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Research Article

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Phytotoxicity and Translocation of Residual Diquat Dibromide from Sandy Loam and Loam Soil to Following Crops Cultivating in the Soils

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Abstract

BACKGROUND: Diquat dibromide is a fast-acting non-selective herbicide and plant growth regulator. In this study, in order to understand the possibility of unintentional pesticide contamination in the following crops, the phytotoxicity and transition of diquat dibromide residue in soil into the following crops such as pepper, radish, lettuce and corn have been assessed through phytotoxicity trial and residual evaluation in the unintentional contamination of the higher residual diquat dibromide.

METHODS AND RESULTS: The pepper, radish, lettuce and corn were cultivated in the sandy soil and loam soil where the 35 mg/kg and 90 mg/kg diquat dibromide were applied, respectively. Mild growth inhibition symptoms were observed in radish, lettuce and corn crops at the 90 mg/kg- diquat dibromide treatment on the 30 day of cultivation. Diquat dibromide was analyzed using liquid chromatography QTRAP (LC-MS/MS). The recovery rates of diquat dibromide from soil and crop were determined within range from 89.1 to 116.4% with relative standard deviation less than 14.7%. Diquat dibromide residues in soil were found to be 23.90-30.22 and 69.59-82.57 mg/kg from the 35 mg/kg and 90 mg/kg of diquat dibromide-treated soil, respectively after 30 days of crop cultivation. This result implicates that diquat dibromide did not convert to metabolites and remained mostly in the soil, even though it was partially decomposed during crop cultivation. In addition, the diquat dibromide in pepper and radish that were grown for 47 days, and lettuce and corn that were cultivated for 30 days were detected to be

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0.01 mg/kg or less in the sandy loam and loam soil where the 90 mg/kg diquat dibromide was applied.

CONCLUSION(S): Diquat dibromide did not cause severe phytotoxicity in the following crops as well as it did not uptake and distribute to the following crops, even though it was considered to be residual in the soil.

Key words: Diquat dibromide, Following crops, Phytotoxicity, Soil residue, Transition

Introduction

Pesticides are essential agricultural materials that maintain high productivity and quality by controlling diseases, pests, and weeds in crop cultivation, and increase agricultural productivity by 45% when pesticides are properly used[1]. On the other hand, it has human and ecological toxicity, causing soil, water, air pollution and bioaccumulation by widespread use, and interfering with the essential functions of living organisms.

In general, weakness is a phenomenon that occurs when most compounds are applied to plants, which is in the form of chlorosis, necrosis, deformation, burns, and abnormal growth, which negatively affects plant growth and development. Likely, the antioxidant defense system is activated within the plant, along withreduced photosynthesis efficiency, reduced chlorophyll and protein synthesis, and reactive oxygen species generation. These weaknesses are investigated as indicators of germination rate, survival rate, length of roots and stems, root and stem ratio, vitality of seedlings, enzyme activity and so on[2, 3].

Diquat dibromide (1,1′-ethylene-2,2′-bipyridyldiylium dibromide, $C_{12}H_{12}N_2Br_2$, MW 344.04) is a fast-acting nonselective herbicide and plant growth regulator (Fig. 1). The diquat dibromide that was applied in soil is absorbed through the roots of the plant and transmitted to the phloem [4, 5]. It is also used as a drying agent for potato vines and seed crops such as cotton, alfalfa, and sunflowers, and flowering inhibitors for sugarcane.

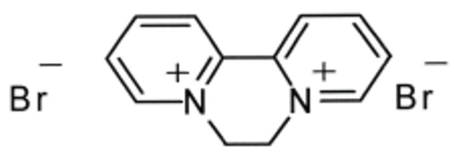

Fig. 1. Chemical structure of diquat dibromide dibromide (1,1′-ethylene-2,2′-bipyridyldiylium dibromide, $C_{12}H_{12}N_2Br_2$, MW 344.04).

This material is soluble in water, volatile, and has a low risk of leaching to the groundwater. It is highly residual in soil and highly toxic and irritate to mammals, however the effects of human health have not been identified[6].

According to peer review of the pesticide risk assessment of the active substance diquat dibromide [EPSA Journal 2015;13(11); 4308], it had been reported that it was very high persistence and the DT_{50} of diquat dibromide showed 598 days ~ >1,000 days under 20℃, pF 2 soil moisture and 40% MWHC. The compound is not approved by the European Union but is registered and used in many countries, including the United States, under the brand name Aquacide, Dextrone, Reglon, Reglox, Weedtrine- D, Aquakill, Vegetrole, Deiquat and Tag. In Korea, the effectiveness of diquat dibromide was confirmed in rye and triticale as a drying effect to promote crop yields[7, 8].

With the recent implementation of the positive list system (PLS) for all agricultural products, there is growing interest in pesticides that are highly residual in the soil as they can be absorbed into the following crop[9, 10]. In particular, diquat dibromide is a herbicide and highly residual in soil after soil treatment, which is worried to cause phytotoxicity and residue for following crops, but more detailed research is needed.

Therefore, the soil treatment of diquat dibromide, a highly residual herbicide, was carried out to assess the possibility of unintentional pesticide contamination through the phytotoxicity and residual evaluation of the absorption of pepper, radish, lettuce, and corn, what were the main and following crops of the upland fields.

Materials and Methods

Reagents and materials

The standard diquat dibromide (1,1′-ethylene-2,2′-bipyridyldiylium dibromide, $C_{12}H_{12}N_2Br_2$, MW 344.04, purity 97.5%) was purchased from Restek (Bellefonte, PA, USA). Distilled water, acetonitrile, dichloromethane, and methanol in HPLC grade were purchased from Tedia (USA). Hydrochloric acid, ammonium acetate, sodium chloride, and sodium sulfate anhydrous were purchased from Junsei Chemical (Japan). Analytical grade formic acid (purity ≥ 95%) and ammonium format (purity ≥ 99.995%) were obtained from Sigma Aldrich (St. Louis, MO, USA). Wagner pots (NF-5/φ174.6×φ160.4×197.5 mm) for cultivating the pepper, lettuce, radish and corn.

Table 1. Physicochemical properties of cultivated soils

Soils	Particle size distribution (%)			pH (1:5 H_2O)	OM[a)] (g/kg)	CEC[b)] (cmol/kg)	Texture
	Sand	Silt	Clay				
Eumseong	83.2	9.6	7.2	6.6	20	8.5	Sandy loam (SL)
Gongju	45.7	38.1	16.3	6.5	39	18	Loam (L)

[a)]Organic matter content, [b)]Cation exchange capacity

Test crops and pot preparation

Pepper (dokyacheongcheong variety), lettuce (top green), radish (Jeilcheongpung myeongwol), and corn (mibaek No. 2) were selected in this study. For the pot cultivation of crops, soil was collected from the topsoil layer (0-20 cm) in Eumseong-gun, Chungbuk province (soil A; sandy loam soil) and Gongju-si, Chungnam province (soil B; loam soil). The analysis of physicochemical properties for soils was conducted in compliance with the analytical method of the Rural Development Administration.

The physico-chemical properties of the test soil were as shown in Table 1. Diquat dibromide (Tech 40% up) was treated with 0, 35 and 90 mg/kg concentration, respectively, and distributed 2 kg to 1/5,000 a Wagner pot. Day 0 of soil samples were collected before distribution to pots after soil mix, and day 30 of soil samples was collected at the 30 days after transplant and used as analysis samples after storage at -20°C.

Phytotoxicity symptom

The phytotoxicity of crops caused by residual diquat dibromide in soil was carried out in accordance with the notice of Rural Development Administration (Pesticide registration standards with test methods for remedial effect and phytotoxicity). After sowing pepper, lettuce, radish, and corn seeds in diquat dibromide treated pot, the red pepper was examined 40 days later, and lettuce, radish, and corn were examined 30 days later for phytotoxicity. In this study, seed germination, shot length of early growth stage, and growth status of the growing crops were examined for phytotoxicity.

Preparation of a standard solution and calculation by a standard calibration curve

1,000 mg/L stock solution by dissolving 20.51 mg of diquat dibromide standard (97.5% purity) into 20 mL of H_2O for LC-MS was prepared. Take 1 mL of solution and dilute it to 10 mL of a 5 M NH_4Cl solution, and add 10 mL of a 12 M sodium hydroxide solution, and 1 mL of a 1% potassium ferricyanide solution to it. After mixing for 30 minutes, it distributed twice to 30 mL of dichloromethane and dehydrated with passing through the sodium sulfate anhydrous layer. Afterwards, diquat dibromidestandard solution of 200 mg/L was prepared by building a decompression concentration with a vacuum concentrator in a bath of 40°C or lower, and then reusing it as 5 mL of an untreated specimen. Dilute each of these as untreated specimens of soil to prepare a standard solution of 0.002, 0.005, 0.01, 0.02, 0.05, 0.1 and 0.2 mg/L, respectively, and inject this standard solution into the LC-MS/MS each to prepare a calibration curve. The lowest quantifiable amount of analyte (0.002 ng) was termed as LOQ that generated a signal ten times higher than the base line noise. Linearity is determined from the co-efficient of determinants (R^2 = 0.9995) obtained from the seven-points calibration curve (y = 1,245,228x+1,627). The LOQ for the analytical method of the diquat dibromide were 0.01 mg/kg under the LC-MS/MS conditions.

LC–MS/MS conditions for detection of diquat dibromide in soil and crop

An ultra-performance liquid chromatograph (Waters AQUITY UHPLC H Class, Waters, Hertfordshire, UK) coupled with a mass spectrometer (AB SCIEX QTRAP 3500 mass spectrometer, SCIEX, Redwood, CA, USA) was employed for detection and quantification of diquat dibromide. The MS/MS was carried out in multi -reaction monitoring mode (MRM) with positive electron spray ionization. For MRM method development a syringe pump was equipped with the MS/MS system for standard infusion and optimization of compound ionization. The chromatographic separation was carried out on a Hilic-Si column (50 x 2.1 mm, 2.7 μm). The column oven temperature was maintained at 45°C. A binary solvent system composed of 50 mM ammonium formate and 0.5% formic acid in water (A), and 50 mM ammonium formate and 0.5% formic acid

Table 2. Multiple reaction monitoring (MRM) conditions of diquat dibromide

Compound	Precursor ion	Product ion	DP [a)] (V)	EP [b)] (V)	CE [c)] (V)	CXP [d)] (V)
Diquat dibromide	183.3	130.1	100	10	50	12
		157.2	150	10	35	3

[a)]Declustering potential, [b)]Enterance potential energy, [c)]Collision energy, [d)]Collision cell exit potential

in 75% ACN and 25% water (B). The gradient mobile phase started with 100% B which was reduced to 65% B at 4 min. At 4.1 min it was suddenly increased to 100% B and remains constant until 7 min. The column flow was maintained at 0.60 mL/min with the injection volume of 10 μL. The ion source temperature was 500°C and the spray voltage was 5,500 V. Nitrogen was used as nebulizer, curtain, and heating gas. The optimized MS/MS parameter is shown in Table 2.

Residual analysis of diquat dibromide in soil and crop

Each 5 g of homogenized soils and crops (pepper, radish, lettuce and corn) was taken in a 50 mL Teflon centrifuge tube to which a 15 mL of 0.5% formic acid in acetonitrile and 5 mL of 0.5% formic acid in water was added and extract with a Geno Grinder (SPEX Sample Prep., USA) for 2 min at 1,200 rpm. The extracts were then centrifuged at 3,500 rpm for 5 min. and transferred the whole solution to another centrifuge tube. The remaining samples were re-extracted with 10 mL of 0.5% formic acid in acetonitrile and transfer and combine the solution to the centrifuge tube following centrifugation. The mixed solutions were vortexed for 10 s and evaporated the organic layer through a gentle N_2 stream at 40°C. The remaining aqueous solution was adjusted to reconstructing until 6.25 mL with 0.5% formic acid in water and additional 6.25 mL of 0.5% formic acid in acetonitrile was added. The total 12.5 mL of mixed solution was vortexed for 10 sec. prior to purification. An aliquot of 2 mL of extract was loaded to a HLB (500 mg) LP cartridges (Oasis, Waters, Milford, MA, USA) that was previously conditioned with 6 mL of 0.5% formic acid in water and 6 mL of 0.5% formic acid in acetonitrile with an solid phase extraction (SFE) manifold under a reduced pressure. The loaded extract was then eluted with a 1.6 mL of 0.5% formic acid in acetonitrile. The eluents were combined in a 15 mL Teflon tube and added with 0.4 mL of 0.5% formic acid in acetonitrile followed by vortexing. One mL was transferred to a 2 mL plastic vial followed by addition of 10 μL of 5000 mM ammonium formate. Diquat dibromide was analyzed using liquid chromatography QTRAP (LC-MS/MS) [11]. The detection of diquat dibromide in soils and crops has been carried out as same as residue analytical method for livestock Using UPLC-MS/MS[12].

Result and Discussion

The soil decomposition rate (half-life) of diquat dibromide has been known at a geometric mean of 2,345 days among 598 ~ 6174 days of four soil types in compliance with the efsa [EPSA Journal 2015;13 (11); 4308, 127 pp]. The throughput per area was 1.2 kg a.i./ha (soil depth of 10 cm, bulk density of 1.0 kg/L) in compliance with the application method. Assuming 1.2 mg/kg, the cumulative equilibrium concentration was calculated by assuming that the number of treatments per a year was three application. If the geometric mean half-life of 2,345 days is adopted, it will reach at 35.0 mg/kg after 50 years, adopting the longest half- life of 6,174 days, reaching 89.6 mg/kg after 200 years.

Therefore, in order to study the possibility of unintentional pesticide contamination in the following crops, the concentration of diquat dibromide for phytotoxicity and translocation trial were recommended to the 35 mg/kg as reference amount and 90 mg/kg as double dose based on soil accumulation concentration of diquat dibromide by DT_{90} from soil dissipation studies [EPSA Journal 2015;13(11); 4308, p57]. The two concentration of diquat dibromide in relation to soil accumulation for phytotoxicity and transition trials were determined at level of 35 mg/kg as reference amount and 90 mg/kg.

Phytotoxicity is damage caused by all hazardous materials including organic compounds and trace metals to plant growth, externally in the form of leaves chlorosis and necrosis, leaf deformation and stunting, and internally in the form of photosynthesis, chlorophyll destruction and reactive oxygen species. This can be seen as an indicator of plant growth, such as germination

Table 3. Recoveries and limits of detection for diquat dibromide in the soils

Soils	Fortification[a)] (mg/kg)	Recovery (%)				LOQ[b)] (mg/kg)	MDA[c)] (ng)
		1	2	3	Mean[a)], RSD		
Eumseong (Sandy loam)	35	89.1	91.6	90.9	90.5, 1.42	0.01	0.002
	90	114.1	116.4	88.3	106.3, 14.7		
Gongju (loam)	35	94.1	90.0	93.5	100.4, 11.5	0.01	0.002
	90	113.8	108.7	95.2	105.9, 9.1		

[a)]Concentration of diquat dibromide in relation to soil accumulation for phytotoxicity and transition trials, [b)]Mean values of three time repetitions with realtive standard deviation (RSD, %), [c)]Limits of quantification, [d)]Minimum detection amount

rate, root and stem length and so on[3].

The recovery rate and detection limit of diquat dibromide represented in the Table 3. The fortified concentration of diquat dibromide were decided to 35 mg/kg and 90 mg/kg in relation to soil accumulation to do phytotoxicity and translocation trials of diquat dibromide from sandy loam and loam soil to following crops cultivating in the soils. The recovery rate of diquat dibromide showed at the range level 88.3% ~ 116.4%, and 90.0% ~ 113.8% in the sandy loam soil (Eumseong) and in the loam soil (Gongju), respectively, with relative standard deviation less than 14.7%. (Table 3). It was within the acceptable range of reclamation (70 ~ 120%) commonly presented by Codex (CXG 90-2017). The limit of quantification for diquat dibromide was 0.01 mg/kg as well as the minimum detection quantity of it was 0.002 ng on the LC-MS/MS condition. There was no significant difference between the germination rate investigated on 30 and 40 days for each crop and the treatment concentration of the shoot length investigated on 40 days after sowing (Table 4). In addition, on the 33 daysof transplanting peppers, lettuce, radish, and corn grown in soil treated with diquat dibromide, mild growth inhibition in lettuce, radish, and corn was observed at 90 mg/kg of diquat dibromide treated soil, but recovered on the 40 days. In the case of red peppers, it was difficult to investigate the germination rate due to insufficient germination until the 33 days after the diquat dibromide treatment.

The phytotoxicity shown in crops grown in diquat dibromide treated soil were found to be phytotoxicity from 0 to 1 grade for pesticide registration and rarely recognizable (Fig. 2, 3). This phytotoxicity test was carried out in accordance with the Korean RDA provisions of the criteria (3-3-1-5) for review of efficacy and phytotoxicity test report of pesticides (February 1, 2012). As for degree of phytotoxicity about crops by the pesticides, the phytotoxicity should be less than 0 for the reference amount and less than 1 for the double dose to register a pesticide in Korea. The residual amount of diquat dibromide in soil without crop, after treating 35 mg/kg and 90 mg/kg of diquat dibromide were 30.43 and 86.55 mg/kg in the sandy loam soil with RSD (relative standard deviation) of 0.75% respectively. The 27.25, 26.70, 28.35 and 26.88 mg/kg of diquat dibromide were persisted in the sandy loam soil grown 30 days with peppers, radish, lettuce, and corn respectively, indicating a decrease in soil residual during crop cultivation. In case of 90 mg/kg diquat dibromide treated sandy loam soil, residue of diquat dibromide in the soil peppers, radish, lettuce, and corn were 71.69, 74.65, 70.80 and 76.25 mg/kg, respectively, which were lower than the residual amount of crop- free soil at day 0 (Table 5). The 24.85, 25.38, 23.90 and 27.28 mg/kg of diquat dibromidewere persisted in the loam soil grown 30 days with peppers, radish, lettuce, and corn respectively, indicating a decrease in soil residual during crop cultivation. In case of 90 mg/kg diquat dibromide treated loam soil, 76.23, 75.94, 72.68 and 76.82 mg/kg of diquat dibromide were detected in the soil where pepper, radish, lettuce and corn were cultivated, respectively (Table 5). These results implicated there are no differences between the soil texture (Sandy loam and Loam) with respect of degradation for diquat dibromide. No significant differences between crops and soil series have been identified in the study. These results signified that diquat dibromide was not converted easily to other metabolites since it has been confirmed to remain in the soil for long time. Basically, when most organic compounds are introduced into the soil, they were dissolved by soil water, run off or leached, and rapidly disappear due to absorption by plants, and decomposition by microorganisms and light. It was mentioned that the residue of diquat dibromidepersists in the soil for many

Table 4. The seed germination and seedling growth on four plants by diquat dibromide

Soils	Plant	Treated amounts of diquat dibromide (mg/kg)	Seed germination (%)		Shoot length±SD[a)] (cm)
			30 days	40 days	
Eumseong (Sandy loam)	Pepper	Control	-	100	2.2±0.2
		35	-	98	2.1±0.3
		90	-	98	2.2±1.5
	Radish	Control	76	-	16.1±2.0
		35	73	-	16.0±2.5
		90	71	-	15.7±2.5
	Lettuce	Control	96	-	9.3±2.7
		35	93	-	9.2±1.2
		90	91	-	8.8±1.7
	Corn	Control	83	-	25.1±2.0
		35	82	-	25.0±0.5
		90	81	-	24.7±0.7
Gongju (Loam)	Pepper	Control	-	99	2.2±0.3
		35	-	99	2.1±0.4
		90	-	98	2.1±0.5
	Radish	Control	82	-	16.0±0.2
		35	79	-	15.9±0.9
		90	78	-	15.6±1.2
	Lettuce	Control	94	-	9.1±2.2
		35	91	-	8.7±2.0
		90	92	-	8.7±2.1
	Corn	Control	84	-	25.5±0.7
		35	81	-	25.4±0.4
		90	83	-	25.2±1.6

[a)]Mean values of three time repetitions

years because it is strongly adsorbed with clay particles and organic matter in the soil when it comes into contact with the soil as a residue[13-15].

The residual amount of diquat dibromide among the crops collected on day 47 for pepper and radish and day 30 for lettuce and corn were grown at the 90 mg/kg of diquat dibromide treated soil, were found to be less than 0.01 mg/kg, respectively, which was the under critical level for the Positive List System (PLS) (Table 6). In general, the PLS means a system in which pesticides registered for each crop must be used within a certain standard, and 0.01 mg/kg is applied uniformly to pesticides without residual tolerance standards. This result implicates that diquat dibromide didnot convert to metabolites and remained mostly in the soil, even though it was partially decomposed during crop cultivation. In addition, the diquat dibromide in pepper and radish that were grown for 47 days, and lettuce and corn that were cultivated for 30 days were detected to be 0.01 mg/kg or less in the sandy loam and loam soil that 90 mg/kg diquat dibromide applied. As a result of these studies, diquat dibromide has long-term residual properties in the soil, but it was judged that the possibility of exceeding 0.01 mg/kg levels in peppers, radish, lettuce and corn was low due to the lower absorption probability of diquat dibromide. The diquat dibromide was rapidly absorbed by the leaves and usually quickly kills the plant before transition to other parts of the plant when it was applied to the leaves of plants. However, diquat dibromide treated in soil showed limited uptake and transfer into plants. One third of diquat dibromide sold worldwide was used as a herbicide, and 90% of it was used as a herbicide in Western Europe, Australia and Japan. The application

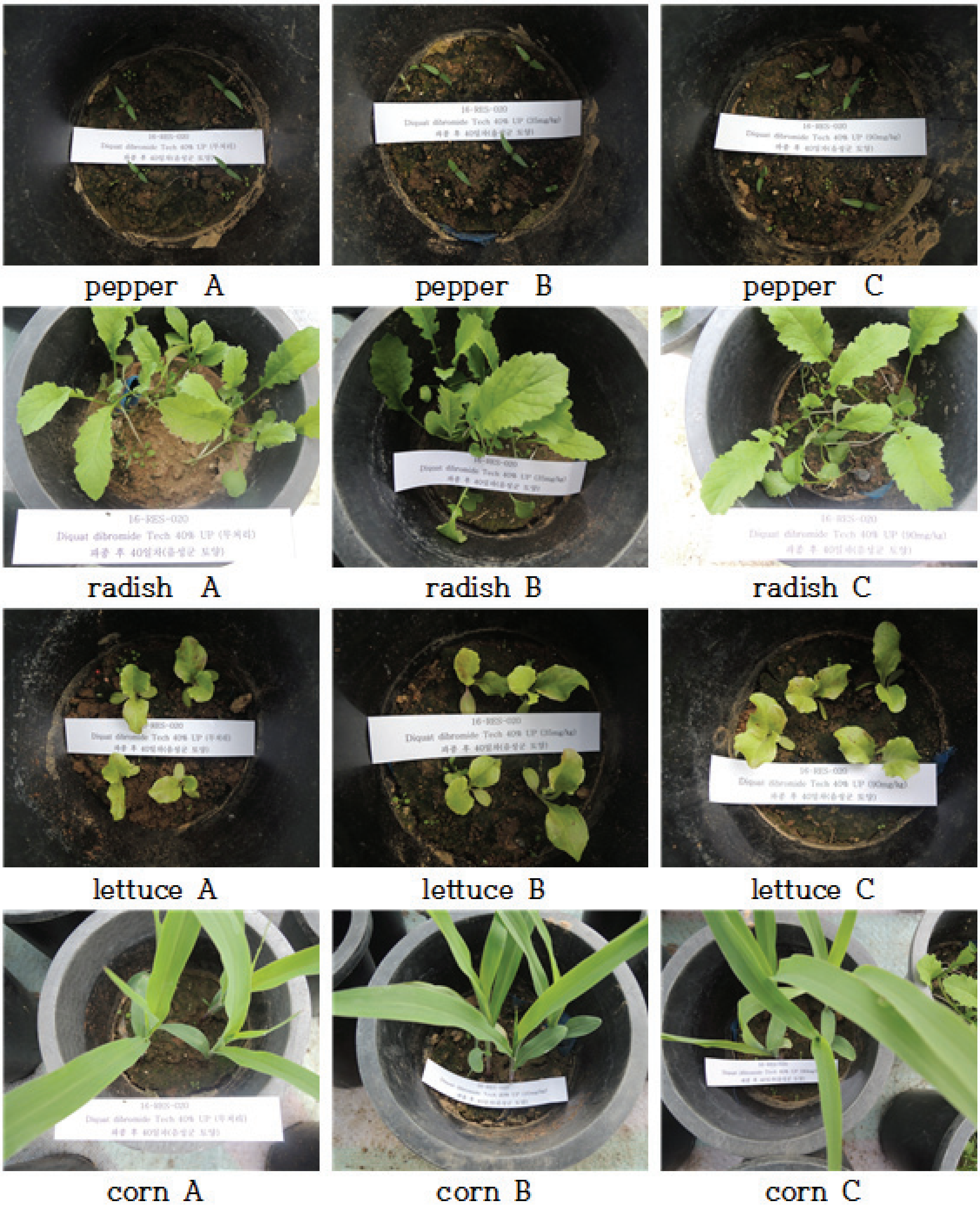

Fig. 2. Phytotoxic symptoms by diquat dibromide on the growth of pepper, radish, lettuce and corn under Eumseong (sandy loam) conditions, as measured on 40 days after treated at the 0 (A), 35 (B) and 90 (C) mg/kg concentration.

method for diquat dibromide was used to apply soil treatment before planting or before crop emergence. In this case, there is no particular problem, but if necessary, weeds arecontrolled by direct spraying and interline spraying after the appearance of the initial crop. In this case, the crop may die or burn severely by contact with young seedlings. A small amount of application method residue was detected at a maximum of 0.07 mg/kg in oats and corn 7-8 weeks after spraying, and in some root vegetables, such as carrots, 2 weeks after spraying, but in most crops, the minimum amount detected in edible parts at harvest 1-4 months after spraying. It was detected to be less than 0.01 mg/kg. These study results were similar as

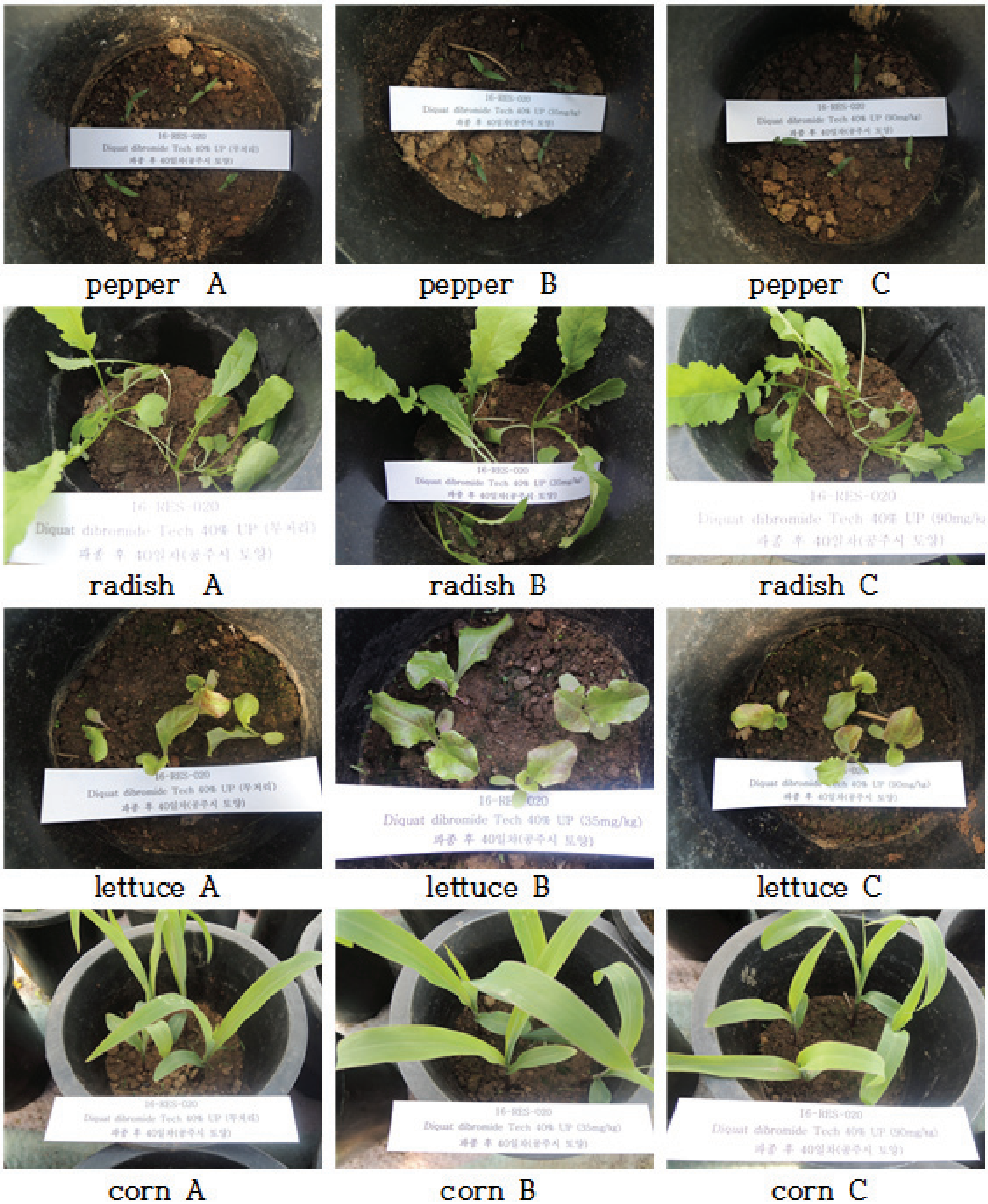

Fig. 3. Phytotoxic symptoms by diquat dibromide on the growth of pepper, radish, lettuce and corn under Gongju (loam) conditions, as measured on 40 days after treated at the 0 (A), 35 (B) and 90 (C) mg/kg concentration.

Edward (1977) and Kennedy (1986) had reported in diquat residue summary: "Residues in fruit and vegetable crops following pre- and post-emergence treatment with diquat for weed control (1961-1976, ICI report No. TMJ1500A)" and "Diquat: Residue in maize from a trial carried out during 1985 in Spain (ICI Plant Protection Division Report No. M4194B)".

In conclusion, diquat dibromide did not cause severe phytotoxicity in the following crops as well as it did not uptake and distribute to the following crops, even though it was considered to be residual in the soil. Therefore we might understand that diquat dibromide was unlikely to violate 0.01 mg/kg that was applied uniformly to unregistered pesticide criteria in

Table 5. Residual amount of diquat dibromide in soil after 30 days cultivation for various crops at the 35 mg/kg and 90 mg/kg-treated soil

Soils	Diquat dibromide[a)] (mg/kg)	Crops	Cultivation days	Residue of diquat dibromide (mg/kg)				LOQ[d)] (mg/kg)
				1	2	3	Mean[b)], RSD[c)]	
Eumseong (Sandy loam)	35	No crop	0	30.22	29.81	31.26	30.43, 0.75	0.01
		Pepper	30	27.42	26.80	27.53	27.25, 0.39	0.01
		Radish	30	27.51	25.67	26.91	26.70, 0.94	0.01
		Lettuce	30	28.27	27.90	28.88	28.35, 0.49	0.01
		Corn	30	27.22	25.63	27.80	26.88, 1.12	0.01
	90	No crop	0	77.38	78.75	79.52	78.55, 1.08	0.01
		Pepper	30	69.59	73.17	72.30	71.69, 1.87	0.01
		Radish	30	75.25	72.70	74.65	74.20, 1.33	0.01
		Lettuce	30	69.75	72.14	70.80	70.90, 1.20	0.01
		Corn	30	72.54	79.16	76.25	75.98, 3.32	0.01
Gongju (Loam)	35	No crop	0	26.38	26.52	27.28	26.73, 0.48	0.01
		Pepper	30	24.42	24.31	24.85	24.53, 0.29	0.01
		Radish	30	24.55	23.40	25.38	24.44, 0.99	0.01
		Lettuce	30	24.12	23.12	23.90	23.71, 0.53	0.01
		Corn	30	26.38	26.52	27.28	26.73, 0.48	0.01
	90	No crop	0	80.78	80.72	82.57	81.36, 1.05	0.01
		Pepper	30	73.48	75.21	76.23	74.97, 1.39	0.01
		Radish	30	75.49	77.10	75.94	76.18, 0.83	0.01
		Lettuce	30	71.99	70.72	72.68	71.80, 0.99	0.01
		Corn	30	76.17	75.26	76.82	74.69, 0.78	0.01

[a)]Treated amount of diquat dibromide in soil (mg/kg), [b)]Mean values of three time repetitions, [c)]Realtive standard deviation (RSD, %), [d)]Limits of quantification (mg/kg)

Table 6. Residual amount of diquat dibromide in various crops cultivated at the 90 mg/kg of diquat dibromide treated soil

Soil	Crops	Cultivation days	Residue of diquat dibromide (mg/kg)				LOQ (mg/kg)
			1	2	3	Mean[a)], RSD(%)	
Emseong (Sandy loam)	Pepper	47	< 0.01	< 0.01	< 0.01	< 0.01, 0	0.01
	Radish	47	< 0.01	< 0.01	< 0.01	< 0.01,.0	0.01
	Lettuce	30	< 0.01	< 0.01	< 0.01	< 0.01,.0	0.01
	Corn	30	< 0.01	< 0.01	< 0.01	< 0.01, 0	0.01
Gongju (Loam)	Pepper	47	< 0.01	< 0.01	< 0.01	< 0.01, 0	0.01
	Radish	47	< 0.01	< 0.01	< 0.01	< 0.01, 0	0.01
	Lettuce	30	< 0.01	< 0.01	< 0.01	< 0.01, 0	0.01
	Corn	30	< 0.01	< 0.01	< 0.01	< 0.01, 0	0.01

[a)]Mean values of three time repetitions with realtive standard deviation (RSD, %)

compliance with the PLS due to low absorption potential by pepper, radish, lettuce and corn even though an application of an agrichemical was considered to be residual in the soil.

Note

The authors declare no conflict of interest.

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