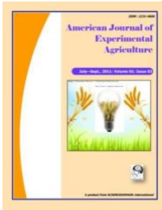

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Distribution of Imidacloprid Residues in two Sandy Soils of Portneuf (Quebec) Under Potatoes (*Solanum tuberosum*) Culture

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Authors' contributions

This work was carried out in collaboration between all authors. Author NB designed the study, wrote the protocol and performed the statistical analysis. Author AZ designed the study and managed the analyses of study. Author NDJP wrote the protocol, wrote the first draft of the manuscript and managed the literature searches. Authors KTW and KNA managed the literature searches. All authors read and approved the final manuscript

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ABSTRACT

Aims: The goal of this work was to confirm the results of leaching and dissipation of the imidacloprid obtained with intact soil columns and draining lysimeters.
Methodology: A study on the distribution of imidacloprid (insecticide) residual deposit was carried out on two potatoes (*Solanum tuberosum*) farms of Portneuf (Quebec). Two farms (PNF47 and PNF51) were selected. The soil was sampled once before and four to five times after imidacloprid application. Imidacloprid residues analysis was performed using HPLC.
Results: The results obtained show that the average concentration of imidacloprid after application is lower or equal to the theoretically expected values. The imidacloprid was recovered in the soil 2 to 6 days after foliar application at rates varying between 23.2 to 23.6%. The heterogeneity of active

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ingredient distribution on the soil at application, differential dissipation of this one and foliar interception explain these results. The coefficients of variation of the concentrations observed in the layer 0 to 5cm, 2 to 6 days after application vary from 76 to 96%. The soil contamination after harvest is 2 to 6% of the amount of imidacloprid applied. The vertical distribution profiles of the residual concentrations of imidacloprid after application show that, these vertical profiles of distribution vary from a sampling point to another. The residual concentrations in the deep layers of the soil are independent of the concentrations in the surface layers (absence of correlation).
Conclusion: The risk of pesticide leaching below the root zone of the soils studied is small in the context of crop rotation. But there is no zero risk. The data obtained can be used in the development, calibration and validation of the various models of digital simulation.

Keywords: Imidacloprid; soil; potato; residues distribution; dissipation; leaching; soil columns.

1. INTRODUCTION

One of the principal problems of the leaching and dissipation of pesticides in the field study is the great variability of residual deposit at application and its dissipation with time [1]. This space variability of the residual concentrations results from the lack of uniformity in pesticides application, variability of soil properties and vegetable biomass [2-4]. The heterogeneity of distribution of the active ingredient in field can also result from errors of calibration of certain pulverizers used in potatoes farms of Portneuf, erroneous mixtures, variability of the advance speed of equipment and overlapping of application bands [5,6].

Imidacloprid was sold and used for the first time in Canada in 1995 for the control of the Colorado potato beetle in the eastern of Canada. Nowadays, imidacloprid is an insecticide with active ingredient used to control sucking insects, such as aphids, leafhoppers, psyllids, thrips, whiteflies and beetles in agricultural crops, to control white grubs in lawns and turfgrass, as well as to control domestic pests such as fleas and cockroaches [7]. Imidacloprid has a toxic aspect. It is neurotoxicity to human [8] and as consequence of its high toxicity mainly to bees, and beneficial predatory, its use was restricted or banned in many countries, such USA. However, in Canada as in many other countries, imidacloprid is still being largely used as insecticide on several crops without any restriction. EPA (US environnemental protection agency) placed imidacloprid in category I as having the highest leaching potential [9]. The knowledge of the space variability of the residual concentrations of pesticides in farms is of particular interest for digital simulation models of deterministic type which use average values relating to a given surface as entry variables. Numeric models of simulation development require a combination of theoretical concepts and empirical data resulting from laboratory and field studies [3]. These empirical data; particularly those obtained from field reality are used for the confirmation of digital simulation models. The numerical and empirical approaches in the study of pesticides leaching and dissipation in the soil profile are thus complementary. The difficulties and the cost associated with pesticides residues analysis justified the development of digital simulation models which require data resulting from farming reality for their calibration and validation [10,11]. All these reasons determined the choice of the empirical approach adopted in this study to evaluate leaching and time-space variability of the residual concentrations in the soil profile. The imidacloprid which is an insecticide widely adopted by farmers and having a high leaching potential according to the Gustafson index was retained as reference molecule.

This work followed upon the detection of pesticides residues (metribuzine, aldicarbe, carbofuran, atrazine and linuron) in wells located in the vicinity of potato farms in the counties of Portneuf and Lanaudière [12]. Some of these pesticides as imidacloprid, had a high leaching potential according to Gustafon index [13]. It contributes to elucidate the origin of this contamination (accidental discharge or leaching beyond the rhizosphere of treated farms) and confirm the results of leaching and dissipation of the imidacloprid obtained with intact soil columns and draining lysimeters. In fact, in a previous experimentation, undisturbed soil columns (65cm high X15 cm diameter) were used in a greenhouse experiment to study the effect of rainfall and mode of application on leaching of imidacloprid. These results showed that imidacloprid was leached in trace amount below 65cm soil columns. Under field conditions, imidacloprid was detected in percolated water below 1m depth lysimeters at concentration less than 3µg/L [14]. In a specific way, the study aims to evaluate the homogeneity of the residual deposits after application; the vertical movement and dissipation of the imidacloprid; and the concentration variability in the soil profile from one point of the farming-field to another at various moments after application.

2. MATERIALS AND METHODS

2.1 Study Farms Description

Two farms (PNF47 and PNF51) were selected in the county of Portneuf located at 25km in the west of the town of Quebec (Canada). The farming succession and cultivated varieties are described in the Table 1. The various cropping operations and phytosanitary treatments were carried out by the farmers according to their usual practice, in order to be able to collect data reflecting as much as possible realities.

2.2 Rainfall and Soil Properties (Characteristics)

The monthly data of Rainfall (except for July with exceeding Rainfall) are comparable with the average monthly data over one 26 years period (Fig. 1). In the Portneuf County, potato is cultivated on sandy soils. The soils of the two selected farms belong to the Morin series.

Soil texture is sandy. Sand content varied from 78% to 90% in the cultural horizon. This content is 100% in the deeper strata. The silt content ranges from 8% to 16% in the surface layers. The clay content is negligible and does not exceed 1% in the 0-35 cm strata. The gravel content ranges from 3% to 20%. The pH varied from 4.7-5.8 and the CEC was 4-7 cmol+/kg. The OM ranged from 3.1 to 5.4% in the surface layer and less than 1% in the deeper strata (50-100cm).

2.3 Pesticides Choice and Application

Imidacloprid was selected on the basis of its adoption by the farmers, its leaching potential and the need for knowledge on its behaviour in the sandy soils under potato culture of Quebec (Table 2). The imidacloprid (Admires® 240 g a.i.l^{-1}) in fluid paste form was applied in foliar treatment at the rate of 48 g ha^{-1} in the farms PNF47 and PNF51 respectively on July 6/17 and June 30. The foliar application took place approximately one month after the potato lifting.

2.4 Soils Sampling

Soil was sampled once before and four to five times after imidacloprid application. Twenty soil samples separated by distances of ten meters on the farms diagonal (surface of 1 and 3 ha) were taken at different depth (0-100cm). Rows sampling was retained to avoid the disturbances of soil in the between-rows because of cropping operations like weeding. The sampling points were marked with stakes at the time of the first passage after application. Subsequent samplings took place in a radius of 1m either on the same row, or on an adjacent row. All the samples were kept in a portable refrigerator containing ice during the sampling period and laboratory transport. In laboratory, the samples were then put drying at free air during 48 hours. After drying, the samples were sieved (sieve of 2mm) and were preserved at - 20ºC in plastic bags until extraction taking place less than two months later. During this period, preliminary test showed that this period did not lead to pesticide dissipation. The extracts were preserved at - 20ºC until the analysis.

The soils were sampled on 12 points of the diagonal of the field (area of 1 to 3 hectares) spaced 20 m. Strata sampled were 0-5 cm, 5-20 cm, 20-35 cm, 35-50 cm, 50-65cm, 65-80 cm, 80-100 cm. Soil samples were dried in the open air for 48 hours at the laboratory, sieved (2 mm screen) and stored at 20ºC until analysis. The particle size analyzes were performed using the densitometric method described by [16]. Analyses of organic matter content, pH and CEC were transferred to the laboratory of Agriculture-Canada land in Sainte-Foy (Québec). The analysis protocols were those described by [17].

Table 1. Description of farming succession, varieties and their yield in the two potatoes farms

Farm[a)]	Typical farming succession	Varieties	Plantation	Average Yield [c)] (t ha^{-1})
PNF47	2 years.P[b)]/1 year oats +mustard	Kennebec	21 may	11.4±3.8
PNF51	2 years.P/2years cereals + hay	Superior ronde	13 may	7.3±1.4

[a)]: the various farms belong to various producers; [b)]: P = potato [c)]: average dry yield for three plots per farm

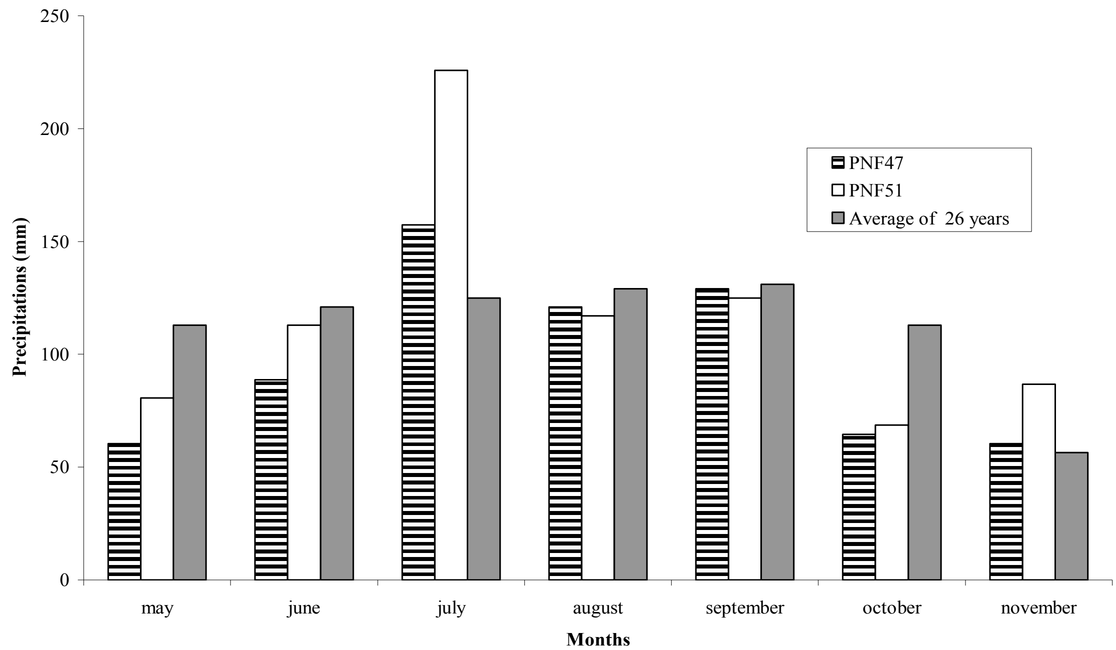

Fig. 1. Monthly precipitations of the two farms as well as the 26 years average for the May-November period

Table 2. Physico-chemical characteristics of Imidacloprid [15]

	Physico-chemicals characteristics
Commercial names	Admire®, Confidor®, Gaucho®, Premier®, remise®, provado®, Marathon®
Class	Insecticide
Active ingredient	Imidacloprid [1-(6-chloro-3-pyridimylmethyl)-Nnitroimidazolidine-2-ylideneamine]
Chemical family	Nicotine derivative
Formulation	Fluid paste
Application mode	Foliar pulverization
Solubility (mg/L)	510-750
Half-life in soil (days)	140-180
K_{oc}	160-400
GUS[a)]	High (H)

a): Gustafson Index of potential leaching = Log(half-life in soil) x (4-log(K_{oc}))

2.5 Imidacloprid Residues Analysis

The soil was dried by free air during 48 hours and extracted with acetonitril. A 10g soil sample in a screwing stopper tube of 50ml is extracted with 20ml of acetonitril during 30 minutes (160 rpm) in an automatic agitator (Standard Innova 2000). After sedimentation, 10 ml of the supernatant are transferred in a dry tube and evaporated under nitrogen jet in a water bath at 70ºC to dryness. The residue was dissolved in 1 ml of aqueous solution of sulfonic acid 1-pentane 5mM at pH 2.2, followed agitation and filtration through 0.45µm Nylon filter syringe (Acrodisc® 0.45µm) before injection in Water HPLC apparatus equipped with a Millennium 2010 control system. A WATERS™ 600 MS gradient solvent delivery system; a WATERS™ 717 automatic sampler, and a (PDA) WATERS™ 996 UV-detector. A spherisorb column ODS C-18 (4.6mm × 250 mm× 25 µm) was used for identification an quantification of imidacloprid using a gradient of elution of mobile phase constituted from aqueous solution (5mM) of sulphonic acid 1-pentane at pH 2.2 (APS) and tetrahydrofurane (THF) as organic solvents of the mobile phase. The sequence of the gradient used was 0, 10, 20, 23, 28min. Pump A (APS solution) sequence was 100, 90, 80, 100, 100% and Pump B (tetrahydrofurane) sequence was 0, 10, 20, 0, 0%. In these conditions, the Rt of imidacloprid was 9.86 min.

3. RESULTS AND DISCUSSION

The data obtained show that, in farm PNF47, imidacloprid was recovered until the layer 20-35 cm 6days, 12days and 23 days after application while at 113 days after application, imidacloprid was only recovered until the layer 5-20cm (Table 3). In farm PNF51, imidacloprid was detected in the layer 80-100 cm, 120 days after application. In the same farm, the pesticide was detected in the layer 65-80 cm 10 and 27 days after application (Table 4).

The observations done in different soil layers of farms as a function of sampling location and time after application showed that the vertical profile of residues distribution varied from one point to another in the farm diagonal at various sampling periods after application (Figs. 2 and 3). For example in the farm PNF47, 12 days after application of imidacloprid, in the layer 0-5 cm, we have about 10 ppb of imidacloprid in the position 1, about 20 ppb in the position 2, about 12ppb in the position 6 and 10 and 5 ppb in the position 19 (Figs. 2 and 3).

Imidacloprid is an insecticide widely adopted by farmers and has a high leaching potential according to the Gustafson index [13]. The residual quantities recovered after application are 23.2% of the applied mass, 6 days after application in farm PNF47 and 23.6% of the applied mass, 2 days after application in the farm PNF51. The insecticide is relatively mobile in the soil since it can be detected beyond 20 cm of soil, 2 days after application (Tables 3 and 4). Its dissipation in the surface layers of soil results from dilution of the active ingredient in the soil volume by in-depth migration. One can detect residues of the active ingredient until some 80-100 cm depths after harvest (Figs 2 and 3). Similar results were obtained by Bonmatin et al. [18] during the study of method of analysis of imidacloprid in soils, in plants and in pollens. These results showed that with the limit of detection 0.1 µg kg^{-1}, imidacloprid recovered in soils, plants and pollens at levels of a few micrograms per kilogram.

The residual deposit after application is heterogeneous. This residual deposit heterogeneity is reflected by the high values of the coefficients of variation of the average concentrations or by the high relative differences between minimum and maximum concentrations detected (Tables 3 and 4). The (Figs 2 and 3) show that the vertical profile of residues distribution varies from one point to another in the farm diagonal at various sampling moments after application. However, the contamination of the soil by imidacloprid residues remains insignificant after harvest.

Xie et al. [19] studied the interception of pulverization deposit of fenoxaprop and imazamethabenz on the oats plants (*Avena fatua*) according to environmental conditions. They obtained rates of interception varying from 68 to 89% for fenoxaprop and from 75 to 97% for imazamethabenz. The imidacloprid solubility is 510 mg l^{-1} and its leaching potential is considered high according to Gustafson index (1989) [13]. In New Brunswick and Prince-Edouard island, Julien et al. [20] studied the potential of imidacloprid to being transported by run off waters and to contaminate rivers adjacent to potato farms. The authors report concentrations of imidacloprid varying between 0.1 to 4.4 µg l^{-1} and 0.06 to 0.052 µg g^{-1} from rivers and from rivers sediments respectively [20]. These data confirm the tendency of the imidacloprid to be transported out of the application site by water movement. Felsot et al. [21]. Studied imidacloprid vertical distribution after application in a sandy loam soil of United States. They reported that residues of the insecticide were detected at 75 to 90 cm depth [21]. However, Rouchauld et al. [22] could not highlight an unspecified leaching of the imidacloprid beyond 20 cm under the depth of sowing of beet seeds (*Beta vulgaris*) treated with insecticide [22]. This result tends to show that the insecticide mobility in sandy soil after 48g/ha application depends on the cropping and environmental conditions but also on soil type (OM contents, clay content, pH), microbial activity, application doses, soil water content, soil temperature and history of use. This corroborates the results of Chris [23] and Chai et al. [24], who worked on similar experiments. In fact the experiment done by Chris [23] showed that the presence of vegetation had little effect on activity of imidacloprid in the soil. Results obtained by Chai et al. [24] in the study of the dissipation and leaching of acephate and chlorpyrifos showed that high dissipation of acephate was in part attributed to precipitation and preferential flow.

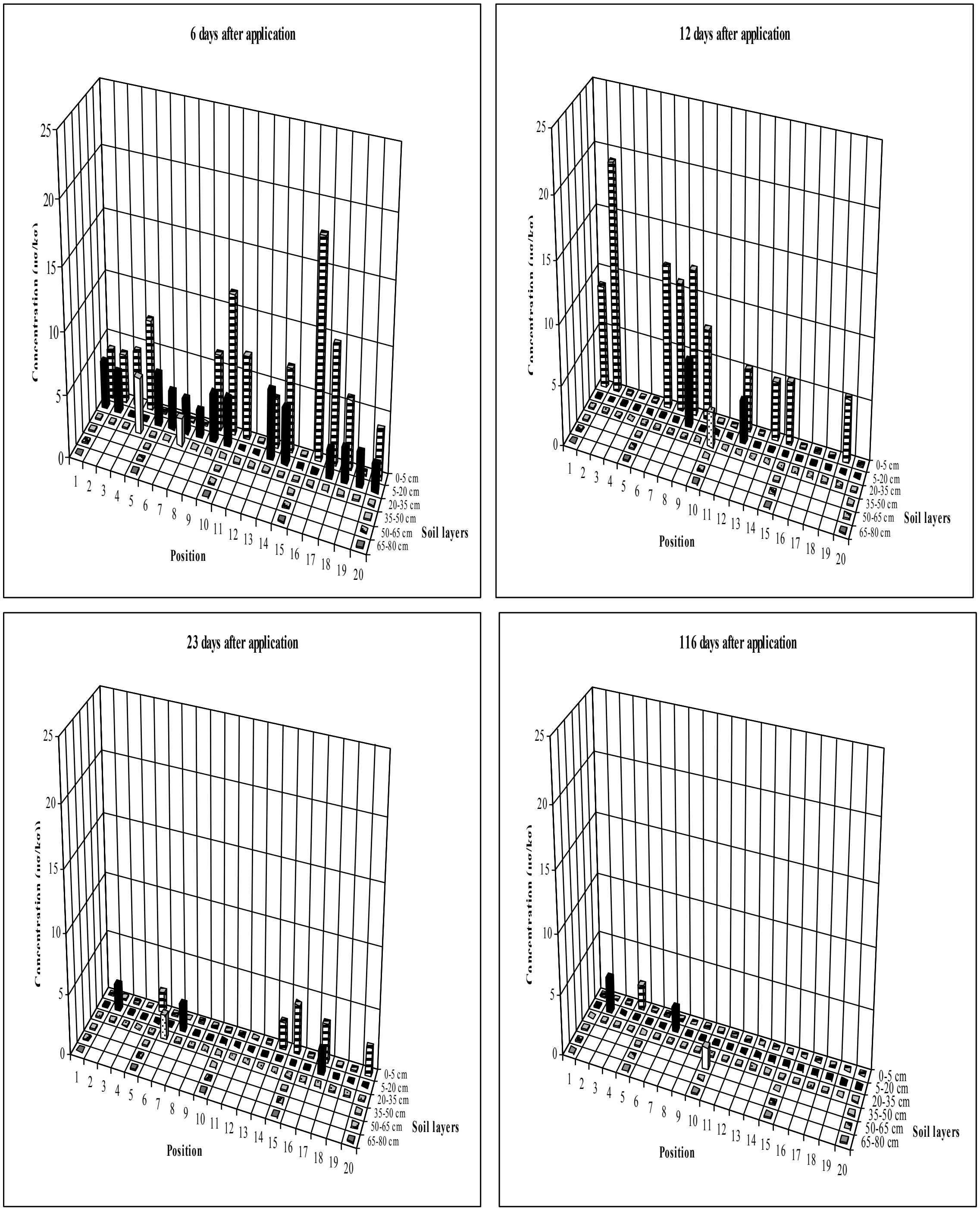

Fig. 2. Vertical distribution of imidacloprid residues (µg/kg) in different soil layers of farm PNF47 as a function of sampling position and time after application

Table 3. Statistical parameters[a] of imidacloprid[b] concentration variation in various soil layers at different days after application (DAA) in farm PNF47[c]

Soil layers (cm)	6 DAA				12 DAA				23 DAA				116 DAA			
	Min	Max	Av	C.V	Min	Max	Av	C.V	Min	Max	Av	C.V	Min	Max	Av	C.V
0-5	<2.2	9.6	5.3	96	<2.2	21.4	5.1	123	<2.2	4.6	0.8	175	<2.2	2.6	0.1	600
5-20	<2.2	6.2	2.6	73	<2.2	6.06	0.5	300	<2.2	4.6	0.5	180	<2.2	3.2	0.3	260
20-35	<2.2	5.0	0.6	250	<2.2	2.8	0.1	600	<2.2	2.2	0.1	500	<2.2	<2.2	<2.2	-
35-50	<2.2	<2.2	<2.2	-[d]	<2.2	<2.2	<2.2		<2.2	<2.2	<2.2		<2.2	<2.2	<2.2	-
50-65	<2.2	<2.2	<2.2	-	<2.2	<2.2	<2.2		<2.2	<2.2	<2.2		<2.2	<2.2	<2.2	-
65-80	<2.2	<2.2	<2.2	-	<2.2	<2.2	<2.2		<2.2	<2.2	<2.2		<2.2	<2.2	<2.2	-
80-100	<2.2	<2.2	<2.2	-	<2.2	<2.2	<2.2		<2.2	<2.2	<2.2		<2.2	<2.2	<2.2	-

[a]: Min. = minimum (μg kg^{-1}); Max. = maximum (μg kg^{-1}); Av. = average (μg kg^{-1}) of 20 samples for 0 – 35 cm layers and 5 samples for 35 – 80 cm layers; C.V. = coefficient of variation (%); the values lower than the limit of detection are considered null [b]: Limit of detection = 2.2 μg kg^{-1} [c]: Two applications of 48 g each, of a.i. ha^{-1} [d]: not estimated

Table 4. Statistical parameters[a] of imidacloprid[b] concentration variation in various soil layers at different days after application (DAA) in farm PNF51[c]

Soil layers (cm)	2 DAA				10 DAA				27 DAA				120 DAA			
	Min	Max	Av	C.V	Min	Max	Av	C.V	Min	Max	Av	C.V	Min	Max	Av	C.V
0-5	<2.2	28.4	9.4	76	<2.2	12.3	4.9	67	<2.2	2.2	0.1	500	<2.2	3.8	0.6	200
5-20	<2.2	7.4	1.7	129	<2.2	4.4	1.6	94	<2.2	3.4	0.9	133	<2.2	5.5	0.6	266
20-35	<2.2	2.2	0.2	300	<2.2	4.4	1.5	100	<2.2	3.8	0.4	260	<2.2	<2.2	0.3	266
35-50	<2.2	<2.2	<2.2	-[d]	<2.2	<2.2	<2.2	-	<2.2	<2.2	0.8	150	<2.2	2.2	0.8	150
50-65	<2.2	<2.2	<2.2	-	<2.2	<2.2	0.9	140	<2.2	<2.2	0.4	225	<2.2	2.6	1.4	92
65-80	<2.2	<2.2	<2.2	-	<2.2	<2.2	0.4	225	<2.2	<2.2	0.4	225	<2.2	2.8	1.4	95
80-100	<2.2	<2.2	<2.2	-	<2.2	<2.2	<2.2	-	<2.2	<2.2	<2.2	-	<2.2	2.6	1.4	92

[a]: Min. = minimum (μg kg^{-1}); Max. = maximum (μg kg^{-1}); Av. = average (μg kg^{-1}) of 20 samples for the layers of 0 – 35 cm and 5 samples for the layers of 35 – 80 cm; C.V. = coefficient of variation (%); the values lower than the limit of detection are considered null [b]: Limit of detection = 2.2 μg kg^{-1} [c]: Two applications of 48 g each, of a.i. ha^{-1} [d]: not estimated

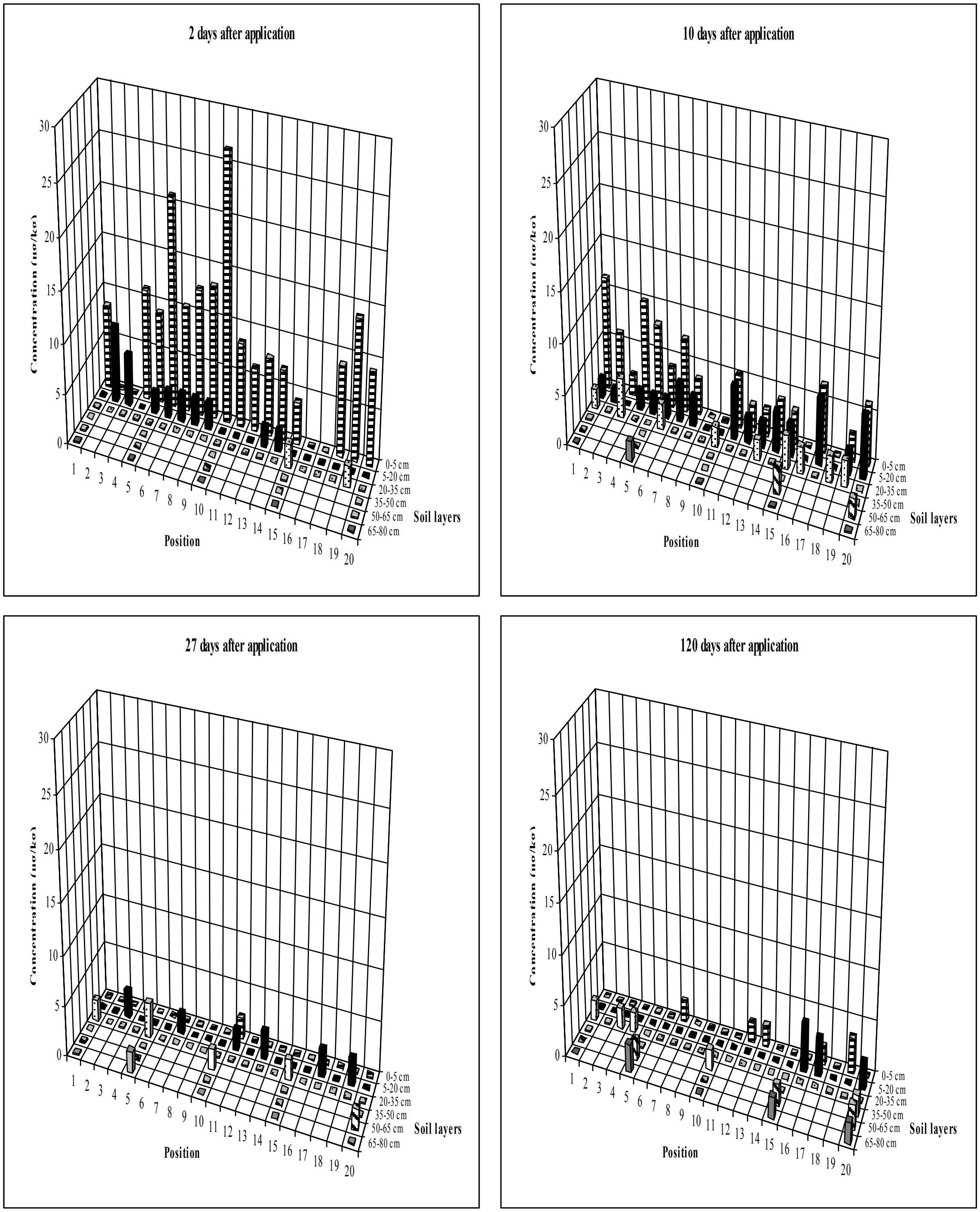

Fig. 3. Vertical distribution of imidacloprid residues (µg/kg) in different soil layers of farm PNF51 as a function of sampling position and time after application

4. CONCLUSION

The residual deposits of imidacloprid after application, in soil, are heterogeneous. This deposit heterogeneity is maintained or developed with time because of the dissipation and differentials migration of active ingredient from one point of the farm to another. The development of the simulation models of the imidacloprid behaviour in the soil must take account of the field reality. These results also call for certain moderation during field extrapolation of the results obtained in laboratory or station with relatively controlled and homogeneous conditions. The quantities of imidacloprid leached beyond the rhizosphere as their remanence after harvest does not seem to pose a relevant risk of pollution. The respect of adequate pulverization practices and cropping rotation can however decrease this risk considerably.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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