

First diet survey in Niger River valley and acute risk assessment for consumers exposed to pesticide residues in vegetables

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ABSTRACT

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To control pests and crops diseases, small scale farmers in the Niger River valley use a wide range of plant protection products which could induce harmful impacts on human health and environment. Dietary exposure to plant protection products residues was assessed in Niger River valley using the 24-hour recall method. Portion sizes were estimated using a collection of pictures previously prepared according to the local usual diet. A total of 45 samples of eight types of vegetables, representative of the most consumed in the study area (Niamey) during the dry hot and the dry cold season were collected. Samples were analyzed using a multi-residue method (QuEChERS) by gas chromatography-mass spectrometry (GC-MS/MS) and liquid chromatography-mass spectrometry (LC-MS/MS) that can detect more than 540 active ingredients. Residues of insecticides were detected in 64.4% of the analyzed samples. Among them, 26.7% contained residues above maximum residue limits (MRLs), 35.5% below MRLs, 2.3% of samples had residue equal to the MRLs. Chili peppers, tomatoes, moringas, head cabbages, sorrel leaves and peppers were the most contaminated vegetables. Their residue levels were, respectively, 4.6 mg/kg of chlorpyrifos-ethyl, 0.29 mg/kg of dichlorvos, 1.8 mg/kg of cypermethrin, 1 mg/kg of chlorpyrifos-ethyl, 0.46 mg/kg of acetamiprid and 0.41 mg/kg of dichlorvos. To evaluate the intake and characterize the risk level for adults and children, the EFSA PRIMO model spreadsheet (Pesticide Residue Intake Model) was used. The exposure results based on consumptions at the 97.5th percentiles show that the highest predicted exposure values for a short-term intake (PSTI) was obtained in the case of consumption of head cabbages (532% of ARfD) for adults and tomatoes (1052% of ARfD) for children. Whatever the product, the risk of exposure was higher for children than for adults for all detected residues.

Keywords: acute exposure, consumers, Niger River valley, pesticide residues, risk assessment

Agriculture represents in Niger the main economic sector and participates

for approximately 40% in the country's Gross Domestic Product (GDP) (INS-Niger 2014). With an average population growth of 3.4%, one of the world's highest, the Niger population was estimated to 17,140,000 inhabitants. This average population growth largely exceeds

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the annual growth rate of agricultural production of the country estimated to 2.2% in recent years (INS-Niger 2010). With this significant food deficit, the Nigerien population is practicing increasingly horticulture in the Niger River valley and its tributaries.

In Niger River valley, horticulture is an important source of agricultural growth and poverty reduction in Niger. In fact, this activity is one of the sectors chosen by the country's policy to support food security and to fight against hunger. The horticulture sector is also an alternative proposed by the United Nations to face the challenges of urbanization (Perrin et al. 2014). The main vegetable crops are onion (*Allium cepa*), Chili pepper (*Capsicum annum*), eggplant (*Solanum melongena*), head cabbage (*Brassica oleracea*), lettuce (*Lactuca sativa*), moringa (*Moringa oleifera*), pepper (*Capsicum annum*), sorrel leaves (*Hibiscus sabdarifa*), tomato (*Lycopersion esculentum*) carrot (*Daucus carota*), cucumber (*Cucumis sativus*), okra (*Abelmoschus esculentus*), etc. However, they are subject to significant pest and disease pressure requiring the use of control methods. Chemical control often uses huge treatments of plant protection products (PPPs) (Andres et al. 2011; Illyassou et al. 2015).

PPPs contain at least one active ingredient and are mainly used to control pests and crop diseases (EU-database 2018). The active ingredients can be chemicals, plant extract, pheromone or micro-organisms including viruses (EC 2009). When they are correctly used, they have clearly shown their benefits in improving horticultural yields. Nevertheless, a possible consequence of their misuse can be the presence of residue levels in the treated products often exceeding Maximum Residue Limits (MRLs), and could induce negative

impacts on human health (Bhanti et al. 2007; Caldas et al. 2004; Nougadère et al. 2012). The presence of these residues in vegetables can be the consequence of poor dosage calculation, poor spraying technique, poor formulations, and repeated applications of the same active ingredient or the non-compliance to the pre-harvest interval (PHI). In recent years, chemical families of some active ingredient are known to have potential adverse health effects. Several diseases such as Parkinson disease, cancers or Alzheimer's could result from chronic exposure to some PPPs formulations (Chourasiya et al. 2015; Darko et al. 2008; Richard et al. 2014).

In order to protect consumer's health and assess the risks related to the use of PPPs, several methods and models have been used in current years in many countries over the world (Machera et al. 2003; Toumi et al. 2016). Risk assessment is a scientific process which consists in four steps: (i) hazard identification, (ii) hazard characterization, (iii) exposure assessment, and (iv) risk characterization. Risk assessment for consumers exposed to PPPs residues in beverages and food, is evaluated by comparing acute and chronic exposure values obtained from risk models to appropriate toxicological reference values (ARfD or ADI, respectively). Average concentrations of residues in food as well as usual and maximal consumptions are needed to estimate the risk level.

Unfortunately, few data are today available in Niger regarding the concentrations of residues in vegetables produced in the Niger River Valley. Therefore, a study was designed to monitor the levels of plant protection product residues in vegetables commonly consumed in this study area and to assess the risk level for different consumer groups, especially adults and children.

MATERIALS AND METHODS

Study area description.

This study was conducted in Niger River valley (Fig. 1), covering the entire study area (region of Niamey). The Niger River valley is the principal surface water resource of the country and therefore this region is one of the most populated. It covers the entire region of Niamey and located between 02°10' East longitude and 13°35' North latitude. It is the third largest river in Africa for its length (4,200 km) and its area of drainage basin (2,100,000 km²). It crosses the Niger territory over a length of 550 km. Entirely isolated in

Tillabery department, the river in Niamey is located in the West of the country where rainfall largely exceeds the national average. The banks of the river have a large capacity of agricultural production, especially horticulture.

Ilyassou et al. (2017) recently performed a study on farmers' practices toward plant protection. They have identified the use of 25 active ingredients including 17 insecticides, 4 selective herbicides and 4 fungicides. The use of various counterfeit pesticides cocktails was also reported.

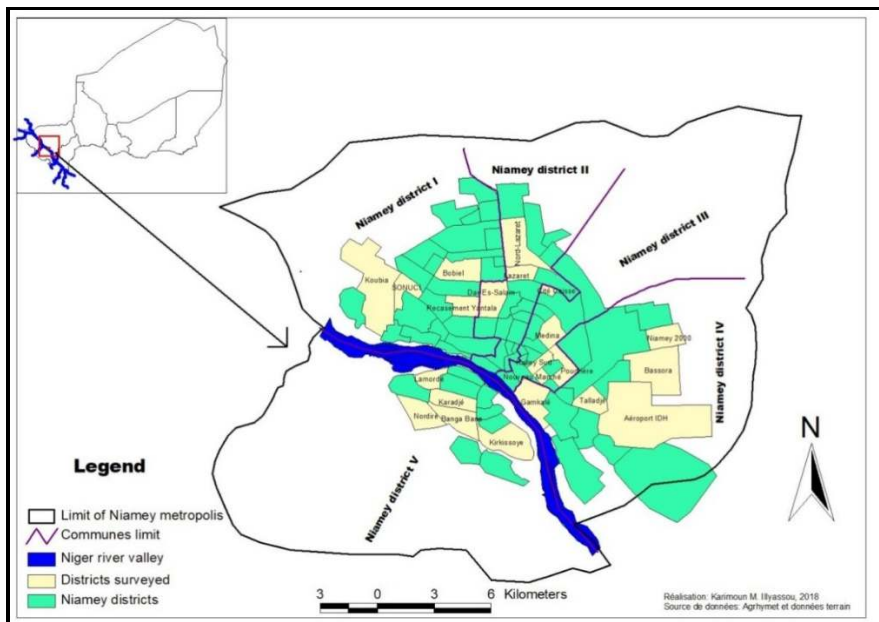


Fig. 1. Study area and municipal stratification of the Niamey region (framed in the figure shows the map of Niger)

Food consumption survey.

To collect reliable and representative food consumption data, a household survey based on European Food Safety Authority (EFSA) guidelines was conducted in Niamey metropolis (EFSA 2014). According to the National Statistics

Institute (NSI) of Niger, Niamey population was estimated to 1,026,848 inhabitants in 2012 and included 166,998 households. The population size for the consumption survey was determined according to the formula by Charan et al. (2013) using a 10% probability for cross

sectional studies about quantitative variables. A total of 100 households (twenty households in each district of Niamey) were interviewed (n = 571 persons). The formula is:

$$n = \frac{1.96^2 * N}{1.96^2 + (2e)^2 (N-1)}$$

where, 'N' is the household's number of Niamey city (166,998), 'e' is the margin of error (10%).

The choice of Niamey is explained by its position on the Niger River and high consumption of vegetables by its inhabitants, compared to the other urban inhabitants of the country (31.5 against 29.0 kg/person/year) (Andres et al. 2012). Using the 24-hour recall method (Gibson et al. 1999), the data collection was conducted with a questionnaire on vegetables and fruits locally produced and consumed in the Niger River valley. Portion sizes were estimated using a collection of pictures previously prepared according to the local usual diet, and percentiles were then calculated. According to the guidelines of Gibson et al. (1999), the list of the raw ingredients of each mixed dish was dressed and the total weight of the mixed dish was estimated. The proportions of each vegetable in all household mixed dishes were calculated and then used to estimate the consumption weight of each ingredient. A digital balance was used for the determination of the average body weight (bw) of the surveyed population.

Vegetables sampling.

In order to take into account the variability and the availability of vegetables produced in the Niger River valley, two sample collections were carried out during two different seasons of production, in Niamey and the surroundings. Thirty samples, representative of the most consumed

vegetables in Niamey were collected from 22 to 23 August 2017 during the dry hot season. Therefore, 15 samples of most consumed vegetables during the dry cold season were collected from 2 to 3 February 2018. Samples included Chili pepper, eggplant, head cabbage, lettuce, moringa, pepper, sorrel leaves and tomato. Vegetable samples (each sample consists of 4 subsamples) were collected randomly downstream of the watershed. According to the guidelines and principles of European Directive 2002/63/CE for the establishment of MRLs in foodstuffs, samples of 1 kg at the precise time of harvest were packed in papers, labeled and weighed. Fresh (unfrozen) samples were immediately sent by plane to a Belgian laboratory for analysis in the following hours. All useful information such as sample number, origin, sampling date, last treatment, pesticide applied, etc. were taken for each sample.

Pesticide residues analysis

Pesticide residues analysis was carried out by an accredited laboratory PRIMORIS (Technologiepark 2/3, 9052 Zwijnaarde-Gent, Belgium). It holds a BELAC accreditation to ISO/CEI 17025 for pesticide residues analysis on vegetables and herbal products in general. The method used for vegetables analysis was based on QuEChERS (Quick Easy Cheap Effective Rugged Safe) multi-residues method for pesticides in foodstuffs. QuEChERS is a separation method based on work done by the U.S. department of Agriculture research center in Wyndmoor (Anastassiades et al. 2003; Toumi et al. 2017). The QuEChERS method used by PRIMORIS helps to screen over 540 active ingredients and their metabolites. All active ingredients and their metabolites can be detected and quantified on harvested products. However, some ingredients which require

specific analytical methods, such as glyphosate or dithiocarbamates, were not investigated by this method but those chemicals are never used on vegetables in the study area.

The residues in extracted samples were identified and quantified by Gas Chromatography coupled with tandem Mass Spectrometry (GC-MS/MS) for small, thermally stable, volatile, non-polar molecules or by Liquid Chromatography coupled with tandem Mass Spectrometry (LC-MS/MS) for larger, thermolabile, non-volatile and polar molecules (Lehotay *et al.* 2007). The limit of quantification (LOQ) for all residues identified in extracted samples is 0.01 mg/kg.

Exposure Risk assessment.

To estimate the exposure level for different groups of consumers, the Predicted Short-Term Intake (PSTI) was used. The PSTI values were calculated with The EFSA PRIMO (Pesticide Residue Intake Model) model version 11 (RASFF 2016). The risk of each active ingredient detected in vegetable was characterized by comparing the PSTI value expressed in mg/kg bw/day to the Acute Reference Dose (ARfD) values obtained from the EU-Pesticides database (Table 1). There is a risk for consumers when the PSTI value of a given active ingredient is higher than the corresponding ARfD value.

Table 1. Toxicological reference values (TRVs) of active ingredients detected in the vegetable samples, the corresponding Acute Reference Dose (ARfD) values, the Acceptable Operator Exposure Level values (AOEL) and the Acceptable Daily Intake (ADI) values are expressed in mg/kg bw/day

Active ingredient	ARfD	AOEL	ADI	Source of TRVs
Acetamiprid	0.100	0.07	0.07	EC 2004
Allethrin	NA	NA	NA	NA
Chlorpyrifos-ethyl	0.005	0.001	0.001	EFSA 2014
Cypermethrin	0.040	0.06	0.02	JMPR 2006
Dichlorvos	0.002	0.0005	0.00008	EFSA 2006
Dimethoate	0.010	0.001	0.001	EFSA 2013
Emamectin	0.010	0.0003	0.0005	EFSA 2012
Fenitrothion	0.013	NA	0.005	EFSA 2006
Imidacloprid	0.080	0.08	0.06	EC 2008
Lambda-cyhalothrin	0.005	0.00063	0.0025	EU 2016

NA: Not Available

Statistical analysis.

All data collected during the survey (from the questionnaires) as well as the predictable exposure values were analyzed using Origin version 6.0 and/or Excel 2007 software.

RESULTS

Socio-demographic characteristics of the study population.

The 100 households interviewed were composed of 310 women and 261 men. People aged 18-39 were the most numerous with 46.4% of the population

(Fig. 2). According to the NIS of Niger (2016), the age distribution found in the population sample was estimated

sufficiently representative of the population of Niamey and therefore no quota method was applied.

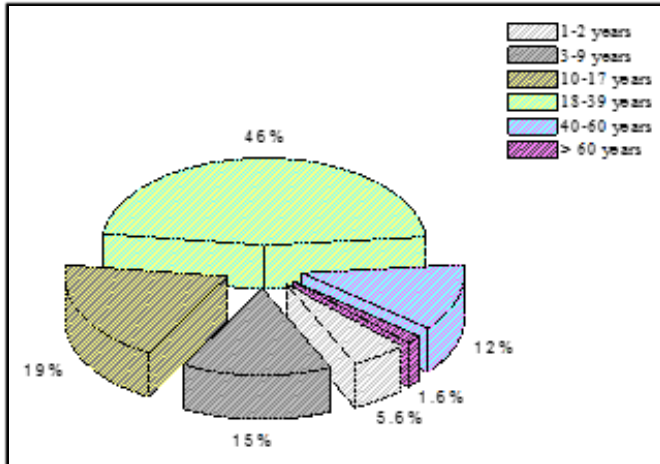


Fig. 2. Age classes of the surveyed population (n = 571) in the Niamey region

The average body weight (bw, used later in the PSTI calculation) of the population was 67 ± 10 kg and 30 ± 14 kg, respectively, for adults and children. The mean frequency of consumption for all the surveyed population was considered equal to two times per day. For the representativeness of the studied population, children aged 1-2 years are excluded for daily intake estimation.

Consumption survey results.

The 97.5th percentiles of consumption (or LP, used later in the PSTI calculation) for the studied population are shown in Table 2. This percentile was chosen as the most extreme value that can be estimated with any reasonable degree of certainty. Thus, are considered as big consumers both adults and children which

consume the 97.5th percentile of each vegetables analyzed. For children, daily consumption was estimated on the assumption that each member of the household participating in the family dish should consume a proportional share to his needs.

Plant Protection Product (PPPs) residues in analyzed vegetables.

PPPs residues were analyzed in 45 vegetable samples. Out of these samples, 29 (64.4%) have active ingredient residues while 16 (35.6%) have no residues ($< LOQ = 0.01$ mg/kg). The percentage of the contaminated samples was important for all the analyzed vegetables except for eggplant and tomato samples (Table 3).

Table 2. The 97.5th percentiles of vegetables consumption for adults and children in Niamey metropolis

Vegetable	97.5 th percentile (g/person/day)	
	Adults	Children
Chili pepper	4.0	2.2
Eggplant	177.2	99.2
Head cabbage	356.5	199.7
Lettuce	221.9	124.3
Moringa	141.7	79.4
Pepper	8.6	4.9
Sorrel leaves	69.0	38.6
Tomato	310.9	174.1

Table 3. Residues detected in vegetables from Niger River valley

Vegetable	Average unit weight (g)	Number of analyzed samples	Samples with residues	
			Number	%*
Chili pepper	4 ± 1.7	5	4	8.9
Eggplant	101 ± 31.7	5	1	2.2
Head cabbage	524 ± 24.4	5	5	11.1
Lettuce	76 ± 28.2	6	5	11.1
Moringa	8 ± 14.4	6	4	8.8
Pepper	29 ± 14.4	6	5	11.1
Sorrel leaves	4 ± 1.9	6	4	8.8
Tomato	64 ± 32.0	6	1	2.2
Total		45	29	-

*: % of the total number of samples

Only insecticides residues were detected above the LOQ. Among the detected residues, chlorpyrifos-ethyl was found with the highest average concentration (1.8 mg/kg) in Chili pepper, followed by cypermethrin (1.6 mg/kg) in

moringa. λ -cyhalothrin and acetamiprid residues were the most predominant active ingredients found in the 45 vegetable analyzed samples (Table 4).

Table 4. Average concentrations of residues with variation and range, detected in each commodity; Limit of quantification (LOQ) = 0.01 mg/kg

Commodity	Active ingredient detected	n	Average concentration (mg/kg)	Range (min-max) (mg/kg)
Chili pepper	Emamectin-benzoate	2	0.012	0.011-0.013
	Chlorpyrifos-ethyl	3	1.799	0.017-4.6
	λ -Cyhalothrin	4	0.121	0.051-0.23
Eggplant	Chlorpyrifos-ethyl	1	0.400	<LOQ-0.40
Head cabbage	Fenitrothion	3	0.035	0.014-0.070
	Imidacloprid	1	0.014	<LOQ-0.014
	λ -Cyhalothrin	5	0.116	0.056-0.26
	Emamectin-benzoate	1	0.039	<LOQ-0.039
	Chlorpyrifos-ethyl	1	1.000	<LOQ-1.00
Lettuce	λ -Cyhalothrin	5	0.049	0.015-0.13
	Acetamiprid	1	0.100	<LOQ-0.10
Moringa	Allethrin	1	0.012	<LOQ-0.012
	Acetamiprid	4	0.093	0.010-0.21
	λ -Cyhalothrin	2	0.114	0.049-0.18
	Cypermethrin	2	1.600	1.4-1.8
Pepper	λ -Cyhalothrin	5	0.096	0.02-0.3
	Cypermethrin	2	0.029	0.019-0.039
	Dichlorvos	1	0.410	<LOQ-0.41
	Acetamiprid	4	0.063	0.014-0.2
	Imidacloprid	5	0.114	0.031-0.24
	Dimethoate	1	0.011	<LOQ-0.011
Sorrel leaves	Acetamiprid	3	0.313	0.12-0.46
	λ -Cyhalothrin	4	0.188	0.095-0.26
	Cypermethrin	1	0.024	<LOQ-0.024
	Dimethoate	1	0.013	<LOQ-0.013
Tomato	λ -Cyhalothrin	1	0.020	<LOQ-0.02
	Dichlorvos	1	0.290	<LOQ-0.29
	Acetamiprid	1	0.038	<LOQ-0.038

Out of the analyzed samples that had residues, more than a half (25 samples) was contaminated with λ -cyhalothrin residue followed by acetamipridone (Fig. 3). Residues levels of imidacloprid and cypermethrin were detected in six and five vegetable samples, respectively. A rate of 90% of the detected

active ingredients is authorized in Niger by the Sahelian Pesticides Committee (CSP 2017). Only dichlorvos active ingredient is banned for use in Niger. All detected residues (100%) are insecticides. Organophosphorus insecticides represent 40% of the residues followed by pyrethroids (30%) and neonicotinoids (20%).

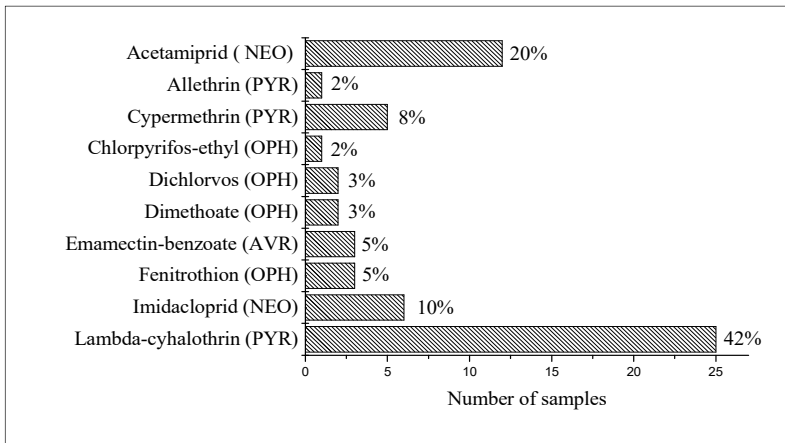


Fig. 3. Active ingredient residues detected in all vegetable samples in the Niamey region (OPH: organophosphorus, NEO: neonicotinoids, PYR: pyrethroids, AVR: avermectin).

In terms of the co-presence of the plant protection product residues, the combination of one or two and more than three insecticides from various chemical families was frequent. In fact, some samples contain multiples residues. Out of

positive samples, 49% contained more than one residue (Fig. 4). A rate of 27% of samples contains two residues and three residues were detected in 18% of the samples. The highest number of residue was found in pepper samples (up to 6).

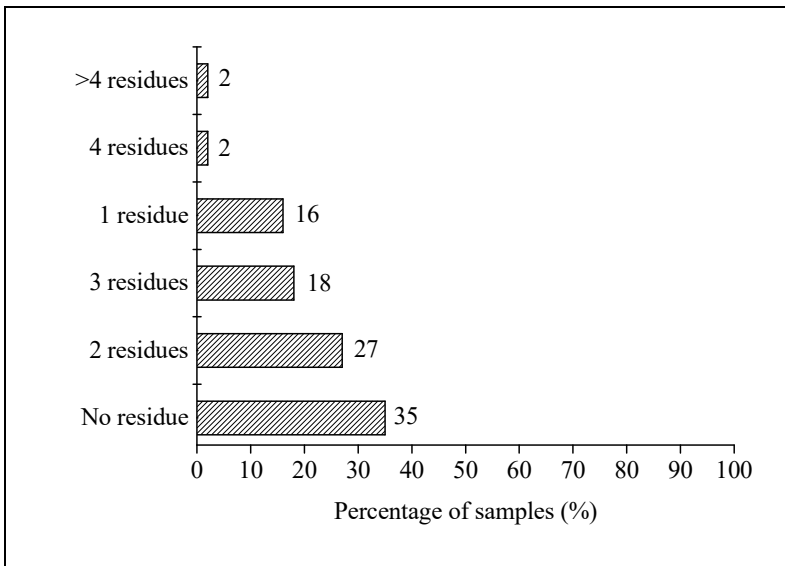


Fig. 4. Percentage of samples with multiple residues in the Niamey region.

Exceeding of maximum residue limits (MRLs).

Among the 45 analyzed samples, 12 contained residues exceeding the MRLs, while 16 contained residues below the MRLs set by European Union. As shown in Table 5, the MRL exceedances appeared particularly critical for chlorpyrifos-ethyl and dichlorvos, respectively, in Chili pepper and pepper.

Taking into account the multiple residues detected in several samples, the exceedances of MRLs per active ingredients were 16 and concern 6 active ingredients: acetamiprid (n = 3), chlorpyrifos-ethyl (n = 4), dichlorvos (n = 2), emamectin (n = 1), fenitrothion (n = 3) and λ -cyhalothrin (n = 3).

Table 5. Concentration of pesticide residues above the EU-MRLs in the vegetable samples

Vegetable	Active ingredient	Concentration (mg/kg)	EU-MRL (mg/kg)	Concentration (% of MRL)
Chili pepper	Chlorpyrifos-ethyl	0.017	0.01	170
Chili pepper	λ -Cyhalothrin	0.230	0.10	230
Chili pepper	Chlorpyrifos-ethyl	0.780	0.01	7800
Chili pepper	Chlorpyrifos-ethyl	4.600	0.01	46000
Chili pepper	λ -Cyhalothrin	0.110	0.10	110
Head cabbage	Emamectin	0.039	0.01	390
Head cabbage	Fenitrothion	0.022	0.01	220
Head cabbage	Fenitrothion	0.070	0.01	700
Head cabbage	Chlorpyrifos-ethyl	1.000	0.05	2000
Head cabbage	Fenitrothion	0.014	0.01	140
Pepper	λ -Cyhalothrin	0.300	0.10	300
Pepper	Dichlorvos	0.410	0.01	4100
Sorrel leaves	Acetamiprid	0.120	0.05	240
Sorrel leaves	Acetamiprid	0.460	0.05	920
Sorrel leaves	Acetamiprid	0.360	0.05	720
Tomato	Dichlorvos	0.290	0.01	2900

Acute dietary exposure and consumers risk assessment.

The exposure (predicted short-term intakes or PSTI) to residues of plant protection products was estimated with the EFSA PRIMO model version 11, an excel-based calculation spreadsheet used to perform the dietary risk assessment for pesticide residues at EU level (RASFF 2016) in which the unit weight of vegetables (U), the body weight of adults and children (bw) and consumptions (LP

of adults and children) were introduced (data collected thanks to the survey).

In this study, the exposure was estimated only for samples in which residues were above the limit of quantification (LOQ). Table 6 summarizes the nature of the detected active ingredients and their concentrations expressed in mg/kg in the vegetables, the calculated PSTI values expressed in mg/kg bw/day, the ARfD values and the PSTI values expressed as a percentage of ARfD

for adults and children consumers. The results based on consumption at the 97.5th percentile show that the most ARfD exceedance was found for children and only 4 exceedances were identified for adults. Among the residues detected, 4 active ingredients (organophosphorus (2) and pyrethroids (2)) were found to have an acute risk for adults and children. These active ingredients (dichlorvos, λ -cyhalothrin and chlorpyrifos-ethyl) have the lowest ARfD values. They are the most toxic in case of ingestion for adults and children. For all analyzed vegetables, the highest predicted exposure value for a short-term intake is obtained both for adults and children in the case of head cabbage consumption.

DISCUSSION

A total of 45 vegetables samples were collected at the fields in Niger River valley and analyzed. More than 64% of the samples contained plant protection product residues. The residues of 10 insecticides, belonging to 4 chemical families (organophosphorus, neonicotinoids, pyrethroids, and avermectins) were detected. The high frequency of detection pointed high levels of some pesticide residues detected in fruits, cereals and vegetables foodstuffs in West Africa (Bempah et al. 2011; Bempah et al. 2012; Kolani et al. 2016; Manirakiza et al. 2003; Ogbeide et al. 2016). It is partially in accordance with a study conducted in Brazil from 2001 to 2010 on pesticide residues in foodstuffs which highlighted that among 1,154 tomatoes and 1,007 lettuces analyzed samples, pesticide residues (\geq LOQ) were found in 59.8% and 33.9% of samples, respectively (Jardim et al. 2012). In another study,

among 724 analyzed samples (fruits and vegetables) collected from 8 South America countries, 72% of samples contained pesticide residues at or below MRL and 19% of the samples were found free of residues (Hjorth et al. 2011).

In the present study only 10 insecticides ($>$ LOQ) were identified. These results confirm that producers usually limit the number of pesticides used on vegetables, the dosage and number of sprays before harvest (Illyassou et al. 2017) to save money. Despite the sporadic use of 4 herbicides and 4 fungicides in the study area, no residues of these chemicals were detected in the harvested products and no residues of the other insecticides previously reported were detected.

Based on their toxicological properties, organophosphorus insecticides are known to disturb the nervous system due to an excessive accumulation of acetylcholine in synapse (Fulton et al. 2001). They act by an irreversible inhibition of the acetyl-cholinesterase enzyme in the synapse (Illyassou et al. 2018). Pyrethroids may also adversely affect the central nervous system, disrupting the function of axons by keeping open the sodium channels which causes a depolarization in the axon. Neonicotinoids act also on the central nervous system by blocking postsynaptic nicotinic acetylcholine receptors. Avermectin chemicals affect the ability of neurotransmitters such as glutamate and γ -amino-butyric acid (GABA) to stimulate an influx of chloride ions into nerve cells, resulting in loss of cell function (Roberts et al. 1998). Therefore, the potential hazard from human exposure to these chemicals is important.

Table 6. Adults and children predicted short-term intakes (PSTI in mg/kg bw/day) for egetables with residues; NA: Not Available

Vegetable	Sample	Active ingredient	Concentration (mg/kg)	ARfD (mg/kg bw)	Adults		Children	
					PSTI	%ARfD	PSTI	%ARfD
Chili pepper	Sample 1	λ-Cyhalothrin	0.230	0.005	9.61E-5	1.9	0.00021	4.3
		Chlorpyrifos-ethyl	0.017	0.005	7.10E-6	0.1	1.58E-5	0.3
	Sample 2	λ-Cyhalothrin	0.051	0.005	2.13E-5	0.4	0.00004	1.0
		Chlorpyrifos-ethyl Emamectin	0.780 0.013	0.005 0.010	0.00032 5.43E-6	6.5 0.1	0.00072 1.21E-5	14.6 0.1
Sample 3	λ-Cyhalothrin	0.093	0.005	3.88E-5	0.8	0.00008	1.7	
	Emamectin	0.011	0.010	4.59E-6	0.0	1.02E-5	0.1	
Sample 4	λ-Cyhalothrin	0.110	0.005	4.59E-5	0.9	0.00010	2.1	
	Chlorpyrifos-ethyl	4.600	0.005	0.00192	38.4	0.00429	85.9	
Eggplant	Sample 1	Chlorpyrifos-ethyl	0.400	0.005	0.00528	105.8	0.01181	236.3
Head cabbage	Sample 1	λ-Cyhalothrin	0.073	0.005	0.00194	38.8	0.00433	86.7
		Emamectin	0.039	0.010	0.00103	10.4	0.00231	23.2
	Sample 2	λ-Cyhalothrin	0.064	0.005	0.00170	34.1	0.00380	76.1
		Fenitrothion Imidacloprid	0.022 0.014	0.013 0.080	0.00058 0.00037	4.5 2.9	0.00130 0.00083	10.1 1.0
	Sample 3	λ-Cyhalothrin	0.130	0.005	0.00345	69.2	0.00772	154.5
		Fenitrothion	0.070	0.013	0.00186	14.3	0.00415	32.0
	Sample 4	λ-Cyhalothrin	0.260	0.005	0.00691	138.3	0.01544	309.0
	Sample 5	λ-Cyhalothrin	0.056	0.005	0.00148	29.8	0.00332	66.5
Chlorpyrifos-ethyl Fenitrothion		1.000 0.014	0.005 0.013	0.02660 0.00037	532.1 0.5	0.05941 0.00083	1188.3 6.4	
Lettuce	Sample 1	λ-Cyhalothrin	0.130	0.005	0.00215	43.1	0.00480	96.2
	Sample 2	λ-Cyhalothrin	0.067	0.005	0.00110	22.2	0.00247	49.6
	Sample 3	λ-Cyhalothrin	0.015	0.005	0.00024	5.0	0.00055	11.1
	Sample 4	λ-Cyhalothrin	0.015	0.005	0.00024	5.0	0.00055	11.1
	Sample 5	λ-Cyhalothrin Acetamiprid	0.019 0.100	0.005 0.100	0.00031 0.00165	6.3 1.7	0.00070 0.00369	14.1 3.7
Moringa	Sample 1	λ-cyhalothrin	0.049	0.005	0.00072	14.5	0.00162	32.4
		Acetamiprid	0.059	0.100	0.00087	0.9	0.00195	2.0
	Sample 2	Allethrin λ-Cyhalothrin	0.012 0.180	NA 0.005	- 0.00266	- 53.3	- 0.00595	- 119.0

	Sample 3	Cypermethrin	1.800	0.400	0.02664	66.6	0.0595	148.8
		Acetamiprid	0.210	0.100	0.00310	3.1	0.00694	6.9
	Sample 4	Acetamiprid	0.010	0.100	0.00014	0.1	0.00033	0.3
		Cypermethrin	1.400	0.400	0.02072	51.8	0.04628	115.7
Pepper	Sample 1	λ -Cyhalothrin	0.032	0.005	2.87E-5	0.6	0.00006	1.3
		Acetamiprid	0.014	0.100	1.25E-5	0.0	2.80E-5	0.0
		Imidacloprid	0.051	0.080	4.58E-5	0.1	0.00010	0.1
	Sample 3	λ -Cyhalothrin	0.300	0.005	0.00026	5.4	0.00060	12.0
		Cypermethrin	0.019	0.400	1.70E-5	0.0	3.81E-5	0.1
		Dichlorvos	0.410	0.002	0.00036	18.4	0.00082	41.1
		Dimethoate	0.011	0.010	9.88E-6	0.1	2.20E-5	0.2
		Acetamiprid	0.200	0.100	0.00017	0.2	0.00040	0.4
		Imidacloprid	0.098	0.080	8.80E-5	0.1	0.00019	0.2
	Sample 4	λ -Cyhalothrin	0.020	0.005	1.79E-5	0.4	0.00004	0.8
		Imidacloprid	0.031	0.080	2.78E-5	0.0	6.22E-5	0.1
	Sample 5	λ -Cyhalothrin	0.085	0.005	7.63E-5	1.5	0.00017	3.4
		Acetamiprid	0.017	0.100	1.52E-5	0.0	3.41E-5	0.0
	Sample 6	Imidacloprid	0.150	0.080	0.00013	0.2	0.00030	0.4
λ -Cyhalothrin		0.046	0.005	4.13E-5	0.8	0.00009	1.8	
Acetamiprid		0.022	0.100	1.97E-5	0.0	4.41E-5	0.0	
Imidacloprid		0.240	0.080	0.00021	0.3	0.00048	0.6	
Sorrel leaves	Sample 1	Cypermethrin	0.039	0.400	3.50E-5	0.1	0.00007	0.2
		λ -Cyhalothrin	0.140	0.005	0.00100	20.2	0.00225	45.1
		Acetamiprid	0.120	0.100	0.00086	0.9	0.00193	1.9
	Sample 2	λ -Cyhalothrin	0.095	0.005	0.00068	13.7	0.00152	30.6
		Cypermethrin	0.024	0.040	0.00017	0.4	0.00038	1.0
	Sample 3	λ -Cyhalothrin	0.260	0.005	0.00187	37.5	0.00418	83.7
		Acetamiprid	0.460	0.100	0.00331	3.3	0.00740	7.4
	Sample 4	Dimethoate	0.013	0.010	9.37E-5	0.9	0.00020	2.1
		λ -Cyhalothrin	0.260	0.005	0.00187	37.5	0.00418	83.7
	Tomato	Sample 1	Acetamiprid	0.360	0.100	0.00259	2.6	0.00579
λ -Cyhalothrin			0.020	0.005	0.00064	13.0	0.00145	29.0
	Sample 1	Dichlorvos	0.290	0.002	0.00941	471.0	0.02103	1051.9
		Acetamiprid	0.038	0.100	0.00123	1.2	0.00275	2.8

About 62% of the samples contain residues of λ -cyhalothrin, followed by residues of acetamiprid and chlorpyrifos-ethyl. The high detection in this study of these residues could in part be due to the availability on the market in Niamey of pesticide formulations containing these active ingredients and their high persistence (Adamou et al. 2011; Illyassou et al. 2015). In fact, by comparing the active ingredients detected each other's, λ -cyhalothrin, dichlorvos and chlorpyrifos-ethyl have the lowest ARfD values. More than a half of the positive samples (75.9%) had multiple residues, with pepper being the vegetable with the highest number of residues in a single sample (up to 6). The presence of multiple residues in many vegetable samples could be explained by both the high frequency of treatments and the use of different formulations (Jallow et al. 2017). It could be also linked to non-compliance of the Pre-Harvest Intervals (PHI) highlighted in the study area (Illyassou et al. 2015). An average of three pesticide residues per vegetable sample was detected in gardening areas in Burkina Faso (Lehmann et al. 2017). Several studies have detected multiple residues in different foodstuffs including vegetables from many countries (Blankson et al. 2016; Dalvie et al. 2009; Jardim et al. 2012; Jallow et al. 2017; Keikotlhaile et al. 2010; Ngom et al. 2013; Son et al. 2017).

The highest concentrations of residues were found more in leafy vegetables than in fruiting vegetables. Nevertheless, the highest concentration of residue of all analyzed samples was detected for chlorpyrifos-ethyl in Chili pepper (4.6 mg/kg), which could be related to its lipophilic property. Cuticle analysis of some fruits revealed that the lipophilic composition of cuticle can help some lipophilic active ingredients to enter into the plant (Parsons et al. 2012; Trapp 2004).

A study conducted by Osman et al. (2010) in Saudi Arabia showed that the highest residue concentrations were found in cabbage (chlorpyrifos-ethyl, 6.207 mg/kg), tomato (tolclofos-methyl, 7.312 mg/kg), eggplant (carbaryl, 1.917 mg/kg), and pepper (carbaryl, 2.228 mg/kg). Nevertheless, the concentrations found were higher than those found in our study. In a previous study carried out in Burkina Faso, having similar agroclimatic conditions like Niger, authors reported the contamination of vegetables with pesticide residues (Lehmann *et al.* 2017). But, the concentration values were lower than those found in the present study.

Despite the high frequency of observed application (Illyassou et al. 2017 and 2018), 37% of samples collected in the dry hot season were free of residues. These findings could be linked to the degradation of active ingredients such as pyrethroids (the most used) by heat and sunlight (Adamou et al. 2011). All residues detected in lettuce samples were below the MRLs. However, 12 vegetable samples (26.7%) including 1 sample of tomato, 2 samples of pepper, 3 samples of sorrel leaves, 5 samples of head cabbage and 2 samples of Chili pepper had residues above the maximum residue limits for pyrethroids. The most exceedances of MRLs appeared for chlorpyrifos-ethyl and dichlorvos residues, respectively, in Chili pepper and head cabbage (46,00% and 2,00% of MRLs, respectively). The violation of MRLs in some vegetable samples could be a consequence of bad agrochemical practices and the non-respect of Good Phytosanitary Practices. In fact, the analysis of agrochemical practices in the Niger River valley has highlighted lack training of farmers for pesticide use. Farmers are not aware about the potential toxicity of pesticide formulations and/or active ingredient (Illyassou et al. 2017, 2018). These

findings corroborate the results of a study performed in Monze district (Zambia) where similar bad practices were highlighted (Mwanja et al. 2017). The results of this study are also in agreement with findings of a research performed in Ethiopia that showed lack knowledge among small farmers as one of the contributing factors of pesticide residues in vegetables (Mekonnen et al. 2002). In the study conducted by Jallow et al. (2017) on pesticide residues in Kuwait, 21% of the detected residues were found to exceed MRLs. A study conducted in Accra (Ghana) on pesticide residues in vegetables showed that okra and eggplant recorded the highest concentrations of allethrin (7.89 mg/kg) and permethrin (5.03 mg/kg). However, the detected concentrations were found to be higher than those detected in our study (Blankson et al. 2016).

PSTI expressed as a percentage of ARfD were in the range of 0.00-532.1% and 0.00-1188.3% for adults and children, respectively. Dichlorvos exposure in tomato consumption exhibited, respectively, acute risks for adults and children of 471 and 1,051%. Acute risk with λ -cyhalothrin and cypermethrin exposure was associated in the case of moringa consumption for children, but not confirmed for adults. An acute risk was identified for both adults and children in the case of eggplant consumption with chlorpyrifos-ethyl (105% and 236% ARfD, respectively). For λ -cyhalothrin, head cabbage consumption confirmed an acute risk exposure for children, but not confirmed for adults. However, the risk was observed both for adults and children in other samples of the same commodity (head cabbage) with residues of λ -cyhalothrin and chlorpyrifos-ethyl. In general, despite the high frequency of detected residues, consumers in Niger River valley do not face a significant acute

risk, except for dichlorvos, λ -cyhalothrin, cypermethrin and chlorpyrifos-ethyl.

These active ingredients present physicochemical and toxicological properties which induce an acute risk for general population. In fact, dichlorvos is expected to have a very high volatility in air (2.1 Pa, 25°C), but even if it is known to be a rather volatile compound, its octanol/water partitioning coefficient is high and its Log K_{ow} is positive ($K_{ow} = 7.94 \times 10^1$; Log $K_{ow} = 1.9$). This reflects its lipophilic property explaining its persistence on treated crops. Chlorpyrifos-ethyl is also expected to be a volatile compound (2.7 10^{-3} Pa, 25°C) (TOXNET-database 2018). But after treatment, it penetrates more deeply into the plant and becomes less available for volatilization thanks to its very high lipophilicity ($K_{ow} = 5.01 \times 10^4$; Log $K_{ow} = 4.7$) explaining also its persistency (Solomon et al. 2014). The dissipation rate (RL_{50}) on and in plant matrix range from 0.95-127.0 days (PPDB-database 2018).

λ -cyhalothrin and cypermethrin are pyrethroid active ingredients, having a low aqueous solubility (0.004 mg/kg, 20°C) are relatively volatiles (1. 10^{-6} Pa and 2.3 10^{-7} Pa, 20°C, respectively). Like organophosphorous pesticides, the two pyrethroids have both a high K_{ow} (3.16 10^5 and 3.55 10^5 , respectively), as they can also strongly penetrate into the treated crops. They are moderately persistent under field conditions on and in plant matrix (RL_{50} range 0.5-15.3 days and RL_{50} range 1.2-10.3 days, respectively) (PPDB-database 2018). Based on its chemical properties, all the active ingredients are not suggested to leach to groundwater except for chlorpyrifos-ethyl which has a high leaching potential index (GUS = 3.63).

Therefore, the presence of these residues could be explained not only by the high frequency of sprays, but also by their lipophilic properties in plant matrix. This

could be explained in part by the persistence of some residues in soils as well. By leaching or runoff, these active ingredients can reach surface or ground waters during a raining phenomenon very common in the Niger River valley. Contamination could also occur during crop irrigation with already contaminated waters. It should be noted that in Niger, vegetable crops are mostly grown in watershed with a steep slope (source of surface waters contamination). Finally, the presence of residues could be linked to non-compliance of the PHI observed at the field level (Illyassou et al. 2017).

In conclusion, the results of dietary exposure of plant protection product residues in vegetables in the Niger River valley show that from 45 vegetable samples, 26.7% exceeded the MRLs established by the European Union. More than a half of the positive samples (75.9%) had multiple residues. PSTI values appear to be relatively low compared to the corresponding ARfD values in all vegetable samples, except in 8 cases where PSTI values exceed several times ARfD. Therefore, to reduce the risk and safeguard the human health of the consumers in the Niger valley, producers need to replace persistent active ingredients and those with

low ARfD. Trainings should be initiated by agriculture agents to enhance farmer's knowledge on pesticide use and handling. More precautions must be taken to reduce dietary exposure especially for children (diversification of the diet). Processes of residue reduction (washing, boiling or cooking) must be taken by consumers to reduce significantly the risk level associated with active ingredient ingestion) (Bonnehère et al. 2012a, 2012b). Household processes like boiling can reduce some residues of pesticide from 20% to 100% in vegetables (Kumari 2008). There is also a need to more investigations on pesticide residues in all foodstuffs including cereals, meat, fish, milk products and water where other ingredients are often mentioned in many studies (Akoto et al. 2013; Darko et al. 2008; Ibigbami et al. 2015; Idowu et al. 2013; Topsoba et al. 2006). Regular close surveillance on potential introduction of herbicides and fungicides in the local agricultural practices and on house use insecticides is advisable. A chronic risk assessment in Niger in order to take overview of contamination levels and investigations on surface waters and sediments are also needed to get a large view on environmental impacts.

RESUME

Illyassou K.M., Adamou R. et Schiffers B. 2018. Première enquête alimentaire dans la vallée du fleuve du Niger et évaluation du risque d'exposition aiguë des consommateurs aux résidus des pesticides dans les légumes. *Tunisian Journal of Plant protection* 13 (2): 243-262.

Pour lutter contre les bioagresseurs des cultures, les agriculteurs de la vallée du fleuve du Niger utilisent une large gamme de produits phytopharmaceutiques. Particulièrement toxiques, les substances actives contenues dans ces produits peuvent induire des effets nocifs importants tant sur la santé humaine que sur l'environnement. Cette étude a porté sur l'exposition alimentaire aux résidus des produits phytopharmaceutiques dans la vallée du fleuve du Niger. L'enquête de consommation a été conduite en utilisant la méthode du rappel de 24 heures. Les tailles des portions sont estimées à partir d'une collection d'images préalablement préparées selon le régime alimentaire local habituel. Au total, 45 échantillons de huit types de légumes, les plus consommés dans la zone d'étude (Niamey) pendant la saison sèche froide et la saison sèche chaude ont été collectés. Les échantillons ont été extraits par la méthode QuEChERS multi-résidus puis analysés moyennant la chromatographie en phase gazeuse (GC-MS/MS) et la

chromatographie en phase liquide couplées à la spectrométrie de masse (LC-MS/MS); cette méthode concerne un jeu de plus de 500 substances actives. Les résultats montrent que des résidus d'insecticides ont été détectés dans 64,4% des échantillons parmi lesquels 26,7% des échantillons contiennent des résidus supérieurs aux limites maximales de résidus (LMR) et 2,3% des échantillons contiennent des résidus égaux aux LMR, 35,5% des échantillons ont des teneurs en résidus inférieures aux LMR. Les légumes les plus contaminés sont le piment, la tomate, le moringa, le chou pommé, les feuilles d'oseille et le poivron avec 4,6 mg/kg de chlorpyrifos-éthyl, 0,29 mg/kg de dichlorvos, 1,8 mg/kg de cyperméthrine, 1 mg/kg de chlorpyrifos-éthyl, 0,46 mg/kg d'acétamipride et 0,41 mg/kg de dichlorvos, respectivement. Pour évaluer l'apport journalier et caractériser le niveau de risque pour les adultes et les enfants, la feuille de calcul du modèle PRIMO de l'EFSA a été utilisée. Les résultats basés sur les consommations au percentile 97,5 montrent que les plus fortes valeurs d'exposition à court terme (PSTI) sont obtenues dans le cas de la consommation du chou-pommé pour les adultes (532% de la valeur de référence) et la consommation des tomates pour les enfants (1052% de la valeur de référence). Quel que soit le type de produit agricole, les enfants semblent être plus exposés que les adultes pour tous les résidus détectés.

Mots clés: évaluation des risques, exposition aiguë, résidus de pesticides, vallée du fleuve du Niger

ملخص

ياسو، كريمون ماسالانثسي ورباني ادامو وبرونو تشيفارس، 2018. أول مسح غذائي في وادي نهر نيجر وتقييم تعرض الحاد لمستهلكين مخلفات مبيدات فلاحية في خضروات.

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لمكافحة آفات وأمراض الزراعات، يستخدم المزارعون في وادي نهر النيجر مجموعة كبيرة من المبيدات الفلاحية. هذه المواد الفعالة، السامة بشكل خاص، يمكن أن تحدث تأثيرات سلبية خطيرة على صحة الإنسان وسلامة البيئة. أجريت هذه الدراسة حول تعرض الغذاء لبقايا مبيدات الآفات في وادي نهر النيجر. تم إجراء مسح للاستهلاك باستخدام طريقة الاستدعاء على مدار 24 ساعة. تم تقدير أحجام الأجزاء من مجموعة من الصور التي تم إعدادها مسبقاً وفقاً للنظام الغذائي المحلي المعتاد. في المجموع، تم جمع 45 عينة من ثمانية أنواع من الخضروات، الأكثر استهلاكاً في منطقة الدراسة (نيامي) خلال موسم الجفاف البارد وموسم الجفاف الحار. وقد تم تحليل العينات بطريقة متعددة البقايا (كويشترز) وباستخدام الكروماتوغرافيا الغازية مقترنة بمطياف الكتلة (GC-MS/MS) والكروماتوغرافيا السائلة مقترنة بمطياف الكتلة (LC-MS/MS). وقد خصت هذه التحاليل 500 عينة. أظهرت النتائج أنه تم الكشف عن بقايا المبيدات الحشرية في 64.4% من العينات، منها 26.7% تحتوي على بقايا أعلى من الحد الأقصى للمادة المتبقية (MRL) و 2.3% ذات مستويات بقايا تتساوى مع الحد الأقصى للمادة المتبقية و 35.5% لديها مستويات متبقية تحت الحد الأقصى للمادة المتبقية. كانت الخضروات الأكثر تلوثاً هي الفلفل الحار والطماطم والمورينغا والملفوف وأوراق الحميض والفلفل الحلو، على التوالي 4.6 ملغ/كغ كلوربيريفوس-إيثيل و 0.29 ملغ/كغ ديكلوروفوس و 1.8 ملغ/كغ سبيرمثرين و 1 ملغ/كغ كلوربيريفوس-إيثيل و 0.46 ملغ/كغ أسيتاميريد و 0.41 ملغ/كغ من ديكلوروفوس. ولتقييم الإسهام اليومي وتحديد مستوى المخاطر للبالغين والأطفال، تم استخدام جدول بيانات نموذج PRIMO لـ EFSA. أظهرت النتائج على أساس استهلاك نموي يساوي 97.5، أنه يتم الحصول على أقوى قيم التعرض على المدى القصير (PSTI) في حالة استهلاك الملفوف للكبار (532% من القيمة المرجعية) واستهلاك الطماطم للأطفال (1052% من القيمة المرجعية). يبدو أن الأطفال أكثر تعرضاً من الكبار لجميع البقايا التي تم الكشف عنها.

كلمات مفتاحية: بقايا مبيدات فلاحية، تقييم المخاطر، تعرض الحاد، مستهلكون، وادي نهر النيجر

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