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Dissipation of imidacloprid in Orthodox tea and its transfer from made tea to infusion

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Abstract

Imidacloprid is a systemic insecticide used widely in controlling mites, mealy bugs and other related pests in fruits, vegetables and tea. The dissipation behaviour of imidacloprid residues in green tea shoots, made tea and its transfer potential from made tea to infusion in hot water was studied. Analysis in tea matrices of imidacloprid was carried out using high-performance liquid chromatography with diode array detection. Under field conditions, imidacloprid dissipation rate was found to be faster in the wet season than the dry season. Half lives in green shoots were in the range 1.14–1.23 and 1.03–1.09 days, and 1.14–1.25, 1.04–1.07 days in made tea, for the dry and wet season, respectively. The percent transfer of imidacloprid residue from made tea to infusion was in the range of 29.2–42.0% during the dry and wet season; however, 38.2% and 57.9% of the residues remained stuck to the spent leaves during the dry and wet seasons, respectively. On the basis of transfer of residues from made tea to hot water infusion, a waiting period of 7 days after pesticide application at a recommended dose for tea plucking is suggested.

Keywords: Imidacloprid; Insecticide; Residues; Green tea shoots; Made tea; Infusion

1. Introduction

Tea is the most popular beverage throughout the world. Among the factors limiting the quality and quantity of tea production, the role of insect pests is important. Management of pests in tea fields has largely depended on the use of conventional, neurotoxic, broad-spectrum, synthetic chemical pesticides, such as organophosphates, carbamates, synthetic pyrethroids and a number of new chemical classes, such as neonicotinoids. Neonicotinoid insecticides represent the fastest growing class of insecticides introduced to the market since the launch of pyrethroids. The current market share of this class of chemical is well above 600 million Euro per year, including imidacloprid, which is the biggest selling insecticide worldwide. Other neonicotinoids commercialised since the introduction of imidacloprid are acetamiprid, nitempyram, thiacloprid, thiamethoxam, clothianidin and dinotefuran. Neonicotinoid insecticides are active against numerous sucking and biting insects, including aphids, whiteflies, beetles and some lepidoptera species as well (Kodaka, Kinoshita, Swakita, Kawahara, & Yasui, 1998; Tomizawa & Casida, 2003).

Imidacloprid (1-(6-chloro-3-pyridylmethyl)-*N*-nitroimidazolidin-2-ylideneamine) (Fig. 1). It is used as the active ingredient in many different formulations including Gaucho® (Bayer, Leverkusen, Germany). Various international organisations (Environmental Protection Agency (EPA), Codex Alimentarius Commission, World Health Organization (WHO), and Food and Agriculture Organisation (FAO) of the United Nations) have regulated the use of pesticides, by fixing maximum residue levels (MRL's) for commercial purposes. Imidacloprid was registered in Japan for use in tea with a MRL provisionally established by the Japanese Union of 10 mg/kg (http://www.m5.ws001.squarestart.ne.jp/foundation/fooddtl.php?f_inq=13200). In recent years, there have been studies on

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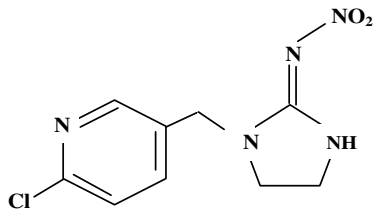

Fig. 1. Structure of imidacloprid.

the behaviour of quinalfos (Jaggi, Sood, Kumar, Ravindranath, & Shanker, 2000), dimethoate, dicofol, deltamethrin (Sood, Jaggi, Kumar, Ravindranath, & Shanker, 2004) and hexaconazole (Kumar, Ravindranath, & Shanker, 2004) pesticides in tea and the influence of various manufacturing processes on their residues found in the made tea (dried black tea leaves) and their fate from made tea to infusion. Nowadays, there is a growing interest in the behaviour and fate of various pesticides in the field and during processing, in order to obtain more realistic data on the residues present in consumable products and provide estimates of the dietary intake of these pesticides. As tea is subjected to infusion prior to consumption, residue levels of many pesticides in tea and in its infusion have been reported (Bhattacharya, Chowdhury, Somchowdhury, Pallarl, & Roy, 1995; Chen & Wan, 1988; Chen, Wan, Wang, Xue, & Xia, 1987; Jaggi, Sood, Kumar, Ravindranath, & Shanker, 2001; Kumar et al., 2004). To the best of our knowledge, no data have been published on the dissipation of imidacloprid residues in Orthodox tea. The present work was carried out to study the dissipation behaviour of imidacloprid residues in green tea shoots, made tea and its transfer potential from made tea to infusion in hot water, in order to protect the consumer, by recommending a waiting period from treatment to harvest.

2. Materials and methods

2.1. Field trials

Field trials were carried out at IHBT tea experimental farm at Banuri, Palampur (32°N × 76°E), HP, India. A plot size of 100 bushes (10 × 10) for each replicate of control and each treatment of the pesticide under study, leaving two rows of bushes as guard rows between the control and the different treatment plots. Imidacloprid was sprayed at two dose levels, 125 ml/ha (recommended) and 250 ml/ha (double of the recommended dose) in three replications with a hand operated Knapsack sprayer, using a recommended formulation volume of 400 l/ha.

2.2. Sampling

For studying the dissipation of imidacloprid in tea, samples (two leaves and a bud) were collected at time 0 day (2 h post application, when the spraying mixture had dried) and 1, 3, 5, 7, 9, 11, 15 and 21 days after the application. About 0.5 kg of the green tea shoots (two leaves and a bud) were harvested from each replicate of both the treatment and control plots and brought to the laboratory.

2.3. Tea leaves processing and infusion preparation

The untreated control and treated green tea shoots from the field were processed in the laboratory's mini-manufacturing unit, using a conventional Orthodox tea manufacturing process. The manufacturing process, in brief, involved withering of shoots (50–55% water loss) at ambient temperature for 15–20 h; rolling (twisting and rupturing the tissue to express the juice) using a piezy roller for about 30 min with pressure, followed by fermentation (oxidation) for 1–2 h at 25–30 °C and 95% RH. Finally, drying in a tea dryer, using hot air at 100 ± 5 °C, gave tea with a final moisture content of 2–3%. Five grams of made tea were infused in 150 ml of boiled water. After 2 min of brewing, the water extract was filtered through a stainless steel filter, cooled and examined for residue transfer from made tea. The matrices used for residue determination were the green tea shoots, made tea, the infusion prepared and the spent leaves left in the stainless steel filter.

2.4. Chemicals

An analytical standard of imidacloprid (CAS No 138261-41-3) was supplied by Sigma-Aldrich and commercial grade Confidence 555 (17.8% SL) was supplied by Crystal Phosphates Ltd. Stock solution (1000 mg/l) prepared in acetonitrile and the solutions required for preparing a standard curve (1.0, 2.5, 5.0, 7.5 and 10.0 mg/l) were prepared from the stock solution by serial dilutions. All the solvents and chemicals used were of analytical grade from E. Merck.

2.5. Analytical procedures

All green tea shoots, made tea, infusion and spent leaves samples were analysed for imidacloprid residues by HPLC with diode array detection (DAD), using a method for imidacloprid determination in tobacco (Liu et al., 2005) with modifications in the extraction, partitioning and clean-up step, in order to remove the interfering coextractives in tea samples. Extraction, partitioning and clean-up step are briefly described below.

2.5.1. Green tea shoots, made tea, infusion and spent leaves extraction and clean-up procedure

Green tea shoots (25 g) and made tea (10 g) were extracted with an appropriate amount of acetonitrile (150 and 100 ml, respectively) with mechanical shaking for 2 h. Extracts were filtered through Whatman No. 1 filter paper containing 2 g of anhydrous sodium sulfate. The filtrate was evaporated to 50 ml and transferred in to a 500 ml separating funnel, to which 100 ml of petroleum ether and

100 ml of 5% NaCl were added. After thoroughly mixing the two phases, the aqueous layer was separated and the organic layer was extracted twice with 100 ml of 5% NaCl. The aqueous phases were combined and then extracted with dichloromethane (50 ml × 3). The combined extract was concentrated and reconstituted in eluent (5 ml) and then transferred to a silica–carbon column (30 cm × 1.1 cm i.d.) [5 g of Merck brand activated silica (60–100 mesh), thoroughly mixed with 0.1 g carbon], prewashed with 50 ml acetone:*n*-hexane (3:7 v/v). The column was eluted with 150 ml of acetone:*n*-hexane (4:6 v/v). The eluent was concentrated to near dryness. The residue was reconstituted in 2 ml of mobile phase (acetonitrile:water) for final analysis. From the infusion prepared (described above), a 50 ml of aliquot (cooled to room temperature) was transferred to a separating funnel (500 ml). The pesticide was extracted with dichloromethane (50 ml × 3). The organic layer was separated, combined and concentrated to 5 ml. The spent leaves were dried between the folds of filter paper and residues were extracted by shaking them with 100 ml acetonitrile for 2 h. The extract was filtered through a Whatman No. 1 filter paper and transferred to a separating funnel. The clean-up procedure for made tea and spent leaves was similar to that used for the green tea shoots.

2.5.2. HPLC determination

A high-performance liquid chromatograph, La-Chrom-Merck, equipped with a LiChrospher® 100 reverse phase (RP) C18 end-capped (30 cm long, 5 μm diameter) column and a DAD was used. The mobile phase was

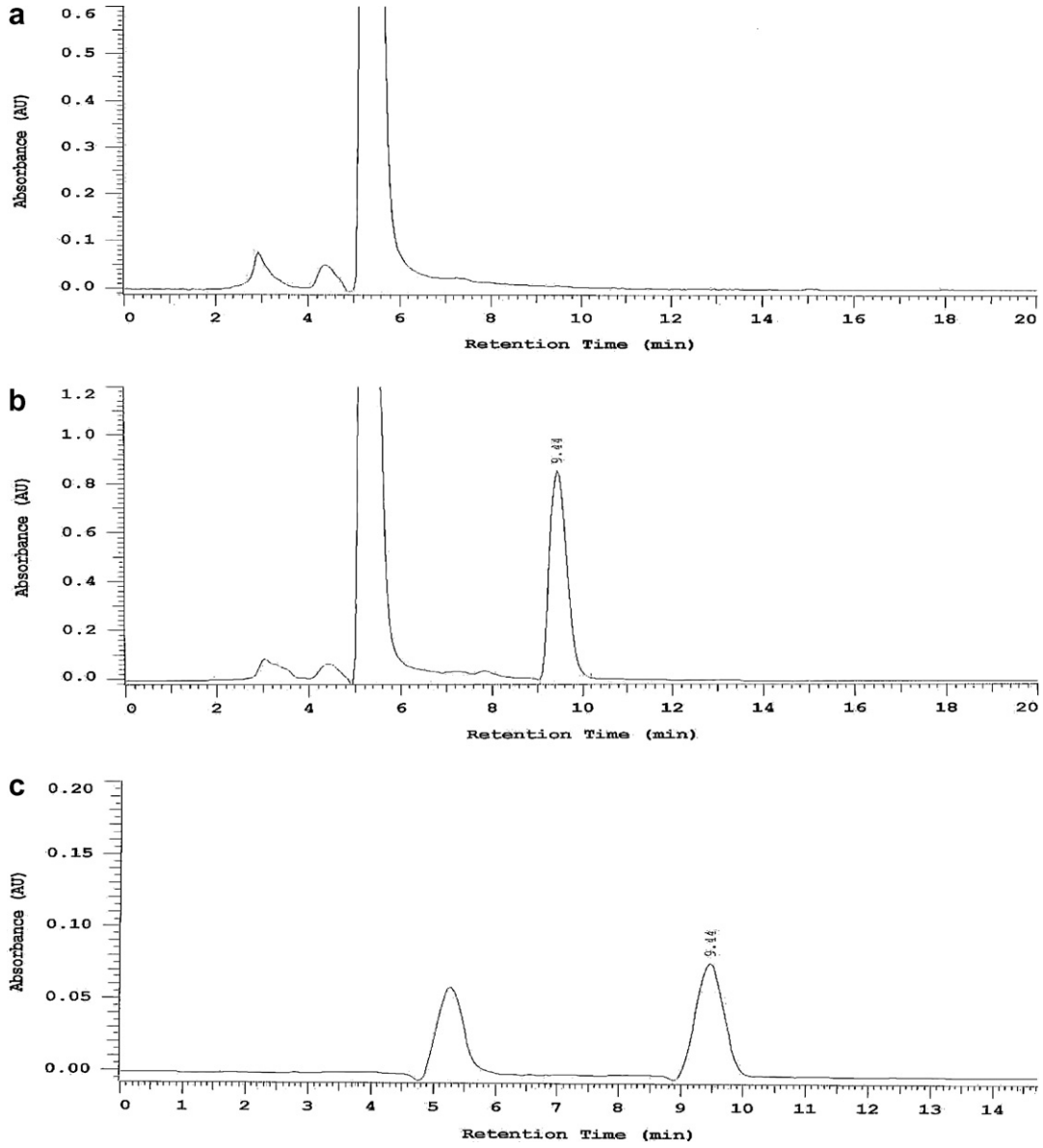

Fig. 2. Chromatograms of imidacloprid ($t_R = 9.44$) in tea: (a) control (untreated sample); (b) made tea (0-day sample with 22.22 mg/kg of insecticide) and (c) standard (solution in acetonitrile:water at 10 mg/l).

acetonitrile:water (30:70 v/v) with 0.50 ml/min flow rate; detection was at 274 nm with an injection volume of 20 μl.

3. Results and discussion

3.1. Imidacloprid determination-method efficiency

The described method of analysis of imidacloprid residues in green tea shoots, made tea, infusion and spent leaves samples by HPLC is fast and relatively simple. Quantification was accomplished by using a standard curve prepared by diluting the stock solution in acetonitrile:water (30:70 v/v). A good linearity was achieved with a correlation coefficient of 0.997. The limit of detection (LOD) was determined, based on the lowest level of standard concentration detected and was found to be 0.05 mg/kg. All control samples show no evidence of chromatographic interference in samples of green tea shoots, made tea, infusion and spent leaves. Confirmation of imidacloprid in samples was performed by assessing its retention time. In Fig. 2, only chromatograms of imidacloprid in made tea are reported because those of green tea shoots, infusion and spent leaves were similar. The efficiency of the method has been evaluated by spiking green tea shoots, made tea, infusion and spent leaves samples with imidacloprid working solutions at various levels (1–10 mg/l). Recovery values for green tea shoots, made tea and spent leaves ranged from 85% to 90% and for infusion from 94% to 96%. All

Table 1
Imidacloprid residues (dry season) in different stages of tea (green tea shoots, made tea, infusion and spent leaves) at different time intervals after application

Time interval (days)	Imidacloprid residue in mg/kg ± standard deviation							
	Green tea shoots		Made tea		Infusion		Spent leaves	
	T_1	T_2	T_1	T_2	T_1	T_2	T_1	T_2
0	4.23 ± 0.55	9.52 ± 1.32	12.0 ± 0.09	22.1 ± 0.16	4.07 ± 0.05 (34.1)	7.99 ± 0.68 (36.2)	5.14 ± 0.30 (43.0)	9.79 ± 0.52 (44.3)
1	2.46 ± 0.28	5.94 ± 0.42	6.26 ± 0.07	13.9 ± 0.23	1.83 ± 0.06 (29.2)	4.89 ± 0.30 (35.1)	2.99 ± 0.06 (47.8)	5.90 ± 0.56 (42.3)
3	0.99 ± 0.05	2.96 ± 0.42	2.67 ± 0.11	7.53 ± 0.18	1.06 ± 0.07 (39.7)	2.74 ± 0.21 (36.4)	1.39 ± 0.04 (52.1)	3.66 ± 0.31 (48.6)
5	0.25 ± 0.03	0.76 ± 0.04	0.68 ± 0.01	2.18 ± 0.10	0.28 ± 0.02 (41.2)	0.69 ± 0.08 (31.7)	0.26 ± 0.05 (38.2)	0.97 ± 0.03 (44.5)
7	0.06 ± 0.01	0.19 ± 0.01	0.17 ± 0.01	0.50 ± 0.02	ND	0.21 ± 0.01 (42.0)	ND	0.20 ± 0.00 (40.0)
9	ND	0.06 ± 0.00	ND	0.15 ± 0.01	ND	ND	ND	ND
11	ND	ND	ND	ND	ND	ND	ND	ND
15	ND	ND	ND	ND	ND	ND	ND	ND
21	ND	ND	ND	ND	ND	ND	ND	ND

T_1: treatment 1 (125 ml/ha); T_2: treatment 2 (250 ml/ha); ND: not detected. Values in parentheses are the % transfer of residues from made tea.

Table 2
Imidacloprid residue (wet season) in different stages of tea (green tea shoots, made tea, infusion and spent leaves) at different time intervals after application

Time interval (days)	Imidacloprid residue in mg/kg ± standard deviation							
	Green tea shoots		Made tea		Infusion		Spent leaves	
	T_1	T_2	T_1	T_2	T_1	T_2	T_1	T_2
0	3.30 ± 0.03	6.93 ± 0.35	9.52 ± 0.15	17.94 ± 0.05	3.41 ± 0.58 (35.8)	6.61 ± 0.63 (36.9)	4.79 ± 0.13 (50.3)	8.87 ± 0.18 (49.4)
1	1.48 ± 0.17	2.88 ± 0.06	3.94 ± 0.10	7.80 ± 0.32	1.32 ± 0.02 (33.5)	2.37 ± 0.05 (30.4)	2.28 ± 0.04 (57.9)	3.31 ± 0.14 (42.4)
3	0.30 ± 0.02	0.82 ± 0.02	0.91 ± 0.07	2.21 ± 0.13	0.37 ± 0.00 (40.7)	0.87 ± 0.02 (39.4)	0.44 ± 0.03 (48.4)	1.13 ± 0.08 (51.1)
5	0.10 ± 0.01	0.25 ± 0.01	0.26 ± 0.02	0.65 ± 0.02	0.10 ± 0.00 (38.5)	0.26 ± 0.02 (40.0)	0.12 ± 0.01 (46.2)	0.26 ± 0.00 (40.0)
7	0.03 ± 0.00	0.08 ± 0.01	0.09 ± 0.01	0.19 ± 0.00	ND	ND	ND	ND
9	ND	ND	ND	ND	ND	ND	ND	ND
11	ND	ND	ND	ND	ND	ND	ND	ND
15	ND	ND	ND	ND	ND	ND	ND	ND
21	ND	ND	ND	ND	ND	ND	ND	ND

T_1: treatment 1 (125 ml/ha); T_2: treatment 2 (250 ml/ha); ND: not detected. Values in parentheses are the % transfer of residues from made tea.

of these values of recovery indicated good method accuracy and repeatability, and are within the accepted range for residue determinations (Commission of the European Union, 2003; Greve, 1984). Half-life of imidacloprid in different matrices was calculated using the first order rate equation:

$$C_t = C_0 e^{-kt}$$

where C_t represents the concentration of the pesticide residue at time t, C_0 represents the initial concentration and k is the rate constant per day. The half life ($t_{1/2}$) was determined from the k value for each experiment, where $t_{1/2} = \ln 2/k$.

3.2. Dissipation of residues

3.2.1. Green tea shoots and made tea

The data relating to the residues in tea from the field experiments carried out in May, 2006 (dry season) and July, 2006 (wet season) are reported in Tables 1 and 2. No residues of imidacloprid were detected in any analysed control tea sample. The initial deposits of the imidacloprid residues in green tea shoots at the two dosages were 4.23 and 9.52 mg/kg in the dry season, and 3.30 and 6.93 mg/kg in the wet season. It is evident from the data that imidacloprid degraded more rapidly in the wet season (rainfall: 27.32 mm; maximum temperature: 25.6 °C; minimum temperature: 20.2 °C; relative humidity: 87.1%; sunshine: 2.01 h) than in dry season (rainfall: 2.91 mm; maximum temperature: 30.0 °C; minimum temperature: 19.2 °C; relative humidity: 52.9%; sunshine: 7.74 h) and after 7 days no residues were found in green tea shoots during the wet season, at both dosages. In the tea field, besides the effect of some physical and chemical factors, like light, heat, pH and moisture (Agnihothrudu & Muraleedharan, 1990; Chen et al., 1987; Cosby, Moilanon, Nakagawa, & Wong, 1972; Miller & Donaldson, 1994; Miller & Zepp, 1983) on degradation of insecticides, growth dilution factor (Agnihothrudu & Muraleedharan, 1990; Bisen & Ghosh Hajara, 2000; Chen & Wan, 1988) might have played a significant role in the degradation of imidacloprid residues. Growth factor was higher during the wet season. In the case of made tea, residues observed on day 0 were 12.0 and 22.1 mg/kg in the dry season, and 9.52 and 17.9 mg/kg in the wet season at the two dosages. The residues in the made tea were 2.32–3.03 times higher than in the corresponding samples of green shoots. During processing, leaves undergo dehydration, which causes a concentration factor of 3–4 (Nagayama, 1996). Therefore, theoretically the residue in made tea should increase by a similar factor, if it is not lost during manufacturing. However, results showed that the residue levels were smaller than the concentration effect. This indicates that during the processing of green tea shoots the residue decreased by approximately 33%. The present finding supports the studies reporting loss of many pesticides during processing (Chen & Wan, 1988; Jaggi et al., 2000). The imidacloprid residues were dissipated by more than 98% (green tea shoots and made tea) in 7 days at the recommended dosage in both the seasons. The

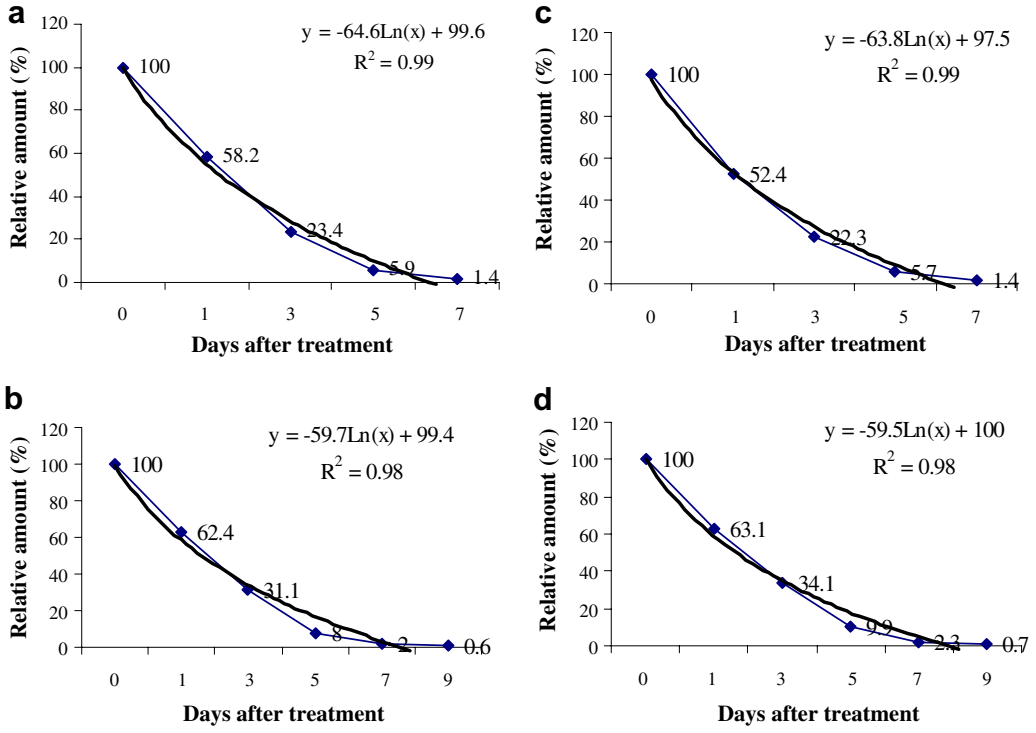

Fig. 3. Relative amount of imidacloprid in (a, b) green tea shoots and (c, d) made tea samples during the dry season. Dose levels are 125 ml/ha (a and c) and 250 ml/ha (b and d).

Table 3
Half life and other statistical parameters for imidacloprid dissipation in tea

Season	Dosage	Regression equation	Determination coefficient (R^2)	Correlation coefficient (r)	Degradation constant (days $^{-1}$)	Half-life $t_{1/2}$ (days)
Green tea shoots						
Dry	T_1	$\text{Log}\, R = -0.262x + 0.676$	0.992	0.996	0.61	1.14
	T_2	$\text{Log}\, R = -0.249x + 1.06$	0.989	0.995	0.56	1.23
Wet	T_1	$\text{Log}\, R = -0.290x + 0.457$	0.993	0.997	0.67	1.03
	T_2	$\text{Log}\, R = -0.272x + 0.775$	0.996	0.998	0.64	1.09
Made tea						
Dry	T_1	$\text{Log}\, R = -0.260x + 1.10$	0.992	0.996	0.61	1.14
	T_2	$\text{Log}\, R = -0.243x + 1.44$	0.984	0.992	0.55	1.25
Wet	T_1	$\text{Log}\, R = -0.288x + 0.904$	0.993	0.996	0.67	1.04
	T_2	$\text{Log}\, R = -0.278x + 1.21$	0.998	0.999	0.65	1.07

T_1: treatment 1 (125 ml/ha); T_2: treatment 2 (250 ml/ha).

relative amount pattern is shown in Fig. 3. Half lives ($t_{1/2}$) and other statistical parameters of imidacloprid residue dissipation were calculated from the experimental data and summarised in Table 3. The dissipation trend of imidacloprid in both seasons followed first order kinetics. The half life values of imidacloprid ranged from 1.14–1.23 and 1.03–1.09 days in green tea shoots, and 1.14–1.25 and 1.04–1.07 days in made tea, in dry and wet seasons, respectively. Sanyal, Hazra, Pal, Somchaudhury, and Chowdhury (2006) reported the half lives of imidacloprid in CTC tea in the range of 0.91–1.16 days; this lower half life may be due to the type of tea. Residues were below the detectable limit in the infusion of the 3rd day samples.

3.2.2. Infusion and spent leaves

The percent transfer of residues during the brewing process was 29.2–41.2 and 31.7–42.0 in the dry season, and 33.5–40.7 and 30.4–40.0 in the wet season, in the single and double dosages, respectively. The percent residues remaining on the spent leaves were 38.2–52.1 and 40.0–48.6 in the dry season, and 46.2–57.9 and 40.0–51.1 in the wet season, in the single and double dosages, respectively. No significant relationship was observed between the transfer of imidacloprid residue and the rate of application. The transfer rate of pesticide residues to the infusion depends on the water solubility (Nagayama, 1996; Wan, Xia, & Chen, 1991) and partition coefficient of the pesticide (Jaggi et al., 2001; Tsumura-Hasegawa, Tonogai, Nakamura, & Ito, 1992). This insecticide has a high water solubility, i.e., 0.51 g/l at 20 °C (Miles Inc., 1993) but a low organic matter adsorption capability, with organic adsorption coefficient values (K_{oc}) from 130 to 310 cm^3/g (Rouchaud, Gustin, & Wauters, 1994); further, it binds to suspended organic matter in the infusion (proteins, carbohydrates, pigments etc.) and has a lower octanol/water partition coefficient ($K_{ow} = 3.72$ at 22 °C) because of the presence of polyphenols and volatile aromatic compounds (Shanker, Sood, Kumar, & Ravindranath, 2001), like *n*-butanol, 1-pentanol (Skobeleva, Bezzubov, Petrova, & Bockuchava, 1979), *n*-hexanol, 2-methyl-2-hexanol (Mick & Schreier, 1984) and 1-octen-3-ol (Fernando & Roberts, 1984). This may be the reason for partial transfer of imidacloprid from made tea to infusion, with a large proportion remaining on the spent leaves.

4. Conclusions

Imidacloprid showed a relatively fast dissipation in tea shoots. The dissipation of imidacloprid in tea varied with respect to the seasons. Imidacloprid persisted both in green tea shoots and made tea a little longer during the dry season, compared to the wet season. The transfer of imidacloprid residue from made tea to infusion was observed to depend on the amount of imidacloprid present in the made tea. On the basis of the above findings, it can be concluded that on or after the seventh day of harvesting (normal plucking round after spraying), there was no detectable residue transfer to the infusion, at the recommended application dose (125 ml/ha) in both seasons. Thus, infusion consumption is safe at the normal harvesting interval after treatment. The half life of imidacloprid in made tea ranged from 1.04 to 1.25 days in both dry and wet seasons and thus this insecticide can be safely recommended for any pest management programme in tea plantations. The described method of analysis of imidacloprid residues is suitable for determination of residues in tea and the method can be suitably applied to other members of the neonicotinoid group. Furthermore, the metabolism of imidacloprid in tea plant can be studied. This study should also be repeated under different agro–climatic conditions for the confirmation of the data.

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