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Leaching Behavior of Imidacloprid Formulations in Soil

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Soil acts as a major sink for bulk of the pesticides used in agriculture and public health programs. Pesticides reach soil by various routes like direct application to soil, spray drift, dislodging and run off. In soil pesticide residues are subjected to various transformation and transportation processes. Leaching is one of the major transportation process responsible for ground water contamination. The presence of pesticide residues in ground water has been reported by many (Mohapatra et al. 1995; Flury 1996; Loague et al. 1996) and is a major concern worldwide as ground water is a source of drinking and irrigation water in many countries. Therefore, pesticide leaching studies, especially for the pesticides having high water solubility, are necessary to determine their potential to contaminate ground water. In soil, in addition to soil properties and chemical nature of the pesticide, the leaching is also affected by type of formulation, surfactant and the adjuvant (Hall et al. 1998; Camazano et al. 1995). It is, therefore, important to study the effect of formulation on leaching.

Imidacloprid (1- [6-chloro -3-pyridylmethyl] -N-nitroimidazolidin - 2- ylidene amine) is a comparatively new insecticide having systemic action. It has been found to be highly effective against various sucking insect pests in crops like cotton (Gupta et al. 1998), sugarcane (Gajbhiye et al. 1997), rice (Mao and Liang 1995), apple (Forti et al. 1997) and citrus (Nucifora 1996). In India, three formulations of imidacloprid are being used viz. Gaucho (70 WS) for seed dressing, Confidor (200 SL) and Admire (350 SC) for spraying. Imidacloprid has relatively high water solubility (0.51 g/L) and low octanol- water partitioning coefficient [Log (Pow) = 0.57] which makes it an ideal case for studying its leaching potential in soil. The review of literature revealed that few reports are available on leaching of imidacloprid in soil (Felsot et al. 1998; Pradas et al. 1999; Perez et al. 1998; Leib et al. 2000; Ndongo et al. 2000). However, there is no report on the effect of commonly used formulations on the leaching of imidacloprid, especially in Indian soils. Therefore, studies were carried out to determine the effect of formulation on leaching potential of imidacloprid in alluvial soil of India.

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MATERIALS AND METHODS

Leaching studies were carried out in soil column in duplicate. Analytical grade imidacloprid (AGI, 96%) and its three formulations viz. water dispersible powder (Gaucho 70 WS), soluble concentrate (Confidor 200 SL) and suspension or flowable concentrate (Admire 350 SC) were supplied by M/s Bayer India Ltd., New Delhi, India.

For leaching studies, fresh soil was collected from the plough layer (0-15 cm) from the cultivated fields of Indian Agricultural Research Institute, New Delhi, with no previous history of imidacloprid application. Soil was air dried under shade, ground and passed through 2 mm sieve. The physico-chemical properties of the soil were : type - inceptisol, texture - sandy loam, clay - 5%, sand - 77.5%, silt - 17.5%, organic matter - 0.864% and pH - 7.69. For soil treatment, 1000 μg/ml solution/suspension were used. Solution of analytical grade (AGI) material was made in acetonitrile whereas formulations were diluted with water to give 1000 μg/ml concentration. Ten gram soil was taken in beaker and required amount of each solution containing 1000 μg of imidacloprid was added, mixed well and left overnight for evaporation of solvent. This treated soil was placed at the top of the leaching soil column.

Soil columns made of PVC tube (50 cm x 7 cm id) were cut longitudinally into two halves and rejoined using packing tape. The columns were capped at the bottom with polythene sheet having tiny holes. The column was packed with soil (1250 g), which gave about 25 cm of soil column. Leaching studies were carried out under saturated flow conditions. The packed column was placed vertically in water for overnight and allowed to saturate. Next day, excess water was allowed to drain out. Columns were clamped to a stand and 10 g treated soil was placed on the top of the soil column. Water was added drop wise with the help of separatory funnel @ 1 ml/min. A constant head of 2 cm water was maintained during leaching cycle. Five fractions of around 500 ml each were collected.

After completion of leaching cycle, two halves of the column were separated by cutting the packing tape. The soil column was cut into five cores of 5 cm each. Soil from each core was mixed and representative sub-samples in duplicate were drawn for analysis.

Different fractions of leachate were filtered separately and transferred into a separatory funnel. About 50 ml saturated sodium chloride solution was added and made acidic (pH 3) by adding dilute hydrochloric acid. The contents were partitioned thrice with hexane and hexane phases discarded. The aqueous phase was then made basic (pH 9) by addition of 5% potassium hydroxide solution and residues of imidacloprid extracted with dichloromethane (3 x 50 ml). The extracts were passed through anhydrous sodium sulfate to remove traces of moisture,

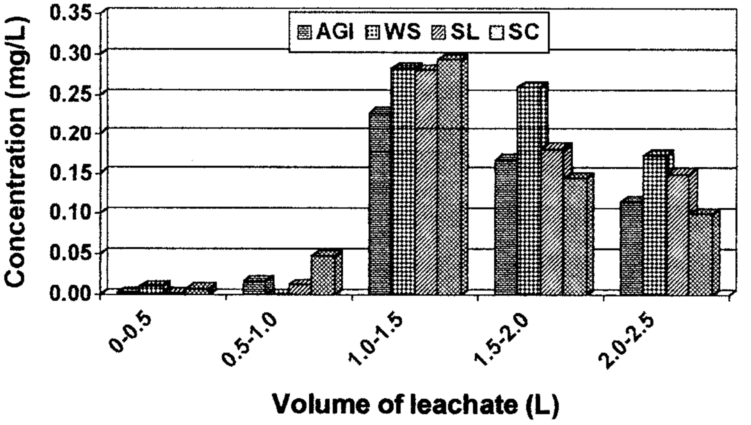

Figure 1. Residues of imidacloprid in leachate fractions

concentrated to dryness and residues dissolved in HPLC grade acetonitrile for analysis.

The soil samples were extracted with acidic mixture of acetone water (2:8) by dipping and shaking. The acetone from the extract was removed by concentrating under vacuum and aqueous phase extracted similar to leachate fractions.

The residues of imidacloprid in various sample extracts were analyzed by Water's HPLC equipped with dual pump (model 501), variable UV-Visible detector (model 484), rheodyne injector (20 µl), column RP C-8 (250 x 4 mm, 10 µm). Analysis was carried out at 270 nm wavelength.

Acetonitrile-water solvent system (flow rate 1 ml/min.) with gradient as given in Table 1 was used. Under these operating conditions, the retention time of imidacloprid was 10.5 minutes.

Table 1. Solvent gradient used in HPLC analysis

Time (min.)	Water (%)	Acetonitrile (%)
0	95	5
4	75	25
11	60	40
12	10	90
17	10	90
18	95	5
20	95	5

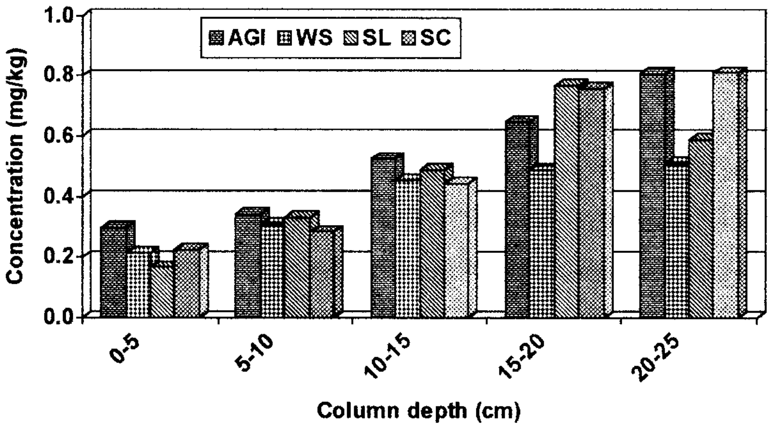

Figure 2. Residues of imidacloprid in soil from different depths

RESULTS AND DISCUSSION

The recoveries of imidacloprid from water and soil samples fortified at 0.1 ppm level varied from 90-95 and 85-89%, respectively. The residues of imidacloprid in different fractions of leachate are presented in Figure 1.

The data revealed that imidacloprid residues were present in all the fractions of leachate. However, the first fraction contained the least amount of imidacloprid in all the treatments. The concentration in leachate increased in each subsequent fraction and maximum was recorded in third fraction (1.0-1.5 L) but decreased thereafter. Similar trend was observed in all the treatments, however, the concentrations differed. Leaching of imidacloprid in soil was more when treated with formulations than analytical grade material.

In all the fractions, the concentration of imidacloprid in leachate was higher in formulation treatment (Figure 1). The highest concentration was recorded in the third fraction of leachate of suspension concentrate (SC) formulation treatment.

The distribution of residues in soil cores at different depths after the passage of 2.5 L water (equivalent to about 65 cm rainfall) is presented in Figure 2. It was observed that imidacloprid got distributed throughout the column. Lowest concentration was recorded in the top 0-5 cm layer. The concentration increased with column depth showing downward movement. Similar type of distribution trend was observed in all the treatments, however, the bandwidth and concentration differed with formulation (Figure 3).

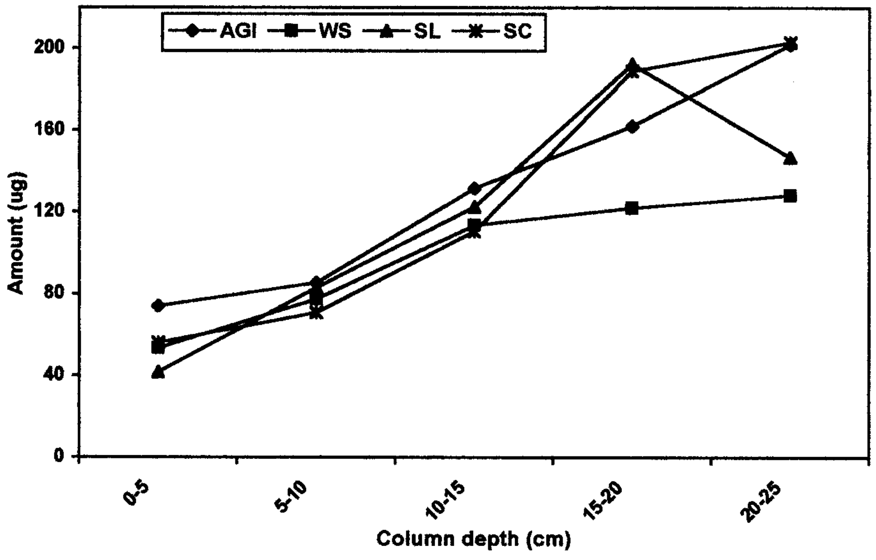

Figure 3. Distribution of imidacloprid in soil column

In analytical grade treatment (AGI), there was continuous increase in concentration with depth and highest concentration was recorded at 20-25 cm depth. In water dispersible powder (WS) formulation treatment, there was increase in concentration from 0-15 cm depth, but there was not much difference in concentration in soil from 15-25 cm depth showing a broad band of almost uniform distribution. In soluble concentrate (SL) formulation treatment, the concentration increased up to 20 cm depth with highest concentration in 15-20 cm depth soil. In suspension concentrate (SC) formulation treatment, the concentration in soil increased with depth and band of highest concentration was recorded at 15-25 cm depth.

Out of the 1000 μg imidacloprid added to the column, 894-926 μg (89.4-92.6% of added) was recovered from leachate and soil. A loss of 74-106 μg (7.4-10.6% of added) could be attributed to degradation of imidacloprid during leaching period and processing of samples.

In analytical grade treatment (AGI), 263.3 μg (26.3% of applied and 28.7% of recovered amount) was present in leachate. This shows quite high potential for leaching of imidacloprid below plough layer. The distribution of imidacloprid in soil cores at different depth also reveals that imidacloprid has moved downward showing its high mobility in soil. Similar results have also been reported by others. Pradas et al. (1999) reported that 82.3% of added imidacloprid leached down from the amended soil column when applied as technical grade material. Perez et al. (1998) observed that imidacloprid could leach down even through a peat layer in a layered soil column. Around 3% of the added technical grade

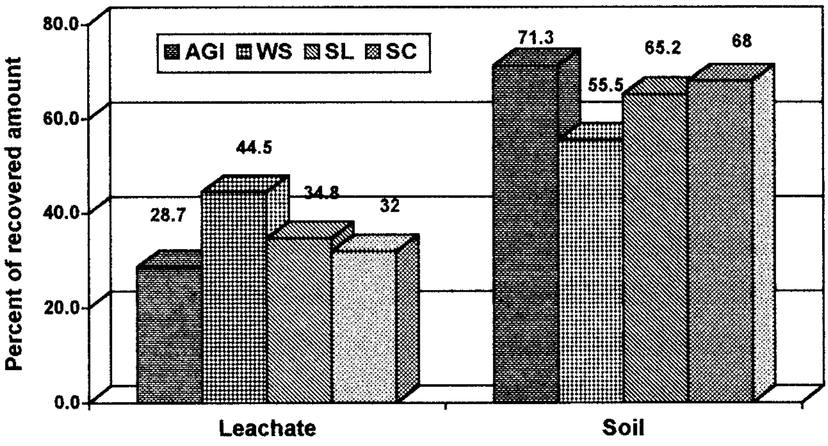

Figure 4. Imidacloprid recovered in column leachate and soil

imidacloprid was recovered in leachate of a layered soil column. Ndongo et al. (2000) have reported that imidacloprid residues were detected in leachate of undisturbed soil column and percolated water below 1 m depth in field lysimeter.

The formulation treatments show still higher potential for leaching of imidacloprid (Figure 4). Amongst formulations, leaching was more in WS formulation (396.9 μg) followed by SL (312.8 μg) and SC formulation (296.7 μg). A higher leaching of imidacloprid when applied as formulated material could be attributed to the effect of formulation ingredients like carrier, surfactant and stickers etc.

Amongst these ingredients, surfactants may have a pronounced effect on the leaching. The main function of the surfactant is to keep the pesticide in soluble/suspended form in water for longer period, which may in some way help in leaching process. Based on the soil TLC mobility studies, Camazano et al (1995) reported that depending on the type of surfactant, its concentration and nature of pesticide, pesticide mobility could increase or decrease. It seems that for WS, SL and SC formulations of imidacloprid, the surfactant and pesticide combination is such that it results in greater mobility. The differences among formulations could be attributed to the type of surfactant and its concentration in formulation.

Hellpointer (1998) has observed low mobility of imidacloprid and its degradation products under practice relevant field conditions. Further, the mobility of imidacloprid in soil could be considerably reduced by use of controlled release formulations (Perez et al. 1998; Pradas et al. 1999). Even though our study showed high potential for leaching of imidacloprid applied as different formulations, but under natural condition the magnitude of leaching could be much lower due to compactness of the field soil and lower rainfall than we used for our experiments.

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