

Original Article

## Dissipation kinetics of glyphosate in tea and tea-field under northwestern mid-hill conditions of India

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A study was conducted to determine the dissipation of glyphosate applied at three dose levels in tea crop in mid hill conditions of northwest Himalaya, India in two consecutive seasons. More than 65% of the initial residues in the soil were found to have dissipated within 30 days following application to the tea irrespective of the dose. Glyphosate persisted in the soil for up to 30, 45 and 60 days at application doses of 0.5, 1.0 and 2.0 kg/ha, respectively. Glyphosate residues in the tea leaves were detected for up to 15 days with all three treatments. Half lives of glyphosate ranged from 5.80 to 19.10 days in the soil of the tea fields and 5.82 to 7.91 days in the tea leaves at the three doses. Glyphosate concentrations in the tea leaves were found to be below the maximum residue limit (1 mg/kg). © Pesticide Science Society of Japan

**Keywords:** glyphosate, soil, tea leaves, residues, dissipation, rate kinetics.

### Introduction

Tea is the most popular nonalcoholic beverage in the world; it has been consumed for thousands of years for its alluring flavors and health benefits. Numerous epidemiological and preclinical studies have demonstrated that drinking tea may reduce the risk of cancer and cardiovascular diseases.<sup>1,2)</sup> India has the unique distinction of being the second-largest producer and consumer of tea in the world. The Indian tea industry produces more than 1000 million kg of tea from an area of 579.35 thousand hectares.<sup>3)</sup> The tea industry in India has an annual turnover of 8 million USD and provides employment to a million people. The state of Himachal Pradesh representing the mid-hills of northwestern Himalaya, contributes 0.01% of the country's tea production. An increase in the production of tea in India is mainly attributed to efficient and integrated agricultural practices including efficient weed management practices.

Weeds compete with crops for nutrients, sunshine and moisture. Besides reducing the yield, weeds also have adverse effects on the growth of tea *viz.* restricted branching, frame development in younger tea shoots, reduced plucking efficiency, and harboring and serving as alternate hosts for many organisms including some important pests of tea. Depending upon the inten-

sity of weed growth, the extent of competition and the weed species, weeds reduce the productivity of tea by 10–50%.<sup>4–6)</sup> Thus, all of these situations make it necessary to rely on herbicides for effective and timely weed control. In tea plantations, the use of herbicides as a tool for controlling weeds is very popular and has been widely used ever since the introduction of herbicides—primarily due to their cost-effectiveness, their efficiency in controlling diverse weed flora and their requirement for less labor. Lately, the use of herbicides in India is on the rise on tea plantations and their increased use may pose opportunities for chemicals to drift to non-target areas; therefore it is imperative to work out the risk of environmental contamination including the quality of farm produce after field application of the chemicals. Although the herbicide application is target specific, there is always potential for the drifting of chemicals onto non-target plants also.<sup>7)</sup>

Glyphosate, *N*-(phosphonomethyl)glycine is a widely used herbicide worldwide in the world in non-cropped and cropped situations including tea plantations, vineyards and roadside.<sup>7,8)</sup> It is a systemic herbicide that is absorbed by foliage and then translocated throughout the plant *via* phloem.

Data on the rate of degradation are extremely important as they permit prediction of the levels likely to remain in the soil and allow assessment of the risk associated with exposure,<sup>8–12)</sup> and contamination of tea leaves. On degradation, glyphosate yields, AMPA (amino methyl phosphonic acid) a major metabolite of glyphosate which finally degrades and mineralizes to CO<sub>2</sub>. Degradation of glyphosate occurs under aerobic and anaerobic

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conditions in soils and sediments.<sup>13–16)</sup>

Although much information on the fate of glyphosate in the soil is available, little information regarding glyphosate in tropical soil is available. Indiscriminate use of glyphosate on tea lands in some Asian countries has adversely affected the health and productivity of tea bushes in recent years.<sup>17)</sup> Furthermore, increasing levels of glyphosate residues have been reported in some Sri Lankan tea exported to other countries. Therefore, the present study was undertaken to study the dissipation and fate of glyphosate residues in tea leaves and soil.

## Materials and Methods

### 1. Design of the field experiment

The field experiment was conducted in plots set up in a tea plantation area (Tea variety *Kangra Jawala*) of the Research Farm of the Department of Tea Husbandry and Technology, CSKHPKV Palampur, India located at 32°6'N latitude, 76°3'E longitude and at an altitude of about 1290 m above mean sea level. The experimental site falls in the subtropical and mid-hill zone of Himachal Pradesh, India. The experiment was laid out in a completely randomized block design with five replications. Field trials were conducted in 2008 and 2009. For the application of glyphosate at three doses, a plot with nine bushes was selected. The physicochemical properties of soil were analyzed prior to spraying of the chemical on the field. The soil of experimental field had a silty clay loam in texture (26.2% sand, 41.1% silt and 29.8% clay) with an acidic pH 5.21<sup>18)</sup> and 2.44% organic carbon<sup>19)</sup> The fertility status of experimental field was medium in relation to the available nitrogen (407.6 kg/ha)<sup>20)</sup> and was higher in available phosphorus (54.41 kg/ha)<sup>21)</sup> and available potassium (353 kg/ha).<sup>18)</sup>

### 2. Climatic conditions

The temperature of the experimental area varied between mean minimum 11.1°C and mean maximum of 27.2°C in 2008 and 2009 (July–October). Total rainfalls of 1262.9 mm were recorded in 2008 and 2009. The average relative humidity ranged from 68–88% and 47–91% in 2008 and 2009, respectively.

### 3. Application of the herbicide

The glyphosate commercial formulation, Glycel (EC) containing 41% active ingredient was obtained from a local dealer in Palampur, India. In July of both years, Glycel was sprayed at 0.5, 1.0 and 2.0 kg/ha kg equivalents to glyphosate/ha along with the control (no herbicide, water only) in both years by using a sprayed volume of 400 L water/ha with the help of a knapsack sprayer with a flat fan nozzle.

### 4. Sample collection

Periodic analysis of soil and tea samples was carried out at 0 (2 hr), 15, 30, 45, 60, 75 and 90 days after herbicide application. On each sampling day, soil and tea samples from all plots were collected and brought to the laboratory in plastic bags. Soil samples from all treatments were air-dried and passed through a

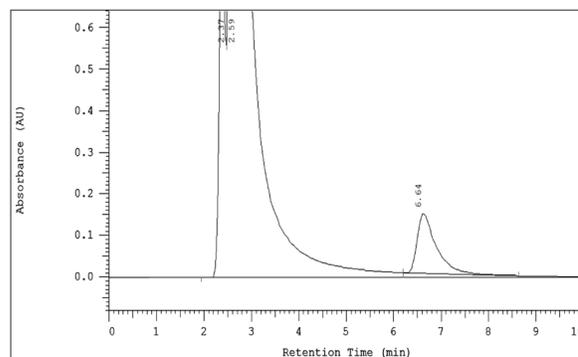


Fig. 1. HPLC profile of glyphosate.

2-mm sieve using CODEX Alimentarius standard protocols of residue analysis.<sup>22)</sup> Tea shoots (each containing two leaves and a bud) were collected, oven dried (45°C), crushed to fine powder and then used for residue analysis.

### 5. Glyphosate analyses

Glyphosate (99.99% purity) reference material was procured from AccuStandard, USA. The stock solution of glyphosate was prepared by dissolving 10 mg of standard glyphosate in 10 mL of sodium tetraborate buffer (1 M, pH 9.0). Working standards (2 mL) of different concentrations prepared from the stock solution were taken in separate vials and 1.0 mL of sodium tetraborate was added to each vial followed by the addition of 1.0 mL of 9-fluoroenylmethyl chloroformate (0.002 M in acetone). These solutions were incubated for 30 min at room temperature for complete derivatization and were analyzed by modifying the method given by Simenson *et al.*<sup>23)</sup>

### 6. Analysis of glyphosate residue

The representative samples (50 g soil or 50 g tea) each in triplicate were taken for the analysis. Extraction in flasks was done with distilled water (200 mL). Flasks were shaken for 3 hr in a reciprocating shaker. The content was filtered through Whatman filter paper No. 1 using activated charcoal. Extraction was repeated with 100 mL of distilled water; the combined filtrate was dried on hot plate at 30°C and 2 mL of sodium tetraborate (1 M, pH 9.0) was added to it and derivatized as described above.

The residues of glyphosate in the derivatized extracts were quantified using a Merck-Hitachi HPLC equipped with a photodiode array detector (PDA) and a Zorbax NH<sub>2</sub> (25 cm × 4.6 mm, 5 μm) column. Estimation was done at 206 nm using mobile phase 0.05 M aq. KH<sub>2</sub>PO<sub>4</sub>: acetonitrile (50:50) buffered at pH 6.0 with 7 N KOH at 0.8 mL/min flow rate using an injection volume of 20 μL. Under these conditions the retention time of glyphosate was found to be approximately 6.64 min (Fig. 1).

### 7. Recovery experiment

To estimate the efficiency of the method, a recovery experiment was conducted by fortifying untreated samples of soil with 0.01, 0.10 and 0.50 μg/g glyphosate and tea leaves with 0.01, 0.50 and

**Table 1.** Method validation by recovery analysis of glyphosate from test samples

Substrates	Amount fortified ( $\mu\text{g/g}$ )	Amount recovered ( $\mu\text{g/g}$ )	Recovery (%)	Average recovery (%)
Soil	0.01	0.007	70.0	72.66
	0.10	0.072	72.0	
	0.50	0.380	76.0	
Tea	0.01	0.006	61.0	72.00
	0.50	0.370	74.0	
	1.00	0.810	81.0	

1.0  $\mu\text{g/g}$  glyphosate (analytical standard 99.99% purity).<sup>24</sup>) The fortified samples were extracted, derivatized and analyzed as per the methods described earlier.

## Results and Discussion

### 1. Recovery studies

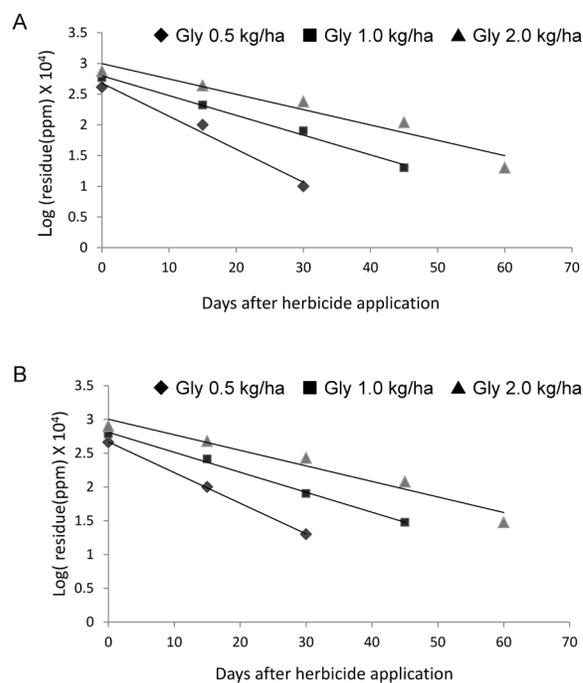
Fortification of the soil with glyphosate (0.01, 0.10 and 0.50  $\mu\text{g/g}$ ) showed that recoveries ranged from 70–76%. Fortification of tea leaves with glyphosate (0.01, 0.50 and 1.0  $\mu\text{g/g}$ ) showed that recoveries ranged from 61–81% (Table 1).

### 2. Glyphosate residues in the soil

Initial deposits of glyphosate residues in the soil immediately after the application of glyphosate during the experiment in 2008 and 2009 were found to be 0.041 and 0.046  $\mu\text{g/g}$ , 0.059 and 0.061  $\mu\text{g/g}$ , and 0.075 and 0.080  $\mu\text{g/g}$  at 0.5, 1.0 and 2.0 kg/ha application rates, respectively. After 15 days glyphosate residues were found to be 0.007, 0.021 and 0.044  $\mu\text{g/g}$  in 2008 at 0.5, 1.0 and 2.0 kg/ha rates of application to tea plants. However glyphosate residues were found to be 0.010, 0.026 and 0.048  $\mu\text{g/g}$ , respectively in 2009 at the three doses of glyphosate application. Thus, the corresponding percentages of dissipation of the applied glyphosate in 2008 and 2009 were 82.98 and 73.07%, 64.00 and 57.37%, and 41.33 and 40.0%, respectively 15 days after herbicide application.

Thirty days after the herbicide application, the dissipation of glyphosate was highest in the glyphosate with a 0.5 kg/ha rate of application (97.56% in Season I and 92.30% in Season II), followed by glyphosate application at 1.0 kg/ha (86.88% in Season I and 86.88% in Season II). The least dissipation was found with the highest dose of glyphosate application *i.e.*, 2.0 kg/ha (68.00% in Season I and 66.25% in Season II). This demonstrated that at higher doses glyphosate persisted in the soil for longer periods than at lower doses. Similar observations have been reported by several researchers.<sup>25,26)</sup>

The period of 30 days after herbicide application included total rainfalls of 636.4 and 626.5 mm during first and second year, respectively. The similar environmental conditions such as heavy rainfall, high average humidity (>86% and 92%) and conducive temperatures during the 30 days after spraying with the herbicide, might have resulted in an almost complete rapid disappearance of the herbicide from the field.<sup>27,28)</sup> In the field, losses due to adsorption or volatilization were also possible. The combined effect on herbicide dissipation by physico-chemical



**Fig. 2.** First order degradation behaviour of glyphosate in soil 2008 (A) and 2009 (B).

parameters and environmental conditions, such as volatilization, vapor pressure, high organic matter, heavy texture of the soil, soil moisture content, high rainfall, and favorable temperatures has also been reported.<sup>28,29)</sup>

During both years of study, residues were found to be below detectable limits 45 days after glyphosate application at 0.5 kg/ha indicated that applied glyphosate persisted for up to 30 days in the soil. The concentration of glyphosate at 1.0 kg/ha, 45 days after herbicide application was found to be 0.002 and 0.003  $\mu\text{g/g}$  and at 2.0 kg/ha was 0.011 and 0.012  $\mu\text{g/g}$  during Season I and Season II respectively. This showed the corresponding dissipation for glyphosate at 1.0 kg/ha application was 96.61 and 95.08% and 85.33 and 85.00%, respectively in 2008 and 2009 at 45 days. This also indicated that 14.67% glyphosate remained in the soil in Season I and 15.00% in Season II at a 2.0 kg/ha application rate. However approximately 3.39 and 5.92% glyphosate remained in the soil in Season I and II, respectively at a 1.0 kg/ha application rate.

**Table 2.** Dissipation of glyphosate in soil

Sampling intervals (In days)	Residues ( $\mu\text{g/g}$ ) and $M^* \pm \text{SD}$ (% Dissipation)					
	$T_1$ (0.5 kg/ha)		$T_2$ (1.0 kg/ha)		$T_3$ (2.0 kg/ha)	
	Season I	Season II	Season I	Season II	Season I	Season II
0	0.041 $\pm$ 0.001 (0.0)	0.046 $\pm$ 0.002 (0.0)	0.059 $\pm$ 0.001 (0.0)	0.061 $\pm$ 0.001 (0.0)	0.075 $\pm$ 0.001 (0.0)	0.080 $\pm$ 0.002 (0.0)
15	0.007 $\pm$ 0.001 (82.98)	0.010 $\pm$ 0.001 (73.07)	0.021 $\pm$ 0.002 (64.40)	0.026 $\pm$ 0.003 (57.37)	0.044 $\pm$ 0.002 (41.33)	0.048 $\pm$ 0.001 (40.00)
30	0.001 $\pm$ 0.0001 (97.56)	0.002 $\pm$ 0.001 (92.30)	0.008 $\pm$ 0.002 (86.88)	0.008 $\pm$ 0.001 (86.88)	0.024 $\pm$ 0.001 (68.00)	0.027 $\pm$ 0.002 (66.25)
45	BDL (100)	BDL (100)	0.002 $\pm$ 0.0001 (96.61)	0.003 $\pm$ 0.001 (95.08)	0.011 $\pm$ 0.001 (85.33)	0.012 $\pm$ 0.001 (85.00)
60	BDL	BDL	BDL	BDL	0.002 $\pm$ 0.0001 (97.30)	0.003 $\pm$ 0.001 (96.25)
75	BDL	BDL	BDL	BDL	BDL	BDL
90	BDL	BDL	BDL	BDL	BDL	BDL

BDL=Below detectable limit ( $<0.001 \mu\text{g/g}$ );  $M^*$ =Mean of five replicates.

**Table 3.** Regression equation, correlation coefficient and half life for dissipation of glyphosate in soil during different years

Seasons	Treatments	Slope of curve (b)	Rate constant (k)	Regression equation	Correlation coefficient	Half-life (days)
Season I (2008)	0.5 kg/ha	0.001	0.119	$y = -0.001x + 0.036$	0.859	5.82
	1.0 kg/ha	0.001	0.069	$y = -0.001x + 0.050$	0.861	10.04
	2.0 kg/ha	0.001	0.027	$y = -0.01x + 0.070$	0.964	25.66
Season II (2009)	0.5 kg/ha	0.001	0.102	$y = -0.001x + 0.041$	0.881	6.79
	1.0 kg/ha	0.001	0.063	$y = -0.001x + 0.053$	0.890	11.00
	2.0 kg/ha	0.001	0.022	$y = -0.001x + 0.075$	0.971	31.50

**Table 4.** Dissipation of glyphosate in tea leaves

Sampling intervals (In days)	Residues ( $\mu\text{g/g}$ ) and $M^* \pm \text{SD}$ (% Dissipation)					
	$T_1$ (0.5 kg/ha)		$T_2$ (1.0 kg/ha)		$T_3$ (2.0 kg/ha)	
	Season I	Season II	Season I	Season II	Season I	Season II
0	0.004 $\pm$ 0.001 (0.0)	0.006 $\pm$ 0.002 (0.0)	0.010 $\pm$ 0.001 (0.0)	0.012 $\pm$ 0.002 (0.0)	0.014 $\pm$ 0.002 (0.0)	0.017 $\pm$ 0.002 (0.0)
15	BDL (100)	0.001 $\pm$ 0.0001 (83.33)	0.002 $\pm$ 0.001 (80.00)	0.003 $\pm$ 0.001 (75.00)	0.003 $\pm$ 0.001 (78.57)	0.004 $\pm$ 0.002 (76.47)
30	BDL	BDL	BDL	BDL	BDL	BDL
45	BDL	BDL	BDL	BDL	BDL	BDL
60	BDL	BDL	BDL	BDL	BDL	BDL
75	BDL	BDL	BDL	BDL	BDL	BDL
90	BDL	BDL	BDL	BDL	BDL	BDL

BDL=Below detectable limit ( $<0.001 \mu\text{g/g}$ );  $M^*$ =Mean of three replicates.

### 3. Dissipation kinetics of glyphosate in soil

The logarithmic plots of herbicides residues *versus* time have been presented in Fig. 2. The plots indicate that the dissipation of glyphosate at all the three levels of application *viz.* 0.5, 1.0 and 2.0 kg/ha fit for the first order kinetics decay curve during both years. The slopes of the curve, coefficients, and the rate constants along with the regression equations are summarized in Table 3. The correlation coefficients ( $r$ ) for the three applied doses in 2008 and 2009 were found to be 0.86 and 0.88; 0.86 and 0.89; and 0.96 and 0.97, respectively indicating a perfect fit. In the first and second season of the experiment, half-life values for the glyphosate in the soil were found to be 5.80 and 6.79 days; 10.05 and 11.07 days; and 16.34 and 19.10 days, respectively (Table 3). Nomura and Hilton<sup>25</sup> reported the half life of glyphosate to

be 19.2 days. However, according to the Weed Science Society of America, glyphosate has moderate field persistence with a typical field half-life of 44–66 days, which is quite significantly higher than the present findings.<sup>30,31</sup> The lower values of half-life values in the present investigation of the soil may be due to the combined effect of high organic carbon, high rainfall and moderate temperatures.

### 4. Glyphosate residue in tea leaves

The concentration of glyphosate and its dissipation in tea leaves as a result of three levels of glyphosate application at 0.5, 1.0 and 2.0 kg/ha is presented in Table 4. The initial deposits of glyphosate residue in tea leaves immediately after glyphosate application at 0.5, 1.0 and 2.0 kg/ha were found to be 0.004,

0.010, and 0.014  $\mu\text{g/g}$  respectively in 2008 and 0.006, 0.012 and 0.017  $\mu\text{g/g}$ , respectively in 2009. Fifteen days after herbicide application, glyphosate residues were found to be below the detection limit (BDL) in lower dose and with the 1.0 and 2.0 kg/ha doses, residues were 0.001  $\mu\text{g/g}$  and 0.002  $\mu\text{g/g}$ , respectively in 2008. Glyphosate residues were found to be 0.003, 0.003 and 0.004  $\mu\text{g/g}$ , respectively in 2009 at 0.5, 1.0 and 2.0 kg/ha application rates. Thirty days after herbicide application, residues were below detectable levels in all glyphosate treatments indicate the complete degradation/disappearance of glyphosate in tea leaves. Prematilake *et al.*<sup>17)</sup> demonstrated that when glyphosate is used in excess of 0.5% ( $>2.7$  kg/ha) on tea lands glyphosate residues have been detected until 14 days after application, whereas glyphosate residues have not been detected in tea seven days after application when the rates are below 0.5%. Beltman *et al.*<sup>32)</sup> observed in one of four repetitions of an experiment that under wet conditions of the brick-soil system, infiltration decreased and therefore the runoff of water and glyphosate increased.

From the data regarding glyphosate residue in tea plants and its corresponding dissipation in the soil, it was found that the rate of the disappearance of glyphosate in plants was rapid during the initial periods which could be due to metabolism by the plants *via* oxidative cleavage of the carbon–nitrogen (C–N) bond to yield aminomethyl phosphoric acid (AMPA) and the breaking of carbon–phosphorus (C–P) by carbon phosphorus lyase to produce sarcosine.<sup>33,34)</sup> Glyphosate is highly soluble in water (11,600 mg/L) and has a very low octanol–water partition coefficient ( $\log K_{ow} = -3.2$  to  $-2.8$ ). However, Edwards *et al.*<sup>30)</sup> demonstrated that, in some field experiments, approximately 2% of the applied glyphosate dissipated from the soil due to run-off. In one case, a loss of 1.85% of the applied glyphosate was observed in a field treated at twice the recommended application rate, with more than 99% of the total runoff occurring during a severe rainstorm the day after application. Wash off by rainfall appeared to be the major route of dissipation.<sup>28,30)</sup>

The glyphosate concentrations in the tea leaves were found to be below the maximum residue limit (1 mg/kg). Presently no residue limit for glyphosate is set by the CODEX Alimentarius. However a maximum residue limit (MRL) is set at 0.2 mg/kg by the CODEX Alimentarius organization for another herbicide that is frequently used in tea gardens in India. The Environmental Protection Agency (EPA), has set the MRL in dry tea leaves at 1 mg/kg, however the European Union has set it at 0.01 mg/kg.

### Conclusion

Glyphosate persisted in the soil for 30 to 60 days, following applications of 0.5 to 2.0 kg/ha in tea crops. Glyphosate residues in the tea leaves were detected up to 15 days at all three treatment doses. The above findings indicate that glyphosate dissipated in tea plants within 15 days of application and that it is safe to use glyphosate in tea plantations at a recommended dose of 1.0 kg/ha in tea gardens. However, on the basis of the present study the mechanism of the degradation behavior of glyphosate could not be confirmed and needs further investigation.

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