Original Research Article

Leaching Behaviour of Combination Product (Novaluron 5.25% + Indoxacarb 4.5% SC) at Different Depths of Sandy Loam Soil

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Abstract

Leaching behaviour of combination product (novaluron 5.25% + indoxacarb 4.5% SC) was investigated in sandy loam soil at 43.31 + 37.13 g a.i. ha⁻¹ (single dose) and 86.62 + 74.26 g a.i. ha⁻¹ (double dose) under laboratory conditions as per average annual rainfall of 400 mm. Residues of novaluron and indoxacarb were estimated at different depths of soil and in leachates with the help of gas liquid chromatography (GLC) equipped with electron capture detector (ECD). Method was validated by performing recovery experiments at different spiking (0.01 - 0.10 mg kg⁻¹) levels. Results revealed that maximum retention of combination product was found upto 10 cm soil i.e. 56.97 - 55.72 % in novaluron and 74.36 - 72.20 in indoxacarb while residues reached below detectable level of 0.01 mg kg⁻¹ after 40 cm depth of soil at both doses. Since, residues of combination product were not detected in any of the leachate fractions of soil. Hence, this combination product may be safe for soil and ground water contamination.

Keywords
Leaching, Novaluron, indoxacarb, Percent retention, Combination product, Sandy loam soil, GLC

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Introduction

Contamination of ground and surface water all over the world had become more evident over the last decade due to extent use of pesticides. Residues of insecticide, pesticides and their degradation products have frequently been detected in field groundwater monitoring programmes. As pesticides are one of the major technological developments of twentieth century, whether natural or synthetic, they have toxicological significance and pose potential risk when persist in the environment. Persistence and leaching of pesticide compounds contribute heavily to possible contamination of groundwater. The presence of insecticide residues in runoff, sediment and leachate, as well as their mobility and persistence in soil, depends on such factors as chemical and physical properties of the compound, soil properties, amount of rainfall, bed construction and the degree of slope (Halimah et al., 2005).

The indiscriminate use of pesticides has given rise to many problems viz. persistence of toxic residues in the environment, development of resistance in insect pests and resurgence of
pests. Soil is an important component of the environment, act as a sink for the pesticides used in agriculture. Such treatments may suppress soil microflora and hence affect soil properties. The pesticides present in soil sometimes act as a source of contamination for succeeding crop also. From soil, the pesticides residues can reach to water bodies by leaching and runoff.

The main processes potentially affecting the ultimate fate of pesticides in soil are retention by soil materials (involving adsorption/desorption processes), transformation processes (biological and chemical degradation) and transport (through soil, atmosphere, surface water, or ground water) (Dagar et al., 2014). In recent years combination product or combination products are widely used by farmers as they are more effective than a single pesticide over multiple pest problems.

A relatively new benzoylphenyl urea insect growth regulator which inhibits the chitin formation on larvae of various insects (lepidoptera, coleoptera, and diptera) ie. Novaluron [(±)-1-[3-chloro-4-(1,1,2-trifluoro-2-trifluoromethoxyethoxy) - phenyl]-3-(2,6-difluoro-benzoyl)urea] was developed by Makhteshim-Agan Industries Ltd. (Anita et al., 2018). Insect growth regulators (IGR) insecticides are comparatively safer to beneficial insects and environment and are compatible for use in an integrated pest management system (Ghosal et al., 2016). This compound is coming up as an eco-friendly or green pest-controlling agent (FAO, 2003).

The other insecticide indoxacarb, {methyl 7-chloro-2,5-dihydro-2-[(methoxy-carbonyl)[4-(trifluoromethoxy)phenyl]amino]carbonyl]inden0[1,2-e][1,3,4]oxadiazine-4a(3h)-carboxylate}, is a non-systemic, synthetic organophosphate replacement insecticide with broad spectrum activity and it was developed by E. I. Du Pont de Nemours Company (Pesticides, 2000). It is highly effective having low side effect on non-target insects (Michand and Grant, 2003) and allows most predators and immature wasp parasites to survive (Hewa-Kapuge et al., 2003). Since ground water is the main source of drinking and irrigation water thus to assess the risk of ground water contamination by both the insecticides, this experiment was carried out to generate information on the leaching behaviour of both the insecticides used as combination product in sandy loam soil at different doses under laboratory conditions.

**Materials and Methods**

Leaching studies of combination product (Novaluron + Indoxacarb) in soil were carried out under laboratory conditions during January-March 2016. Experiment was conducted in sandy loam soil collected from Research Farm of CCS HAU, Hisar. The physico-chemical properties of the soil are given in Table 1.

**Packing of glass column**

Plexi glass columns (90 cm x 2.2 cm i.d.) fitted with perforated plexi glass sieve covered with filter paper (Whatman No. 1) at the bottom was used for the leaching study. Each column was packed with soil up to 60 cm height to a uniform bulk density (BD) of 1.35 g cm$^{-3} \pm 0.1$. Weighed amount of soil was poured in the columns each time with the help of a funnel and tapped gently from a fixed height. This process was repeated till each column was uniformly packed to a height of 60 cm. The soil in each column was covered with filter paper which was tapped by a glass wool swab. Soil column so filled were installed vertically on wooden stand with their bottom resting on the fixed perforated sieve, so as to facilitate collection of effluents during
leaching. Leaching studies were carried out in triplicates. Soil was fortified with single and double dose solution of combination product. This fortified soil was added at the top of the column. Cotton swabs pre washed with acetone and dried were placed in between the column to avoid disturbance of soil. One column was packed with soil was kept as control to which no pesticide was added.

**Leaching of soil columns**

Each soil column was leached with 50 mm of water at a time with the help of pipette, with the care that no spattering of soil took place. This procedure of leaching was carried out again after 24 hrs of the disappearance of the standing water in the column. Leaching was repeated till each leaching cycle of 400 mm depth was completed.

**Extraction and clean up**

After completion of leaching, intact soil cores were taken out of the plexi glass column. The cores were sliced in to pieces of height 10 cm each and air dried, grinded than soil sample is sieved through a 2 mm sieve and analyzed for quantification of combination product (Novaluron + Indoxacarb) residues. Leachates oozed out of all the columns were collected and processed to check the presence of combination product (Novaluron + Indoxacarb) residues. Water extract was taken in separating funnel and 4 g of NaCl was added and dissolved in it. Then extract was partitioned thrice with dichloromethane (50, 30, 20 ml) by vigorous shaking for 5 min to remove the non-emulsifying impurities. Each time, organic phase was collected, passed through the 2-3 cm pad of anhydrous sodium sulphate and pooled together. Extract was concentrated twice with the help of rotatory vaccum evaporator in n-hexane and final volume made up to 2 ml. After this GLC analysis was carried out.

**Results and Discussion**

**Leaching behaviour of novaluron**

The data on leaching potential of novaluron in sandy loam soil is presented in Table 2 and figure 1. Total amount of novaluron recovered out of 3.23 and 6.46 μg were 81.16 and 79.87 per cent from single and double dose, respectively. Per cent distribution of novaluron in different soil cores (0-10, 10-20, 20-30, 30-40, 40-50 and 50-60 cm) was 56.97, 16.10, 6.19 and 1.90 per cent at single dose where in double dose 55.72, 16.87, 5.42 and 1.86 per cent, respectively. Residues reached below detectable limit after 40 cm depth of soil at both the doses. None of the leachate fractions from both the treatments showed the presence of novaluron residues.

**Leaching behaviour of indoxacarb**

Leaching potential data of indoxacarb in sandy loam soil is presented in bar diagram of figure 2 and Table 3. Total amount of indoxacarb recovered out of 2.77 and 5.54 μg were 93.48 and 88.98 per cent from single and double dose, respectively. Per cent distribution of indoxacarb in different soil cores (0-10, 10-20, 20-30, 30-40, 40-50 and 50-60 cm) was 74.36, 13.71, 4.69 and 0.72 per cent at single dose while in double dose it was 72.20, 11.01, 5.23 and 0.54 per cent. Residues reached below detectable level after 40 cm depth of soil at both the doses. As in above case none of the leachate fractions from both the treatments showed the presence of indoxacarb residues. Eliana et al., (2009) determined the leaching behaviour of diuron in soil and water. In percolated water, diuron was detected with low frequency but in relatively high concentrations (up to 6.29 μg L⁻¹). In runoff water and soil, diuron was detected in decreasing concentrations until 70 days after application, totalizing 13.9% during the whole sampling period.
Table 1 Physico-chemical characteristics of soil

| Soil Type     | Sandy Loam Soil |
|---------------|-----------------|
| Texture       | Loam            |
| **Ph**        | 7.6             |
| EC (dSm⁻¹)    | 2.0             |
| O.C. (%)      | 0.67            |
| **Kc**        | 10.08           |
| **P₂O₅ (kg ha⁻¹)** | 15             |

Table 2 Leaching behaviour of novaluron in sandy loam soil and in leachates

| Soil depth (cm) | Novaluron Residues (µg)* | % Retention | T₂ (86.62 g a.i. ha⁻¹) µg ± SD | % Retention |
|-----------------|--------------------------|-------------|--------------------------------|-------------|
| 0-10            | 1.84±0.03                | 56.97       | 3.60±0.28                      | 55.72       |
| 10-20           | 0.52±0.02                | 16.10       | 1.09±0.06                      | 16.87       |
| 20-30           | 0.20±0.03                | 6.19        | 0.35±0.03                      | 5.42        |
| 30-40           | 0.06±0.01                | 1.90        | 0.12±0.01                      | 1.86        |
| 40-50           | BDL                      | BDL         | BDL                            | BDL         |
| 50-60           | -                        | -           | -                              | -           |
| Leachate        | -                        | -           | -                              | -           |
| **Total recovered** | **81.16**             |             | **79.87**                      |             |

*Average residues of three replicates

Table 3 Leaching behaviour of indoxacarb in sandy loam soil and in leachates

| Soil depth (cm) | Indoxacarb Residues (µg)* | % Retention | T₂ (74.26 g a.i. ha⁻¹) µg ± SD | % Retention |
|-----------------|---------------------------|-------------|--------------------------------|-------------|
| 0-10            | 2.06±0.04                 | 74.36       | 4.00±0.03                      | 72.20       |
| 10-20           | 0.38±0.04                 | 13.71       | 0.61±0.06                      | 11.01       |
| 20-30           | 0.13±0.03                 | 4.69        | 0.29±0.03                      | 5.23        |
| 30-40           | 0.02±0.01                 | 0.72        | 0.03±0.01                      | 0.54        |
| 40-50           | BDL                       | BDL         | BDL                            | BDL         |
| 50-60           | -                         | -           | -                              | -           |
| Leachate        | -                         | -           | -                              | -           |
| **Total recovered** | **93.48**             |             | **88.98**                      |             |

*Average residues of three replicates
Diuron concentrations in water collected in the lysimeters varied in detected samples from 0.02 to 6.29 μg L⁻¹. The average mass of leached diuron in the lysimeters was 9.013 μg, representing 0.08% of the amount applied. Losses of 0.02 and 0.54% of the applied amount by leaching and runoff, respectively were reported.

Dissipation studies of diflubenzuron, flufenoxuron and novaluron and their effects on bacterial diversity in soils were carried out by Hsiao et al., (2013). The proportion of residual benzoylureas in sterilized soils remained up to 83% at the end of the incubation, which implied that the dissipation was mainly by microorganisms. All three benzoylureas were not detected below 10 cm in soil column experiments. Comparison of initial pesticides concentrations (50 mg kg⁻¹), diflubenzuron was detected at <1%. However, flufenoxuron and novaluron remained at >30% and 50% in Pu and Wl soil, respectively after leaching for 70 d. According to Garcia et al., (2006), pesticides recoveries ranged between 92.3 and 109.5%.
This methodology was used to determine benzoyleureas in ground water samples at levels lower than 0.1 µg/L.

Campbell et al., (2005), determined that indoxacarb have strong binding with soil tested after 30 days. According to findings of Sun et al., (2012), field trials indicated that the dissipation of indoxacarb enantiomers followed first-order kinetics in soil at two locations. The half-lives of two enantiomers in soil range from 23 to 35 d. The changes of enantiomeric fraction values proved that enantioselective degradation of indoxacarb happened in soil. From above discussion it is clear that very low amount of novaluron as well as indoxacarb residues were detected in soil and water samples in various experiments which were in agreement with our result.

From the results it was concluded that residues were not detected after 40 cm depth of soil in both the cases and none of the residues of combination product (Novaluron + Indoxacarb) were found in leachates, hence it may be safe for both soil as well as ground water.

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