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Researches using radio-labelled Herbicide in Korea

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ABSTRACT: The research results using radio-labelled herbicides performed by Korean researchers were reviewed. All the research works were used ^{14}C-labelled chemicals and generally carried out to know the behavior of herbicides in soils and plants. The degradation, mineralization and bound-residues formation are the major concerning area in soil studies, and uptake, translocation, metabolism, selectivity and resistance are in plant studies. Also few papers covered synthesis, formulation and animal metabolism.

Key Words: radio-labelled herbicide, korean researchers

INTRODUCTION

As it mentioned in the former review for insecticides[1], the radioactive isotope (RI) techniques in pesticide researches showed three advantages in detection of small quantity, distinguishment between pesticides and the constituents of plants or soil, and identification of metabolites. In herbicide research, RI technique is useful and efficient in study on the mode of action and in plant-soil environment. In mode of action study, RI technique was introduced to various areas of researches. For example, the adsorption onto surface of plants, the absorption by plants, chemical transformations by plant metabolism, reaction with primary action sites, the secondary metabolic changes and symptoms of herbicidal effects could be included. Also, the bioaccumulation of herbicides and metabolites in soil to plants was the staple concerning area in plant- soil environment.[2] Except for them, another research area with radio-labelled herbicides was the behavior in soil environment. Kyung and Lee (1993)[3] already reviewed the effectiveness using lysimeter study with ^{14}C-pesticides. They suggested that the study of movement and leaching of chemicals, adsorption and absorption, formation of bound residues, metabolism in soil and volatilization into air could be performed using lysimeter and radio-labelled pesticides.

In this paper, the research works with radio-labelled herbicides which carried out by the Korean researchers were reviewed. Researches were divided into 3 categories including plant, soil and miscellaneous. Rice plant, weeds and other crops were the objects of plant studies, and also degradation and metabolism, bound residues were main interesting point in soil studies.

1. Plant

In plant studies researches could be divided into 3 groups such as absorption and translocation, metabolism, and selectivity and resistance. Also they were experimented mainly with rice plant, and the other crops and weeds.

1.1 Absorption and Translocation

1.1.1 Rice plant

Lee *et al*. (1989)[4] studied on the bioavailability of soil-aged residues of ^{14}C-bentazon to rice plants. They found that the uptake of ^{14}C-bentazon residues by rice plants decreased remarkably with increasing the aging period of 3 months, and much differences were between 3-month and 6-month aging (Table 1).

Kwon and Pyon (1993)[5] studied with ^{14}C-bensulfuron

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Table 1. Plant uptake of radiocarbon from soil containing ^{14}C-bentazon or bentazon residues during the growth period of 42 days.

Treatment	Description	Uptake (%)[a,b]					
		Root		Shoot		Total	
T-1-1	Freshly treated	25.8	A	12.52	A	38.32	A
T-1-2		29.36	A	12.67	A	42.03	A
T-2-1	3-Month-aged	2.54	B	1.55	B	4.08	B
T-2-2		4.3	B	1.36	B	5.66	B
T-3-1	6-Month-aged	1.34	B	1.15	B	2.48	B
T-3-2		1.9	B	1.14	B	3.04	B

^{14}C-Activity in soil at day 0 = 100%

[a] All data represent averages of 2 replicates in each treatment. Each replicate involves 3 samples.

[b] Numbers followed by the same letter within a column are not significantly different at the 5% levels using Duncan's multiple range test.

(Lee *et al*, 1989)[4]

to know the selectivity mode of action between rice and *cyperus serotinus* Rottb. The absorption rate increased by time course from 3 to 48 hrs after ^{14}C-bensulfuron (10^{-6}M) by root dipping method. Also, they mentioned that the root was shown higher absorption than shoot. And the translocation rate from root to shoot increased within 1 day after treatment and maintained upto 7 days.

Pyon *et al*. (1994)[6] also studied with ^{14}C-dithiopyr to find the selectivity mechanism between rice and barnyard grass. They compared by different sites of absorption such as root alone and roots + basal shoot (3cm) with the absorption rate by time course. In root alone treatment, they found very low translocation rate from root to shoot as 3% at 48 hrs after treatment.

Kang and Pyon (1995)[7] experimented a similar research with ^{14}C-bensulfron. They compared with two rice cultivars and three paddy weeds at different temperatures for absorption, translocation and metabolism of ^{14}C-bensulfron.

The absorption of ^{14}C-bensulfron (10^{-6} M) by rice cultivars was increased by time course at high temperature (30/25: day/night), and cv. Samgang showed higher absorption than cv. Sampung after 24 hrs of treatment (Fig. 1).

And Lee (1995)[8] investigated for selectivity of ^{14}C-oxyfluorfen in 21 different rice cultivars. From the results he found that cv. Hwachung showed the highest resistant and cv. Chuchung was the most sensitive cultivars. The absorption amounts of ^{14}C-oxyfluorfen by shoot were almost 2 times higher in cv. Chuchung than cv. Hwachung from 60 mins until 120 mins after treatment (Fig. 2).

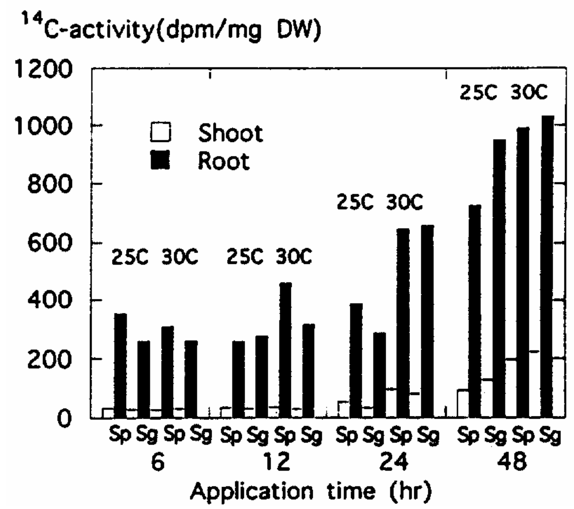

Fig. 1. Concentration of ^{14}C-activity of two rice cultivars, Sangpung (SP) and Samgang (SG) grown in nutrient solution treated with ^{14}C-bensulfuron at different temperatures. (Kang and Pyon, 1995)[7]

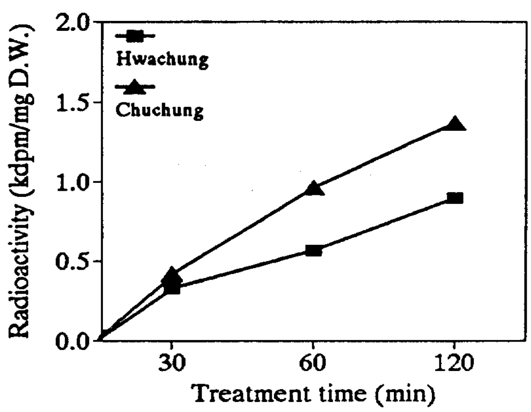

Fig. 2. Absorption of ^{14}C-oxyfluorfen by shoots of the plants.

(Lee, 1995)[8]

Lee and Kyung (1995)[9] treated with ^{14}C-TCAB to soil and grown rice plants. They compared fresh-treated, 3-month aged and 6-month aged soil with ^{14}C-TCAB. At 42 days after growth of rice plants the uptake rate in roots/shoots were 0.72/0.02%, 0.92/0.02% and 0.97/0.02% for fresh treated, 3-month aged and 6-month aged soil, respectively.

Lee *et al*. (1996)[10] applied ^{14}C-bentazon onto two lysimeters (0.25 m^3) and rice plants were grown over four consecutive years. They reported that the ^{14}C-activities translocated into rice straw in the first year ranged from 1.4 to 2.2% of the originally applied amount and reduced remarkably in the second year, and those into brown rice grain were below the MRL of FAO/WHO, 0.1 mg·kg^{-1}.

Kyung *et al*. (1997)[11] applied the ^{14}C-quinclorac onto the lysimeter and the rice plants were grown on the lysimeter. At 22 days after transplanting rice the herbicide was applied and cultivated by conventional method for 88 days until harvest. They found that the average ^{14}C-activities detected in straw, ear without grains, and chaff after harvest amounted to 0.41, 0.10 and 0.19 mg·kg^{-1} of quiclorac equivalents, respectively, and residue amount of 0.15 mg·kg^{-1} distributed in brown rice grain was shown far less than the MRL of Japan, 0.5 mg·kg^{-1}.

Lee *et al*. (1997)[12] also experimented with ^{14}C-cinosulfuron in rice plants grown lysimeter over two consecutive years. They measured the ^{14}C-radioactivities in straw, ear without grains, chaff and brown rice grains after harvest each year. And they found the ^{14}C-radio activity in straw to be 47-116 times higher than that in brown rice grains in the first year, whereas 38-51 times higher in the second year.

Also, Lee *et al*. (2002)[13] studied with ^{14}C-cinoculfuron in lysimeter planted with rice over four consecutive years, and they compared with the total ^{14}C-radioactivity translocated to rice plants in the third and the fourth year. The results from this study are shown in Table 3

1.1.2 Other Crops.

Lee *et al*. (1987)[14] applied ^{14}C-bentazon to two different soils and grown the maize plants. The chemical treated 3 different methods with freshly added (T-O), 105days pre-incubation (T-1) and soil after extraction with water and 0.1M $CaCl_2$ solution of 105 days pre-incubation soil (T-2).

At 21 days after growth of maize plants absorbed from 36.0 to 42.0% of ^{14}C-activity in T-O, from 8.2 to 14.2% in T-1, and from 1.8 to 2.3% in T-2, respectively, and the distribution ratio of ^{14}C-activity in root showed much higher than that of shoot (Table 4).

Also Lee *et al*. (1988)[15] prepared two different soils with ^{14}C-bentazon, the freshly added and the bound residue soil. The bound residue soil was prepared by pre-incubation for 6 months and planted rice, and

Table 3. Amounts (%) of ^{14}C-radioactivity absorbed and translocated in each part of rice plants grown on the lysimeter soil in the third and fourth year after a single ^{14}C cinosulfuron treatment.

Part of rice plant	Lysimeter I		Lysimeter II	
	Third year	Fourth year	Third year	Fourth year
Straw	0.581±0.006[a]	0.537±0.008	0.872±0.061	0.753±0.025
	(0.059±0.01)[b]	(0.047±0.001)	(0.089±0.006)	(0.049±0.002)
Ear without rice grains	0.06±0.000	0.004±0.000	0.009±0.000	0.005±0.000
	(0.014±0.001)	(0.010±0.000)	(0.019±0.001)	(0.015±0.000)
Chaff	0.057±0.001	0.041±0.001	0.084±0.005	0.052±0.001
	(0.04±0.000)	(0.023±0.001)	(0.034±0.002)	(0.034±0.001)
Brown rice grain	0.048±0.003	0.021±0.001	0.057±0.003	0.026±0.001
	(0.005±0.000)	(0.003±0.00)	(0.006±0.000)	(0.004±0.000)
Total	0.692	0.603	1.022	0.836

Radioactivity applied = 100%

a) Figures represent mean ± standard deviation of triplicates.

b) Concentration of cinosulfuron equivalent (μg/g) calculated on the basis of the specific ^{14}C-radioactivity (1.66 MBq/mg) of the cinosulfuron applied.

(Lee *et al*, 2002)[13]

Table 4. Uptake of ^{14}C-activity and its distribution between shoots and roots of maize plants after growing for 21 days in soils containing fresh (T-0), aged (T-1), and bound (T-2)

Soil	Treatment	^{14}C-Activity in plants (%)	Distribution (%)*	
			Shoot	Root
Parabraun	T-0	36.0	5.9	94.1
	T-1	14.2	5.7	94.3
	T-2	2.3	9.6	90.4
Korean	T-0	42.8	2.7	97.3
	T-1	8.2	9.3	90.7
	T-2	1.8	5.0	95.0

^{14}C-Activity in soil on 0 day = 100%.
* Radioactivity in the plants = 100%

(Lee *et al*, 1987)[14]

extracted with distilled water by centrifuged at 13,000 rpm for 15 mins repeatedly until no-radio activity in water.

The amounts of ^{14}C-bentazon and its metabolites absorbed by soybean and radish were 45.41 and 21.48% in freshly treated soil, whereas those were 3.92 and 1.23% in bound soil, respectively. And translocation ratios of radioactivity from root to shoot were much higher in radish than in soybean.

Hwang and Park (1994)[16] examined the absorption and translocation of ^{14}C-alachlor in soybean and Chinese cabbage seedlings. 10 day-old seedlings were immersed into the solution containing ^{14}C-alachlor ($3.93x10^{-6}$ M), and measured ^{14}C-activity until 48 hrs by time course.

As shown in Table 5, soybean seedling absorbed more ^{14}C-activity than Chinese cabbage, and the distribution was higher in root than in shoot.

Park *et al*. (1999)[17] were studied with ^{14}C-primisulfuron to know the synergistic effect of phytotoxicity in combination of insecticide and sulfonylurea herbicide. They compared with two different corn cultivars such as pioneer 3751 (susceptible to imidazolinone herbicide) and Pioneer 3751IR (resistant to imidazolinone).

Also they treated PBO (piperonyl butoxide), the synergist for herbicidal activity. The absorbtion results obtained summarized in Table 6.

Table 5. Distribution of radioactivity in soybean and Chinese cabbage seedling.

	Exposed time (hr)	Radioactivity (dpm/mg dry wt)		
		Absorption	Root	Shoot
Soybean seedling	3	148	459	2
	6	332	1,172	3
	12	876	2,632	19
	24	1,136	3,728	30
	48	1,246	4,186	36
Chinese cabbage seedling	3	78	267	16
	6	98	319	26
	12	168	463	66
	24	186	511	99
	48	344	424	319

(Hwang and Park, 1994)[16]

Table 6. Effect of terbufos and PBO on absorption of ^{14}C-primisulfuron in various corn cultivars.

Treatment	Pioneer 3751 IR			Pioneer 3751		
	Control	Terbufos 1.8kg/ha	PBO 2kg/ha	Control	Terbufos 1.8kg/ha	PBO 2kg/ha
Time after treatment (hour)			%			
24	20.9	28.4	41.0	21.8	28.1	39.2
48	61.4	77.8	81.3	51.9	74.5	75.3
96	77.1	80.7	83.0	82.0	77.6	79.3

(Park *et al*, 1999)[17]

Table 7. Translocation of Foliar-Applied ^{14}C Oxyfluorfen in wheat and Barley

		Radioactivity recovered (% of applied)		
	Time after		Shoots other than	
Species	application(h)	Treated leaf	treated leaf	Root
Wheat	6	97.86±0.54	0.12±0.08	0.13±0.05
	24	96.69±0.41	1.13±0.41	0.34±0.15
	72	96.70±0.63	1.32±0.46	0.41±0.18
	120	97.87±0.75	1.11±0.21	0.20±0.04
Barely	6	95.61±0.74	1.40±0.41	0.72±0.03
	24	95.11±0.59	2.19±0.66	0.99±0.48
	72	95.22±0.24	3.14±0.60	0.74±0.37
	120	96.45±1.16	2.44±0.94	0.84±0.51

* Note. Values are the means ± SE of triplicate experiments.

(Chun *et al*, 2001)[18]

Chun *et al*. (2001)[18] compared with wheat, relatively tolerant to diphenyl ether herbicide, and barley, relatively susceptible to ^{14}C-oxyfluorfen about absorption and translocation. Absorption of ^{14}C-oxyfluorfen appeared to be similar in the leaves of both species and little translocation of the herbicide out of the treated leaf was observed in either species, but slightly more in wheat leaves (Table 7).

1.1.3 weeds

Kwon and Pyon (1993)[5] compared the absorption and translocation characteristics of ^{14}C-bensulfuron in rice and *cyperus serotinus* Rottb Rice absorbed a greater amount of bensulfuron than *cyperus serotinus*, and the translocation rates of ^{14}C-bensulfuron from root to shoot were much faster in *cyperous serotinus* than rice plants (Fig 3).

Pyon *et al*. (1994)[6] treated with ^{14}C-dithiopyr to barnyard grass (*Echinochloa Crusgalli Beauv.*) in Kasugai nutrient solution by root application at 2~4 leaf stage. The absorption and distribution of ^{14}C-activity in barnyard grass were measured by time course until 48 hrs after treatment (Table 8).

Hwang and Park (1994)[16] also studied barnyard grass seedling with ^{14}C-alachlor and measured the distribution of radioactivity by time course. The absorption was increased until 24 hrs after treatment, and then slightly increased upto 48 hrs. And the pesticide translocated to the shoots during the experiment (Table 9).

Kang and Pyon (1995)[7] compared the absorption and translocation of ^{14}C-bensulfuron with three different

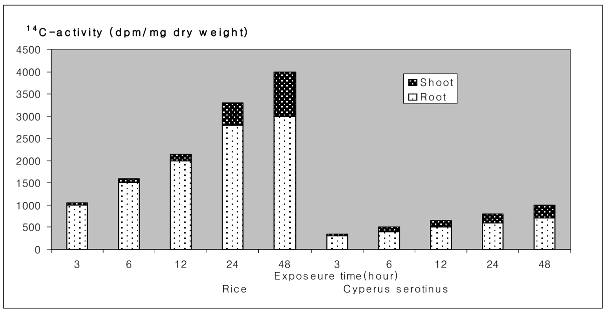

Fig. 3. Absorption of ^{14}C-bensulfuron (10^{-6} M) by roots of rice and *Cyperus serotinus* at the two leaf stage over time. (Kwon and Pyon, 1993)[5]

Table 8. Dry weight of rice plants and barnyard grass at 2 leaf stage as affected by dithiopyr treatment under nutrient solution culture

Concentration	Dry weight (mg/plant)			
	Rice		Barnyard grass	
	Shoot	Root	Shoot	Root
Control	59.6a[z]	26.2a	65.1a	52.0a
1×10^{-8}	56.0a	18.1b	59.6a	31.9b
5×10^{-8}	44.4b	11.4c	52.9b	7.8c
1×10^{-7}	33.1c	10.3c	45.5d	4.9c
5×10^{-7}	29.6c	8.1d	20.4c	3.5c
1×10^{-6}	27.8c	7.4d	19.6d	3.6c

[z] Means within a column followed by the same letter are not significantly different at the 5% level by the Duncan's multiple range tests.

(Pyon *et al*, 1994)[6]

weeds such as *Cyperus serotinus* (CS), *Sagaittaria pygmaea* (SP) and *Echinochloa crusgalli* (EC) in 10^{-6} M of ^{14}C-bensulfuron solution by time course. Also, they compared two different growing temperatures at 25/20℃ and 30/25℃ (day/night), As shown in Fig 4, the treated weeds absorbed more chemical by time course. At 25/20℃ the absorption was the order of SP > CS > EC. And CS showed more absorption at lower temperature than higher temperature (Fig. 4).

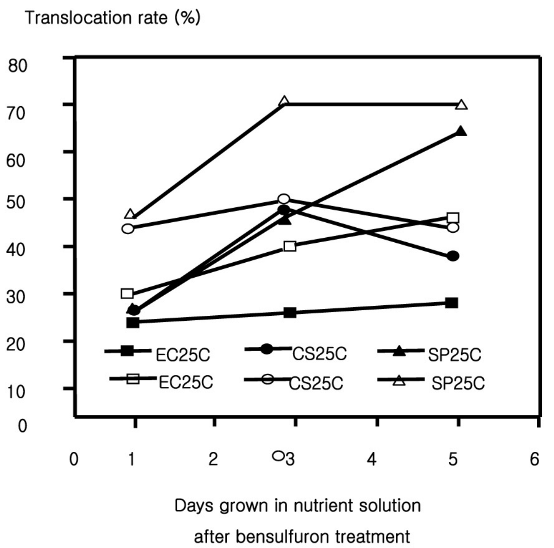

Fig. 4. Translocation rate of ^{14}C-bensulfuron (10^{-6} M) from root to shoot *Echinochloacrus-galli* (EC), *Sagittaria pygnaea* (SP), and *Cyperus serotinus* (CS) grown in nutrient solution after 24 hrs absorption of ^{14}C-bensulfuron at different temperatures.

(Kang and Pyon, 1995)[7]

Kim *et al*. (2005)[19] investigated the absorption and translocation of the systemic herbicide, ^{14}C-glyphosate applied in combination with the contact herbicide, carfentrazone-ethyl. They compared with two different weeds such as *commelina communis* L. and *Echinochloa crus-galli* (L.) P. Beauv. The absorption of ^{14}C-glyphosate was rapidly increased upto 4 days after application (DAA), and carfentrazone-ethyl treatment (30g ai ha^{-1}) was not greatly affected in mixture or in 1 day before ^{14}C-glyhposate application. The translocation of ^{14}C-glyphosate was generally affected by carfentrazone-ethyl treatment, and the ^{14}C-activities at 10 DDA were different between two weeds. The relatively higher or amount of ^{14}C-glyphosate was translocated to other untreated plant parts in *C. communis*, whereas in *E. crus-galli* it remained more in the treated leaf (Table 10).

Table 9. Distribution of radioactivity in barnyard grass seedling.

Exposed	Radioactivity (dpm/mg dry wt)		
	Absorption	Root	Shoot
3	70	149	12
6	159	306	24
12	365	702	47
24	605	1,286	119
48	617	1,095	247

(Hwang and Park, 1994)[16]

Table 10. Translocation of ^{14}C *in C.communis* and *E. crus-galli* at 10 days after application with ^{14}C glyphosate alone or 0 and 1 day following application of 30 g ai ha^{-1} carrentrazone-ethyl (Carf.)

Treatment	^{14}C-Recovered (%)		
	Treated leaf	Untreated leaf	Root
		C. communis	
^{14}C-glyphosate	13.2a	44.4a	7.9b
Carf+^{14}C-glyphosate, 0-day sequence	11.6a	47.6a	19.6a
Carf+^{14}C-glyphosate, 1-day sequence	14.4a	32.9b	6.6b
		E.crus-galli	
^{14}C-glyphosate	26.1b	7.5b	5.1b
Carf+^{14}C-glyphosate, 0-day sequence	41.4a	12.8b	4.0b
Carf+^{14}C-glyphosate, 1-day sequence	36.5a	20.7a	10.9a

(Kim *et al*, 2005)[19]

Kim *et al*. (2005)[20] selected paraquat-resistant and paraquat susceptible biotype *of Conyza canadensis* (L.) Cronq, and compared their absorption, translocation and binding constant to paraquat. The amount of epicuticular wax were about 10% higher in resistant than in susceptible, also the amounts of cuticle were 1.5 times higher in resistant and the penetrating amounts of ^{14}C-paraquat into cuticle were 38.4% and 32.3% in resistant and susceptible biotype, respectively. Nevertheless, those differences might not be explained correct reason for 7.8 times higher chlorophyll loss in susceptible than in resistant.

1.2 Metabolism

Park and Hwang (1987)[21] found the phytotoxicity to be inversely proportional to the peptide contents of the test plants, soybean, Chinese cabbage and barnyard grass. They concluded that conjugation reaction involving glutathiones and xenobiotic alachlor, a typical phase II reaction, contribute to the selectivity of alachlor.

Table 11 represented the distribution and recovery of radioactivity in plants. The recovered radioactivities ranged from 73 to 88%, and among them about 95% of radioactivity remained in water-soluble fraction regarding as the metabolite fraction within 24 hrs after treatment.

Hwang and Park (1994)[16] treated with ^{14}C-alachlor to soybean, Chinese cabbage and barnyard grass seedlings and investigated the metabolites at 10 mins and 3 hrs after application. The results are shown in Fig. 5. At 10 mins after application, the soybean resistant to alachlor showed the lowest radioactivities and the barnyard grass, susceptible to alachlor, showed the highest radio activity. Also the unchanged alachlor were 3, 5 and 36% in soybean, Chinese cabbage and barnyard grass, respectively. And they also found that 1, 45 and 40% of glutathione conjugate and 60, 28 and 8% of cysteine conjugates, respectively (Fig. 5).

Kang and Pyon (1995)[7] treated with ^{14}C-bensulfuron and analyzed it's metabolites in two cultivars of rice and three different weeds. The amounts of bensulfuron were higher in cv. Sangpung, susceptible one than cv.

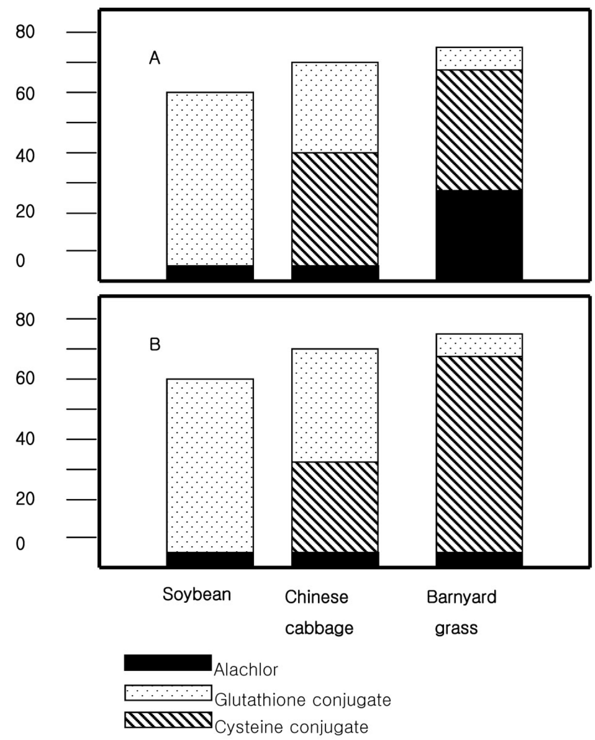

Fig. 5. Percent distribution of radioactivity into alachlor, glutathione conjugate, and cysteine conjugate in seedlings. Roots of seedlings were immerged into nutrient solution containing ^{14}Calachlor for 3 hrs. Seedlings were transferred to alachlor-free nutrient solution and harvested 10 min (A), and 3 hrs (B) later.

(Hwang and Park, 1994)[16]

Table 11. Conversion of alachlor to water soluble metabolites in intact plants[a)]

Plant	Radioactivity (%)		Recovery (%)
	Water-soluble fraction	Hexane-soluble fraction	
Soybean[b)]	95.4	4.6	73
Chinese cabbage[b)]	96.7	3.3	88
Barnyard grass[b)]	96.0	4.0	87

a) Incubated for 24hrs

b) Labelled alachlor (0.2$\mu\ell$ in 2.0$\mu\ell$ of acetonitrile) was applied on hypocotyls (Soybean and Chinese cabbage) and leaf blades (barnyard grass)

(Park and Hwang, 1987)[21]

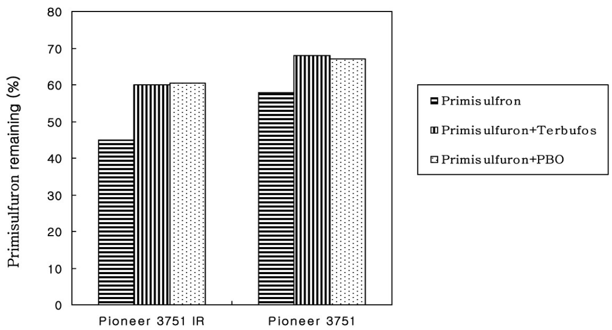

Fig. 6. Effect of terbufos on metabolism of ^{14}C-primisulfuron in corn cultivars.

(Park *et. al*, 1999)[17]

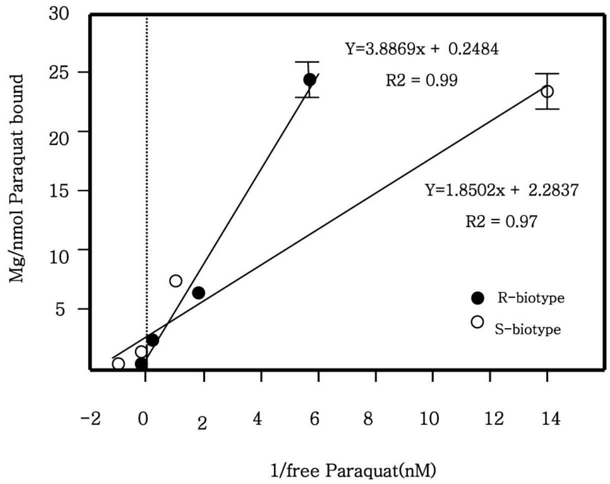

Fig. 7. Binding constant of ^{14}C-paraquat to thylakoid membrane purified from *Conyza Canadensis* biotypes (R, Resistant; S, Susceptible).

(Kim *et al*, 2005)[20]

Samgang, resistant one. Also, they mentioned that amount of bensulfuron in shoot showed higher in *Sagittaria pygamaea* (Sp) and *cyperus serotinuns* (Cs) than *Echinochloa traw-galli* and the ratio of sulfonamide, a metabolite of bensulfuron, were in Sp and Cs at 24 hrs after treatment both in shoot and root.

Park *et al*. (1999)[17] compared two different varieties of corn, cv. Pioneer 3751IR (R) and cv. Pioneer 3751 (S) based on the resistant ability to imidazolinone herbicides. They treated with ^{14}C-primisulfuron in 4 hrs and after then left another 4 hrs in nutrient solution, and measured the radioactivity. The result is shown in Fig. 6.

The resistant variety, pioneer 3751IR remained only 45.54% of total radioactivity, whereas 61.58% in susceptible pioneer 3751. Also, the similar results were shown in the treatment of terbufos and piperonylbutoxide.

Chun *et al*. (2001)[18] compared the metabolic differences of foliar applied ^{14}C-oxyfluorfen in wheat and barley. They mentioned that they had no special metabolites from leaf extracts of either species.

1.3 Selectivity and Resistance

Pyon *et al*. (1994)[6] carried out the nutrient culture study with ^{14}C-dithiopyr in rice and barnyard grass. They suggested the selectivity of dithiopyr between rice and barnyard grass might contributed to the absorption and translocation of dithiopyr in each plant.

Hwang and Park (1994)[16] examined with ^{14}C-alachlor for the selectivity among soybean, Chinese cabbage and barnyard grass. They concluded that both absorption and translocation contribute undoubtedly to the selectivity by influencing the active internal concentration of alachlor in plants. However, the most important primary function was the rate of glutathione conjugation, which detoxifies alachlor and plays an important role in selectivity.

Lee (1995)[8] also mentioned that the absorption difference among rice cultivars and barnyard grass was the main reason of selectivity with ^{14}C-oxyfluorfen.

Chun *et al*. (1997)[22] reported that the resistance of aqueous extract from the paraquat- resistant plant, *R. glutinosa* was less absorption and translocation in one hand, and might be chemically transformed or physically bound to a substance present in the aqueous extract in other hand. Also Chun *et al* (1997)[23] studied with *R. glutionsa*, extremely tolerant to paraquat at the level of leaf disk and chloroplast with ^{14}C-paraquat. They concluded that the tolerance of *R. glutinosa* to paraquat might mostly due to the consequence of paraquat metabolism outside of the chloroplast, although paraquat binding to the cell wall is also likely to confer the tolerance to some extent.

Kim *et al*. (2005)[20] compared paraquat-resistant and paraquat-suceptible biotype of *Conyza conadensis* (L.) Cronq. to investigate its resistant mechanism with ^{14}C-paraquat. They observed that the binding constant of paraquat to the cell wall and thylakoid membrane were 7.4 and 16.9-fold, respectively, higher in R-biotype than in S-biotype. So, they suggested that the resistance mechanism of *C. canadensis* biotype is due partly to high binding affinity of paraquat to the cell wall and thylakoid membrane (Fig. 7).

2. Soil

2.1 Degradation and Metabolism

Lee and Fournier (1978)[24] studied the fate of 3,4-DCA and TCAB in 4-different French soils with uniformly ^{14}C- ring labelled 3,4- DCA and TCAB. They observed the breakdown of ^{14}C-3,4-DCA into ^{14}C-CO_2 was 6.5% at 1.5 mg·kg^{-1} dose on 187 days of incubation with alkaline soil (pH 7.9). Meanwhile, in organic acid soil (pH 5.5) showed 4.9% and 4.24% at 1.5 and 94 mg·kg^{-1} dose, respectively. Similarly, they mentioned the $^{14}CO_2$ evolution from ^{14}C-TCAB in soil. Their results are shown in Table 12.

From Table 12 the $^{14}CO_2$ evolution amount was much lower by time course. Among tested soils argillaceous soil showed the highest evolution rate.

Lee *et al*. (1982)[25] studied microbial metabolism of ^{14}C-propachlor in soil suspension. They incubated aerobically the uniformly ring-labelled ^{14}C-propachlor in soil suspension at 27℃ for 20 days. They observed the radioactivity absorbed in soil was 20% and 5.6% of them was methanol-extractable and remaining 14.4% was in soil. Also, they identified the metabolites by GC/MS including *N*-isopropylamiline, *N*-isopropylacetanilide, *N*-(1-hydroxy-isopropyl) acetanilide and, *N*-isopropyl-2-acetoxy acetanilide.

Lee *et al*. (1989)[4] experimented with ^{14}C-bentazon aged in soil for 3 and 6 months. And they found that 6.1 and 14.8% of the original radioactivity evolved as $^{14}CO_2$ during 3 and 6 months, respectively.

Lee *et al*. (1991)[22] added ^{14}C-TCAB to MM_2 medium as a sole carbon source, and isolated 5 different micro organisms. Also they found that no $^{14}CO_2$ was evolved from ^{14}C-TCAB for 6 week-incubation with shaking at 30℃. And, they observed free Cl^- in culture medium as an evidence of dechlorination, and identified two metabolites with m/z 250 and m/z 278 by GC/MS.

Lee and Kyung (1991)[26] treated with ^{14}C-TCAB into soil and observed the $^{14}CO_2$ evolution and volatilization loss by the soil aging period. The amounts of $^{14}CO_2$ evolution and volatilization were negligible, but increased by time course. 3 month after treatment, the $^{14}CO_2$ and volatilization were 0.06 and 0.03% of the original radioactivity, and also 0.14 and 0.12% at 15 months after treatment, respectively.

Kyung *et al*. (1997)[11] applied ^{14}C-quinclorac to a lysimeter simulating the rice paddy conditions. They observed that the amount of $^{14}CO_2$ evolved from the surface of the lysimeter soil was 0.71% of the original radioactivity upto the 14th week after treatment, while that of volatile ^{14}C lost from the soil surface exhibited the background level.

Kwon and Lee (1997)[27] studied with ^{14}C-imazapyr in eight types of soils with the different physico-chemical properties. They investigated the mineralization to $^{14}CO_2$ and adsorption characteristics. They found that the amounts of $^{14}CO_2$ evolved from 8 types of soils ranged from 1.5 to 4.9% of the original applied ^{14}C-activities, also the lower pH and higher organic matter soils showed low $^{14}CO_2$ evolution than higher pH and lower organic matter soils. They also observed that the organic matter content was the most influential factor in imazapyr adsorption on soil.

Kwak *et al*. (2000)[28] treated with ^{14}C-bifenox in silty loam and sandy loam soils, and incubated under anaerobic conditions at 25℃ for 180 days. They found that the relative amounts of $^{14}CO_2$ were 1.97 and 0.9% of applied ^{14}C in silty loam and sandy loam soils, respectively. Also, they observed some

Table 12. $^{14}CO_2$ evolution from ^{14}C-TCAB in soils (Percentage of the initial radioactivity liberated in the form of $^{14}CO_2$)

Incubation day	Soils	Organic acid soil (pH5.5)		Alkaline soil (pH7.9)		Organic neutral soil (pH6.9)		Argillaceous oil (pH7.3)	
		1	2	1	2	1	2	1	2
24		0.042	0.035	0.048	0.042	0.011	0.028	0.076	0.055
52		0.06	0.054	0.087	0.075	0.018	0.041	0.10	0.070
84		0.08	0.070	0.13	0.12	0.028	0.053	0.14	0.11
128		0.10		0.17		0.042		0.18	
169		0.11		0.19		0.051		0.20	

(Lee and Fournier, 1978)[24]

metabolites such as nitrofen, 5-(2,4-dichlorophenoxy) -2-nitrobenzoate, 2,4-dichlorophenoxy aniline and methyl 5-(2,4-dichlorophenoxy) anthranilate by GC/MS analysis.

Lee *et al*. (2002)[13] treated ^{14}C-cinosulfuron in two lysimeters with a clay loam soil, and rice plants were grown for four consecutive years. And they found that 2.03~3.58% of the original radioactivity evolved to $^{14}CO_2$ and from 2.43 to 2.99% of ^{14}C found in leachates.

Kim *et al*. (2003)[29] studied with new sulfonyl urea herbicide LGC-42153, [*N*-((4,6-dimethoxy pyrimidin-2-yl) -aminocarbonyl]-2-(1-methoxy acetoxy-2-fluoropropyl)-3-pyridinsufonamide], under an aerobic condition in a loamy soil for 120 days. They observed that the material balance ranged from 90.7 to 101.5% of the applied ^{14}C-radioactivity. And they identified the three metabolites such as *N*-((4.6-dimethoxypyrimidin -2-yl) amino carbonyl)-2-(1-hydroxy-2-fluoropronyl)-3-pyridinesulfonamide (M1), 2-(1-hydroxy-2-fluoropropyl) -3-pyridine sulfonamide, (M2) and 4.6-dimethoxy-2-aminopyrimidine (M3) (Fig. 8).

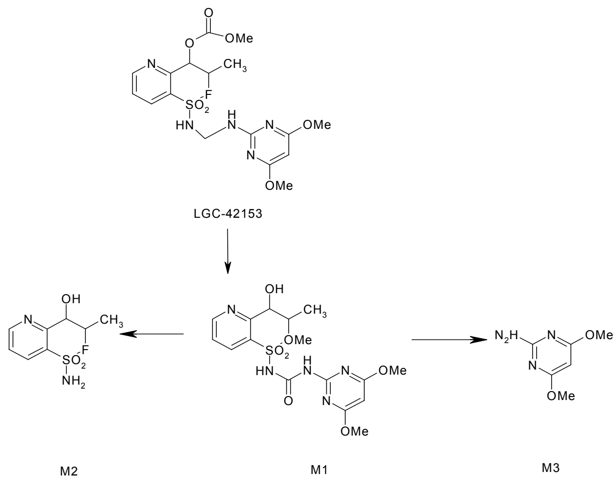

Fig. 8. Propose metabolic pathway of LGC-42153 in soil under aerobic conditions.

(Kim *et al*, 2003)[29]

Kim *et al*. (2003)[30] also studied with LGC-42153 under flooded soil condition. They suggested that the half-life of LGC-42513 was approximately 3.0 days. And the mass balance over 120 days ranged from 94.0 to 104.2% of applied radiocarbon and no significant amount of volatiles or $^{14}CO_2$ were observed. The major metabolic reaction was the cleavage of the carboxyl ester bond, similar as the aerobic condition, to give 1-(4,6-dimethoxypyrimidin-2-yl)-3-[2-(1-hydroxy -2-fluoropropyl)pyridine-3-ulfonyl] urea.

2.2 Bound residue

Lee and Fournier (1978)[24] treated with ^{14}C-3,4-DCA and ^{14}C-TCAB onto various French soils and observed 47.7% of initial radioactivity bound to soil in organic acid soil and 29.49% in alkaline soil at 1.5mg·kg^{-1} dose rate, and 38.4 and 20.30% in 94 mg·kg^{-1} dose rate, respectively.

Lee *et al*. (1987)[31] applied ^{14}C-bentazon to two different soils and found the $^{14}CO_2$ evolution rate and non-extractable radioactivity in soils. They also compared with the two different application doses, 5.51 and 25.05 mg·kg^{-1} soil. The results are shown in Table 13.

Lee *et al*. (1989)[4] studied with ^{14}C-bentazon and found the amount of non-extractable bound residue increased more than 70% of the original radioactivity with the aging period upto 3 months. Also, they mentioned that the ^{14}C-activity associated with the organic matter fractions were humin > fulvic acid > humic acid, in turn.

Lee and Kyung (1991)[26] experimented with ^{14}C-TCAB, and looked over the formation of bound residue by aging. The amounts of non-extractable bound residue of ^{14}C-TCAB increased gradually from 7.55% in

Table 13. Comparison of the non-extractable bound residues of ^{14}C-Bentazon formed in soil after an aerobic incubation for 105 days at 23±1℃ and 50% of the maximum water-holding capacity of the soil in the dark.

Soil	Initial conc. (mg/kg soil)	Mineralization to $^{14}CO_2$ (%)	^{14}C-activity extracted with water (%)	^{14}C-activity remaining in soil (%)	Recovery (%)
ParaBraun	5.51	8.89	41.71	46.55	97.24
	25.05	3.44	71.76	25.94	101.14
Korean	5.51	8.79	30.95	56.69	96.43
	25.05	3.18	54.8	43.99	101.97

^{14}C-activity applied = 100% (Lee *et al*, 1987)[31]

Table 14. Change in the characteristics of the non-extractable[1)] ^{14}C residues formed after applied to soil samples as ^{14}C-TCAB and aged for different periods of time.

Aging period (month)	Non-extractable bound residue (%)[1)]	Fluvic acid acid (%)	Humic acid acid (%)	Humin (%)
3	7.55	0.53	2.84	4.18
6	11.96	0.98	4.94	6.04
9	13.72	1.27	4.37	8.08
12	16.27	2.01	5.68	8.58
15	19.32	2.11	6.59	10.62

1) Non-extractable bound residue = Fulvic acid + Humic acid + Humin (Lee and Kyung, 1991)[26)]

Table 15. Characteristic of ^{14}C TCAB residues in soil as affected by residue age and the presence/ absence of growing rice plants

Treatment	Distribution of bound residues[a] (%)			
	Total bound	Furvic acid	Humic acid	Humic
Freshly treated	1.42	0.31	0.27	0.84
3-Month aged	8.93	1.70	1.20	6.03
6-Month aged	15.88	0.62	3.01	12.25
Freshly treated + 42-day planting	19.50	0.73	5.07	13.70
3-Month aged + 42-day planting	21.50	0.59	5.48	15.43
6-Month aged + 42-day planting	21.91	0.71	6.78	14.42

a) Fulvic acid + Humic acid + humin = percent total bound; in parentheses, fulvic acid + humic acid + humin = 100%. Numbers followed by the same letter within a column are not significantly different at the 5% levels using Duncan's multiple range test. (Lee and Kyung, 1995)[9)]

3-month aging to 19.32% in 15-month aging (Table 14).

Lee and Kyung (1995)[9)] examined the bioavailability of soil residues of ^{14}C-TCAB to rice. Rice plants were grown on soil treated with ^{14}C-TCAB after aging for 0, 3, and 6 months. The bound residues in soil gradually increased by time course, and the rice planting plots showed higher bound residues. The results are summarized in Table 15.

Lee *et al*. (1996)[10)] applied ^{14}C-bentazon onto two of 0.25m^3 lysimeters by conventional methods and rice plants were grown over four consecutive years. The ^{14}C-activities remaining in the 0~10, 10~20, 20~30, and 30~40 cm soil layers were approximately 14.07, 16.45, 27.53 and 21.28% (lysimeter 1) and 3.47, 10.06, 10.45 and 9.34% (lysimeter 2), of the originally applied ^{14}C, respectively, after 2 years.

Kyung *et al*. (1997)[11)] mentioned that 95% of original ^{14}C-activity of ^{14}C-quinlorac remained in the 30 cm layer from the surface after rice harvest under lysimeter cultivation method.

Lee *et al*. (1997)[12)] treated with ^{14}C-cinosulfuron onto rice grown in lysimeters by the conventional method for two consecutive years. They found that the ^{14}C-radioactivity distributed down to 30 cm soil was from 89 to 92% of the original amount in the first year and from 60 to 71% in the second year.

Kwak *et al*. (2000)[28)] treated with ^{14}C-bifenox in silty loam and sandy loam soil at the rate of 2.1 mg·kg^{-1}, and incubated under anaerobic conditions at 25℃ for 180 days. They found that the non-extractable residues gradually increased in both soils, and the higher organic matter soil and sandy loam soil showed higher content of non-extractable ^{14}C-activity than the lower organic matter soil and silty loam soil. After 180 days the amounts of non-extractable ^{14}C-activity were 60.72 and 79.12% of the original radioactivity in silty loam and sandy loam soil, respectively.

Lee *et al*. (2002)[13)] applied ^{14}C-cinosulfuron onto two rice-growing lysimeters and cultivated rice plants over four consecutive years, and found that the distribution of ^{14}C-activity in soil profile. The total bound residues were 56.71% and 57.52% in lysimeter 1 and 2, respectively. The distribution of ^{14}C-activity in soil showed in Fig 9.

Kim *et al*. (2003)[30)] examined with ^{14}C-LGC-42153 in flooded soil condition, and found that the

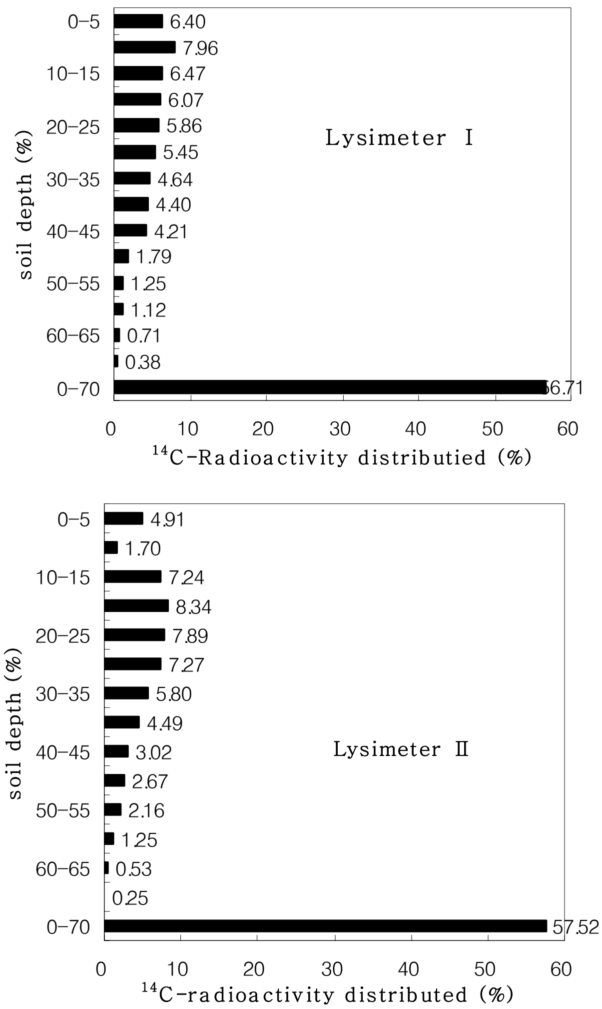

Fig. 9. ^{14}C-Radioactivity (%) remaining in different layers of the two lysimeter soil after rice cultivation for four consecutive years (radioactivity applied = 100%)
(Lee *et al*, 2002)[13]

non-extractable radiocarbon reached about 11-14% of applied radioactivity at 120 days after treatment. Also they observed that the most of the non-extractable radiocarbon was in the humic and fulvic acid fractions with time course.

Kim *et al*. (2003)[29] also studied with ^{14}C-LGC-42153 under aerobic soil condition for 120 days in a loamy soil. They mentioned that the non-extractable ^{14}C-residue reached from 14.4 to 30.5% of applied concentration at 120 days after treatment, and radioactivity was distributed mostly in the humic and fulvic acid fractions.

2.3 Others

Not many research works were performed except the studies for plants and soils.

Lee *et al*. (1977)[32] published about a new synthetic method for ^{14}C-ring labeled TCAB. They started from ^{14}C-3,4-dichloroaniline (3,4-DCA) avoiding the reduction of 3,4-dichloronitrobenzene. ^{14}C-TCAB was produced by aerial oxidation of ^{14}C-3,4-DCA catalyzed by CuCl with pyridine as solvent at 60℃ for 5-12 hrs, giving 80.2% yield.

Oh (1990)[33] prepared controlled release formulation with ^{14}C-butachlor with alginate-kaolin matrix. He investigated that the influence of kaolin addition on the formulation characteristics and release profiles under a closed dark and an opened sunlight condition. He observed that the release rate from alginate formulation was slower than that of the commercial granule, impregnated in zeolite. Also the more content of kaolin showed the slowest release among alginate formulas.

Liu *et al*. (2001)[34] studied pharmacokinetics of

Table 16. Tissue-to-blood ratio after a single oral administration ^{14}Cpyribenzoxom (1000 mg·kg^{-1}) to male Sprague - Dawley rats.

	Tissue to blood ratio (±SD)[a]									
Sample	6h		12h		24h		48h		168h	
Stomach	5.66	(±1.69)	2.08	(±0.99)	0.09	(±0.03)	0.04	(±0.02)	0.05	(±0.01)
GIT	61.80	(±2.80)	19.00	(±3.10)	1.30	(±0.18)	0.60	(±0.15)	0.29	(±0.10)
Cecum	197.00	(±66.00)	55.00	(±6.80)	3.20	(±1.09)	0.87	(±0.21)	0.46	(±0.11)
Liver	0.72	(±0.12)	1.14	(±0.21)	1.58	(±0.15)	1.34	(±0.25)	0.64	(±0.10)
Kidney	0.40	(±0.05)	0.35	(±0.01)	0.30	(±0.03)	0.34	(±0.03)	0.27	(±0.02)
Lung	0.26	(±0.02)	0.28	(±0.02)	0.30	(±0.02)	0.25	(±0.03)	0.32	(±0.02)
Heart	0.26	(±0.05)	0.23	(±0.01)	0.28	(±0.01)	0.31	(±0.03)	0.30	(±0.02)
Testis	0.20	(±0.02)	0.19	(±0.02)	0.25	(±0.00)	0.22	(±0.03)	0.25	(±0.02)
Spleen	0.12	(±0.02)	0.10	(±0.01)	0.13	(±0.02)	0.12	(±0.01)	0.12	(±0.01)
Thymus	0.09	(±0.02)	0.09	(±0.01)	0.11	(±0.03)	0.10	(±0.02)	0.11	(±0.01)
Brain	0.02	(±0.00)	0.02	(±0.00)	0.02	(±0.00)	0.02	(±0.00)	0.02	(±0.00)

[a)] Tissue to blood ratio were calculated for each animal (n = 3) (Liu *et al*, 2001)[34]

^{14}C-pyribenzoxim in rat. They treated a dose of 1000 mg·kg^{-1} to male Sprague-Dawley rats by oral administration. They found the majority of radioactivity in feces (88.6%) by 168 hrs after treatment, and total radioactivity in tissue decreased from 96% at 6 h to 0.4% at 168 hrs. They also found that the highest concentration of radioactivity among the tissues was observed in liver, while the lowest residues were found in brain. The tissue-to-blood ratio of ^{14}C-pyribenzoxim is shown in Table 16.

CONCLUSION

It was reviewed the research results using radio labelled herbicides performed by Korean researchers during 1977-2005. The papers were divided into 3 categories such as plants, soil and miscellaneous. Total 31 research papers were reviewed as 20 in plants, 16 in soil and 3 in miscellaneous. Some papers covered both plants and soil area. The major interesting points of plant studies were the absorption, translocation and selectivity mechanism between rice plants and weeds. And the resistance mechanism of paraquat was the another favorable object among some researchers. Also uptake and distribution of herbicide into plants from soil was the another concerning point of research. In soil studies, almost of research works with rice carried out under flooded condition, and many of them were experimented for a few consecutive years. The degradation, metabolism and mineralization of chemicals in soil were investigated and the formation of bound residues, too. Although it is not many research papers except plant and soil area, synthesis, formulation and metabolism in animals were introduced. These miscellaneous researches also are valuable subjects to study. In future, we expect that the more researches using radio labelled compounds would be studied with many new scientists in this field.

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