, 29.5, and 64.1%, respectively. It appears that variability in adherance and analysis of pesticides brings

Table 2. Reduction of pesticide residues in production of red pepper powder

Red pepper sample[1)]	Chlorpyrifos		Cypermethrin		Deltamethrin		Diazinon		Endosulfan		EPN		Fenitrothion		Fenvalerate		Methidathion		Profenofos	
	Conc.[2)] (mg/kg)	R[3)] (%)	Conc. (mg/kg)	R (%)	Conc. (mg/kg)	R (%)	Conc. (mg/kg)	R (%)	Conc. (mg/kg)	R (%)	Conc. (mg/kg)	R (%)	Conc. (mg/kg)	R (%)	Conc. (mg/kg)	R (%)	Conc. (mg/kg)	R (%)	Conc. (mg/kg)	R (%)
Raw	0.406±0.008	100	0.625±0.014	100	0.316±0.005	100	0.367±0.009	100	0.062±0.001[4)] 0.250±0.021[5)]	100 100	1.99±0.001	100	0.692±0.041	100	1.83±0.029	100	0.756±0.037	100	1.62±0.036	100
Washed	0.431±0.003	106	0.613±0.032	98.0	0.312±0.020	98.8	0.400±0.001	109	0.067±0.003 0.273±0.005	108 109	1.65±0.138	82.7	0.537±0.049	77.7	1.77±0.122	95.7	0.780±0.007	103	1.35±0.130	93.3
Dried 26 hr	0.771±0.002	32.7	3.97±0.002	109	1.87±0.003	102	0.668±0.002	31.3	0.201±0.001 1.31±0.003	55.8 90.3	10.3±0.006	80.2	1.23±0.004	27.7	11.3±0.012	106	2.19±0.002	49.8	6.21±0.024	59.7
Dried 26 hr + UV 12hr	0.683±0.001	28.9	2.31±0.001	63.5	1.03±0.002	56.0	0.620±0.001	29.0	0.169±0.000 1.01±0.003	47.0 69.5	7.06±0.020	55.2	1.10±0.001	24.7	5.03±0.007	45.8	1.24±0.005	28.2	5.63±0.021	54.1
Dried 26 hr + UV 26 hr	0.636±0.000	27.0	1.86±0.006	51.0	0.771±0.000	42.0	0.566±0.001	26.5	0.198±0.000 0.960±0.001	55.0 66.2	5.27±0.053	41.2	1.17±0.006	26.4	3.75±0.007	35.0	0.886±0.002	20.2	5.23±0.030	50.2

[1)]Harvested red pepper was washed with water followed by hot-air drying either under UV rays or not.
[2)]Quantities of analyzed samples were 20 g for raw and washed samples and 4 g for dried samples equivalent to the raw. Residue concentration was expressed as mean±SD of triplicate runs.
[3)]Relative ratio of remaining residue.
[4)]Alpha-endosulfan.
[5)]Beta-endosulfan.

about difference or consistence among the results above.

The residue data showed a quite different pattern between organophosphorus and pyrethroids. The removal of organophosphorus was approximately 30-80%, whereas pyrethroids were not removed at all. It is assumed that this difference is due to the different vapor pressure of the pesticides. Since the vapor pressure of pyrethroid fenvalerate (0.019 mPa at 20°C) is much lower than the vapor pressure of organophosphorus chlorpyrifos (2.7 mPa at 25°C) (9), the removal of the pyrethroids during hot-air drying was negligible. The vapor pressure of EPN (0.041 mPa) was the lowest among tested organophosphorus compounds, and this was well reflected in the removal data. The residue ratio of EPN was 82.7% after washing and 80.2% after hot-air drying, indicating that EPN is rarely removed during hot-air drying due to its low vapor pressure.

During production of red pepper powder, the pesticide residues in the raw pepper fruits may be removed partly or not at all depending on the properties of the pesticides; thus attention should be paid to regulation or quality control of pesticide residues.

Effect of UV irradiation on residue removal during hot-air drying Removal of pesticide residues by UV irradiation was attempted by many investigators in expectation of their photodegradation. This type of study is particularly common in the field of environmental research to accelerate the photodegradation of organic wastes by use of catalysts such as TiO_2 or H_2O_2 (10-12). Lee *et al.* (13) investigated photosensitizing agents for the reduction of pesticide residues. Lee & An (14) studied the effects of H_2O_2 and UV irradiation to reduce the residues of benomyl and chlorothalonil applied on solid surfaces to prove that 20% H_2O_2 solution and 350 nm wavelength were the best. Chung *et al.* (2) undertook an experiment to use UV rays in the drying of red peppers artificially contaminated with pesticides. The present study was attempted to verify Chung's experimental results (2) on the effect of UV irradiation employing naturally contaminated red pepper samples with many different pesticides.

Concomitant UV irradiation and hot-air drying for 26 hr brought about 70% reduction of pesticide residues on the average, as compared with a hot-air drying only. The residue ratios of individual pesticides in irradiated and non-irradiated pepper samples are given in Table 2 and Fig. 1. In particular, pyrethroids, EPN, and β-endosulfan, which are not removed easily by drying only, were removed to a greater extent. These results are consistent with the results of Chung *et al.* (2). In general, synthetic pyrethroids revealed persistence in the environment. The higher removal ratio of pyrethroids in comparison with organophosphorus compounds may be explanined from their easier photodegradation.

Red color is important in hot pepper, and it should not be discolored in UV irradiation. The color of hot pepper powder was expressed in terms of Hunter a/b values, which were 4.27 for hot-air dried peppers and 4.56 for peppers hot-air dried under UV irradiation (Table 3). Since the a/b value doesn't change significantly with UV irradiation, the preferred red color in hot pepper will not be affected by UV. Therefore, the concurrent use of hot air and UV radiation should be a very simple and effective way to remove pesticide residues which are not easily removed by hot air only, without damaging the color quality.

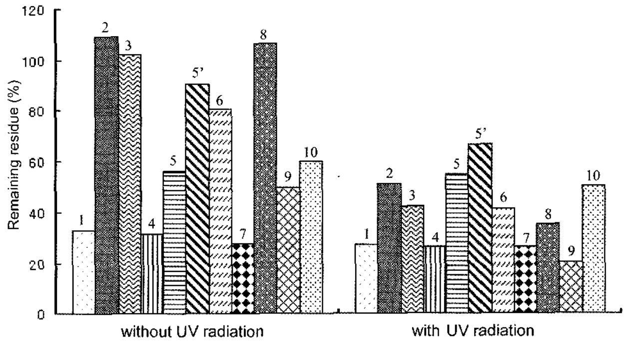

Fig. 1. Removal effects of pesticide residues in red pepper by concomitant UV radiation in hot-air drying. Red pepper was UV irradiated or not during drying after water washing. 1, chlorpyrifos; 2, cypermethrin; 3, deltamethrin; 4, diazinon; 5, alpha-endosulfan; 5', beta-endosulfan; 6, EPN; 7, fenitrothion; 8, fenvalerate; 9, methidathion; 10, profenofos.

Removal of organophosphorus residues during washing with oxidizing agents Red pepper fruits naturally contaminated with pesticides were washed with hydrogen peroxide (20%) or chlorine dioxide solution (50 mg/kg) and hot-air dried under UV irradiation. The residue ratios (%) after washing with either hydrogen peroxide solution or chlorine dioxide solution were 78.1, 68.3 in chlorpyrifos, 83.0, 69.5 in diazinon, 58.4, 58.8 in EPN, 71.6, 74.6 in fenitrothion, 68.1, 70.2 in methidathion, and 69.0, 71.9 in profenofos, respectively (Table 4). In addition, followed by hot-air drying under UV irradiation after such washing, residue ratios (%) of organophosphorus revealed 17.7, 19.6 in chlorpyrifos, 18.3, 21.9 in diazinon, 26.4, 29.3 in EPN, 21.4, 21.9 in fenitrothion, 16.4, 19.1 in methidathion, and 34.0, 34.4 in profenofos, respectively (Table 4). Comparing with the results from washing peppers without oxidizing agents mentioned above (Table 2), effects of oxidizing agents were negligible. Therefore, the use of oxidizing agents in washing of pepper fruits may not be recommended.

Comparison of artificially and naturally contaminated samples In experiments to obtain reduction factors of pesticides in processing, naturally contaminated crop samples are recommended in general. It is, however, quite

Table 3. Hunter color of red pepper after air-drying under UV radiation

Red pepper sample	Hunter value [1)]			a/b
	L	a	b	
Dried 26 hr	24.8	10.3	2.40	4.27
Dried 26 hr + UV 26 hr	24.6	9.96	2.19	4.56
Pr > T	0.34	0.46	0.07	

[1)]L, lightness; a, redness; b, yellowness.

Table 4. Reduction of pesticide residues in drying after washing red pepper with oxidizing agents

Red pepper sample[1)]	Chlorpyrifos		Diazinon		EPN		Fenitrothion		Methidathion		Profenofos	
	Conc.[2)] (mg/kg)	R[3)] (%)	Conc. (mg/kg)	R (%)	Conc.[2)] (mg/kg)	R[3)] (%)	Conc. (mg/kg)	R (%)	Conc. (mg/kg)	R (%)	Conc. (mg/kg)	R (%)
Raw	0.353±0.002	100	0.339±0.002	100	1.61±0.016	100	1.21±0.072	100	1.22±0.073	100	2.58±0.056	100
Washed with H_2O_2 sol'n	0.276±0.020	78.1	0.282±0.011	83.0	0.940±0.117	58.4	0.868±0.074	71.6	0.830±0.069	68.1	1.78±0.140	69.0
Dried 26 hr + UV 26 hr	0.328±0.000	17.7	0.326±0.002	18.3	2.23±0.014	26.4	1.36±0.003	21.4	1.05±0.006	16.4	4.60±0.016	34.0
Raw	0.353±0.002	100	0.339±0.002	100	1.61±0.016	100	1.21±0.072	100	1.22±0.073	100	2.58±0.056	100
Washed with ClO_2 sol'n	0.245±0.001	68.3	0.236±0.009	69.5	0.946±0.042	58.8	0.904±0.031	74.6	0.855±0.029	70.2	1.86±0.052	71.9
Dried 26 hr + UV 26 hr	0.363±0.001	19.6	0.390±0.001	21.9	2.47±0.025	29.3	1.39±0.011	21.9	1.22±0.011	19.1	4.66±0.028	34.4

1)Two groups of raw red pepper were washed with H_2O_2 solution (20%) or ClO_2 solution (50 mg/kg), respectively, followed by hot-air drying under UV rays.
2-3)Refer to Table 2.

Table 5. Comparison of results obtained from artificially or naturally contaminated red pepper

Red pepper sample	Remaining residue, %											
	Chlorpyrifos		Diazinon		EPN		Fenitrothion		Methidathion		Profenofos	
	A[1)]	B[2)]	A	B	A	B	A	B	A	B	A	B
Raw	100 0.406[3)]	100 0.309	100 0.367	100 0.588	100 1.99	100 1.26	100 0.692	100 2.08	100 0.756	100 1.83	100 1.62	100 1.38
Washed with water	106	90.9	109	98.6	82.7	85.8	77.7	93.9	103	90.5	83.3	93.2
Dried 26 hr	32.7	53.3	31.3	41.1	80.2	91.3	27.7	51.1	49.8	57.2	59.7	69.8
Dried 26 hr + UV 26 hr	27.0	18.4	26.5	18.8	41.2	40.3	26.4	33.1	20.2	25.1	50.2	46.8
Raw	100 0.353[3)]	100 0.309	100 0.339	100 0.588	100 1.61	100 1.26	100 1.21	100 2.08	100 1.22	100 1.83	100 2.58	100 1.15
Washed wiith ClO_2 soln	68.3	81.6	69.5	85.8	58.8	80.8	74.6	86.4	70.2	82.0	71.9	83.0
Dried 26 hr	-[4)]	20.4	-	19.5	-	62.9	-	39.3	-	47.0	-	51.9
Dried 26 hr + UV 26 hr	19.6	19.2	21.9	20.0	29.3	38.3	21.9	33.3	19.1	23.9	34.4	46.1

1,2)'A' represents naturally contaminated; "B" artificially contaminated red pepper.
3)Value in second row of raw sample means residue concentration in unit of mg/kg.
4)Hyphen means absence of data.

laborious and time-consuming to obtain such samples, and data accumulation by natural contamination is consequently quite limited. If it is proved that artificially and naturally contaminated crop samples show similar results in cooking or processing, there is no reason to require the use of field-contaminated crop samples in reduction studies.

Additional experiments were conducted using artificially contaminated samples to find if there is any difference with respect to water washing, hot-air drying, concomitant UV irradiation, and washing with ClO_2 oxidizing agent. The results for six organophosphorus pesticides showed a 10% difference in residue ratio between two different samples in most cases (Table 5). Exceptional cases showing more than 10% difference in residue ratio (%) are fenitrothion 77.7, 93.9 in water washing; chlorpyrifos 32.7, 53.3 and fenitrothion 27.7, 51.1 in hot-air drying; chlorpyrifos 68.3, 81.6, diazinon 69.5, 85.8, and EPN 58.8, 80.8 in chlorine dioxide washing.

It can be preferred by an experimenter to use artificially contaminated samples, because naturally contaminated samples in the field show very high variability in residue levels from sample to sample, as well as being difficult to obtain field samples. The major reason to recommend the use of naturally contaminated crops is based on the assumption that residues can be more easily removed from artificially contaminated samples, especially in the case of systemic pesticides. The residue ratios of tested pesticides, all non-systemic pesticides, were actually higher for artificially contaminated samples, showing more than a 10% difference. It is, therefore, recommended that artificially contaminated red pepper fruits can be used to accumulate reduction data for pesticides tested here.

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