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RESEARCH ARTICLE

LEVELS OF ORGANOCHLORINE, ORGANOPHOSPHORUS AND PYRETHROID RESIDUES IN BENINESE VEGETABLE FARMS SOIL

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ABSTRACT

Pesticides are number of chemicals used in intensive vegetable farming to control pathogen, pest and weed. Further, pesticides play a significant role by keeping many dreadful diseases. However, their use with high frequency may be an environmental problems. This study disingned insecticide residues (Organochlorine, Organophosphorus and Pyrethri-noid) in vegetable farms soil of Cotonou and Seme-kpodji area (Southen of Benin). Twenty soil samples from four vegetable farms were collected. Multiclass residues extraction was performed using soxhlet with solvent system hexane : acetone 3:2 (v/v). Residue analysis was performed using a GC-MS in SIS mode. MedCalc Statistical Software version 15.0. was used for the statistical analysis. The results revealed that most of the samples are contaminated by Organo-chlorine Organophosphorus and Pyrethroid residues. Among Organochlorine residues analyzed, 60 % of dieldrin, 35% of DDT, 65% of DDE, 75% of endosulfan II and 70% of endosufan II were detected. For Pyrethroid residues 25% of cypermethrin, 40% of cyfluthrin, 50% of fenpropathin and 95% of lambda cyhalothin were found in the soil samples. For Organophosphorus analyzed, 55% of profenofos 55% of chlorpyrifos and 30% of parathion were quantified. Levels of pesticide residues found in vegetable farms soils may be consider like an environmental problem.

Key words: Chemicals, Soil, Insecticide, Vegetable farming, Southen of Benin.

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INTRODUCTION

Soil is a compartment of the environment as well as water and air (Zhang *et al.,* 2017). It main's function is agricultural and forestry production and ensure the development of natural vegetation, a support for biodiversity (Ganeteg *et al.,* 2017). In Republic of Benin and particular in Southen of Benin, severals hectare of soils are cultivated in vegetable farming and constituted the crop support (Akomagni, 2006). Benin climate is favourable of pest proliferation and so it's necessary to use pesticides by vegetable farmers to control pests and diseases during farming (Adjrah *et al.,* 2013; Arun et Ghimire, 2017; Mengistie, 2017). Pesticides are known to be the most important tool for the production of adequate food supply for an increasing world population and for the control of vector-borne diseases (Riwthong *et al.,* 2017). There are several group of pesticides such as Herbicide, Fungicide and Insecticides (Carvalho, 2006; Regan *et al.,* 2017). In Southen of Benin, the most insecticide used in vegetable farming and found in vegetable belong Organophosphorus and Pyretroid class (Agnandji *et al.,* 2018). Although active ingredient of pesticides have beneficial effect for the crops, their use with regular frequency pose health and environment problems. Many studies about pesticides used in the world particular in Africa reported pesticide residues in agricultural soils (Sichilongo et Banda, 2013; Jemutai-Kimosop *et al.,* 2014;

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Han *et al.,* 2017). In Southen of Benin, despite pesticides using in vegetable farming since 38 years ago, few research was about levels of pesticide residues in agricultural soils. Last study has been done on Houéyiho site, the first one vegetable farming site in Cotonou (intra-urban area). This study shown Organochlorine pesticide residues such as endosulfan, DDT, heptachlor, aldrin and lindane in the soils (Assogba Kolman *et al.,* 2007). Nowadays, during last decade, number of vegetable farms site and farmers are increased in intra-urban and sub-urban area (Cotonou, Seme-kpodji, Grand-Popo, Porto-Novo). Paradoxically, none research was done to evaluate the presence of pesticide residues in those agricultural soils. It's in this way we have done this study to have assessment of pesticide residues distribution in soils of four vegetable farm site localized in Cotonou (intra-urban) and Seme-kpodji (sub-urban) area.

MATERİALS AND METHODS

Study Areas: Soils sample collection was done during Octorber 2016, the small rainy season in Benin. The samples were collected on two vegetable farms sites located in Cotonou, intra-urban area and two vegetable farms site located in Seme-kpodji, suburban area.

Sampling: A total of 20 soils samples at 15 cm depth were collected from the 4 vegetable farms site. A composite sample of 750 g was taken in aluminium foil and then put in a sterile polyethylene bag which was placed in an iced chest box and transported to laboratory. The samples were stored in refrigerator à 4°C until analysis.

Glassware and Chemicals reagents: All glassware used for extraction and cleaning was rigorously washed with water and detergent and rinsed twice with distilled water. They were again rinsed with acetone and finally dried in an oven. Pesticide standards, with certified purity at least 97% and pesticide grade solvents (acetone, acetonitrile, and hexane) were purchased from Sigma–Aldrich (Saint Quentin Fallavier, France), and VWR (International bvba/spr/ Geldenaaksebaan, Leuven Belgium).

Analysis of pesticide residues

Extraction procedure: Being wet, the soil samples were dried at 40 °C in an oven for 24 hours. Pesticide residues extraction was performed by Soxhlet. This method involves introducing 15g ± 0.1 of soil, finely ground into a Whatman® extraction cartridge (22mm x 80mm, Sigma-Aldrich, France). The whole is introduced into the soxhlet and is extracted with 100 ml of a hexane / acetone mixture (3: 2, v / v) (solvent system using to extract multiclass pesticide residues from the soils samples (El-Saeid *et al.,* 2010). Final extract is evaporated to dryness by a gentle nitrogen stream to obtain 1 ml.

GC–MS analysis: Gas chromatograph (Varian Saturn GC 2200, Agilent Technologies, Les Ulis, France) with a mass spectrometry (Varian CP-3800) detector equipped with autosampler (Varian CP 4800) injector was employed. A fused silica ZB-5MS capillary column (30 m x 0.25 mm x 0.25 µm), was used with the following oven temperature program : initial temperature 70°C, increased at 50°C/min to 150°C, ramp at 5°C/min to 180°C and finally increased at 1°C/min to 183°C and held for 6 min. Helium (99.9999%) was used as carrier gas (1.0 mL/MIN). A volume of 3.0 µL was injected in splitless mode. Mass spectrometer in electron impact mode (70 eV), scanning from m/z 50 to 650 was used in SIS mode. MS wokstation version 6.9.1 enabled data aquisition in single ion monitoring mode.

Calibration of Selected Ion Storage (SIS): SIS mode was used to selecte specific ions in the trap. The ejection of the unwanted ions is performed by applying a multifrequency waveform, which inclides those frequencies required to eject the unwanted ions and misses the fraquencies corresponding to the stored ions. Specific ions and retention time of pesticides standard we used to build SIS method is given in the Table 1 below. The residues of pesticide in the soils samples was identified based on comparison of the measured relative retention times and specific ions of standards. The residue levels of Organochlorine, Pyrethrinoid and Organophosphorus pesticides were quantitatively determined by the standard method using peak area. Measurement was carried out within the linear range of the detector. The peak areas whose retention times coincide with the standards were extrapolated on their corresponding calibration curves to obtain the concentration. The quality was assured through the analysis of solvent blanks, procedure blanks. The method was optimized and validated using spiked (together) with the internal standard to evaluate the recovery of compounds. The recoveries of the standards ranged between 92.18-98.12% for all Organochlorine, Pyrethrinoid and Organophosphorus pesticide residues. The limit of detection was 0.015 µg/kg.

Table 1. Standard of pesticide residues with their retention time and specifics ions

Pesticide residues	Retention time(minute)	Specifics ions of pesticide residues
Organochlorine residues		
Bêta HCH	12.894	109.1 – 183 – 218.9
Aldrin	18.937	66.1 – 263.1 - 293
Endosulfan I	25.290	195.2–241.2–269.1-338.8
Dieldrin	28.120	79.1 – 279.0 – 337.0
DDE	28.300	246.5 – 318.1
Endosulfan II	32.111	195.2 – 267 – 338.9
DDD	33.772	235
Endosulfan S	37.487	239.2-272.1–386.9-423.9
DDT	38.985	235.2
Organophosphorus residues		
Methamidophos	6.602	64 – 94 - 141
Dimethoat	12.517	87 – 93 - 125
Pirimiphos-m	17.849	233.3 – 290.2 - 305
Chloptrifos	19.067	197.1 – 258.2 - 314
Parathion	19.739	97 - 109.1– 291.0
Profénofos	28.031	97.1-139.2–208.2-269.2- 337.2
Fenpropathrin	43.810	97.1 – 181.1 – 265.1
Pyrethroids residues		
Lambda cyhalothrin	45.541	141.2 – 181.2 – 208.1
Cyfluthrin	47.485	91.1–127.1-163.2-206.3
Cypermethrin	47.758	91.1–127.2–163.2–206.2-226.1
Fenvalerate	48.986	125.2-167.2-181.3-225.2-419.2
Deltamethrin	49.938	172.2 – 181 – 253.2

Statistical Analysis: Results obtained are expressed in mean ± Ecartype of the mean. MedCalc Statistical Software version 15.0. was used for the statistical analysis.

RESULTS AND DİSCUSİON

Intensive vegetable cultivation involving widespread use of different pesticides driven by more returns. After pesticides use, they contaminate the soils by their residues which may be quantified using various analytical instruments like gas chromatography coupled with a mass detector. Our results confirm those obtained by several authors.

Levels of Organochlorine pesticide residues in vegetable farms soils: Avarage of Organochlorine pesticide residues found in soils samples is 41.0±44.4 µg.kg^{-1}. β HCH and aldrin weren't detected while DDT and endosulfan I were occurred in 35% and 70% of the soil samples. Aldrin metabolite (dieldrin), endosulfan I metabolite (endosulfan II) and DDT metabolite (DDE) were found at 60, 70 and 65 % proportion in the soil samples, as shown in Table 2. Dieldrin detection at this frequency due to aldrin conversion (Wandiga, 1995). It's mean is 24.7±31.3 µg.kg^{-1} and ranged from 6.7-88.6 µg.kg^{-1}. Fosu-Mensah *et al.,* 2016 repported similar mean of dieldrin (0.02 ± 0.00 mg.kg^{-1}) and ranged from 0.005–0.05 mg.kg^{-1} with its occurring most frequently in cocoa farms soil. Contrary to our results, *Abongo et al., 2015;* found Aldrin at 18.317±0.276 µg.kg^{-1} and suggested that the presence of aldrin and other of Organochlorine residues in soils in the basin could be impacting negatively on the ecosystem health of the area. We found in this study, low concentration of DDT and it metabolite DDE in soil samples. The mean of DDT level is 0.9±1.2 µg.kg^{-1} with ranged from 2.3-2.8 µg.kg^{-1}. Highest level of DDT (mean 0.3813 mg.kg^{-1} ranged from 0.00721-2.9100 mg.kg^{-1}) was reported by Shi *et al.,* 2005 in China soils. Recently Fosu-Mensah *et al.,* 2016, DDT was found in cocoa farm (Ghana) at average level 0.03 ± 0.01 mg.kg^{-1} and ranged from 0.005–0.04 mg.kg^{-1}. Among Organochlorine residues quantified, alone endosulfan compound like as endosufan I and endosufan II were present in the soil samples with high levels. Endosufan I mean is 95.2±132.2 µg.kg^{-1} with ranged from 0.3-429.9 µg.kg^{-1} while Endosufan II average is 81.3±103.7 µg.kg^{-1} and ranged from 1.5-312.6 µg.kg^{-1}. Endosulfan sulfate which is a degradation product of endosulfan wasn't detected in the soils. In Vakhsh (Republic of Tajikistan) High concentrations (>5 ppm) of endosulfan II were also present in some of agricultural site soil samples (Barron *et al.,* 2017). Contrary to our results, endosulfan sulfate was present in Island (India) farm soils and ranged from 1.24-38.16 ug.kg^{-1}. Kumari *et al.,* 2007 reported in his study, low level of endosulfan I (0.002-0.039 µg.g^{-1}) in farm soils of Haryana (India). Low level of Endosulfan I (n.d.–5.1 µg.kg^{-1}) and endosulfan II (nd.–14.8 µg.kg^{-1}) were found in Jujube soil (China). This result does not reveal any recent application of OCP in the fields but it shown their resistance to degradation in soils and their persistance in the environment after their using.

Levels of Organophosphorus pesticide residues in vegetable farms soils: Mean of Total concentration of Organophosphorus residues found in soils samples was 36.1±64.1 µg.kg^{-1}. Three of Organophosphorus residues (Methamidophos, Dimethoate and Pirimiphos-methyl) weren't detected in the soils samples while parathion, profenofos and chlorpyrifos were detected at 30%, 55% and 55%, indicated in Table 3. Low level (0.9±1.5 µg.kg^{-1}) of parathion was found in the soil samples and ranged from 1.9-5 µg.kg^{-1}. Liu *et al.,* 2015 results is opposed to our finding. They didn't detect parathion in Persimmon and Jujube soils. In the same way, Fosu-Mensah *et al.,* 2016 quantified pirimiphos-methyl (which we didn't detect in our soil samples) at 0.02 ± 0.01 mg.kg^{-1} and it ranged from 0.01–0.04 mg.kg^{-1} in cocoa soil samples (Ghana). Chlopyrifos and profenofos was the two first Organophosphorus found at high concentration in our soil samples. Mean of chlorpyrifos is 148.2±191.6 µg.kg^{-1} and ranged from 68.1-607.5 µg.kg^{-1} and then profenofos average was 31.6±40 µg.kg^{-1} and range from 8.6-112.8 µg.kg^{-1}. Profenofos was found in a Philippines farm soil at level 0.10 ppm in last study carried out by Prado-Lu, 2014. Recently, Fosu-Mensah *et al.,* 2016 found in cocao soils of Ghana, Chlorpyrifos mean 0.03 ± 0.01 mg.kg^{-1} and ranged from 0.01–0.04 mg.kg$^{-1;}$ profenofos mean 0.03 ± 0.01 mg.kg^{-1} and ranged from 0.02-0.04 mg.kg^{-1}.

Table 2. Minimum, maximum concentration ranges of Organochlorine pesticide residues and their means in South of Benin vegetable farms soil(µg/kg)

Organochlorine residues	Min-Max	Mean ±Ecartyp	Frequency%
Bêta HCH	ND	--	--
Aldrin	ND	--	--
Dieldrin	6.7-88.6	24.7±31.3	60
DDT	2.3-2.8	0.9±1.2	35
DDE	3.2-10.6	2.9±2.7	65
DDD	ND	--	--
Endosulfan I	0.3-429.9	95.2±132.2	75
Endosulfan II	1.5-312.6	81.3±103.7	70
Endosulfan S	ND	--	--

ND = No determined

Table 3. Minimum, maximum concentration ranges of Organophosphorus residues and their mean in South of Benin vegetable farm (µg/kg)

Organophosphorus residues	Min-Max	Mean ±Ecartyp	Frequency%
Methamidophos	ND	--	--
Dimethoate	ND	--	--
Pirimiphos-methyl	ND	--	--
Chlopyrifos	68.1-607.5	148.2±191.6	55
Parathion	1.9-5	0.9±1.5	30
Profenofos	8.6-112.8	31.6±40	55

ND = No determined

Table 4. Minimum, maximum concentration ranges of Pyrethroid pesticide residues and their mean in South of Benin vegetable farm soil (µgkg^{-1})

Pyrethroid residues	Min-Max	Mean ±Ecartyp	Frequency%
Fenpropathrin	18.4-275.1	70±95.3	50
Lambda cyhalothrin	0.4-301.4	82.6±98.2	95
Cyfluthrin	1.5-62.5	44.4±34.1	40
Cyperméthrin	2.6-7.5	1.2±2.3	25
Fenvalérate	ND	--	--
Deltaméthrin	ND	--	--

ND = No determined

Levels of Pyrethroid pesticide residues in vegetable farms soils: Average of Pyrethroid residues found in samples soils is 32.7±40.2 µg.kg^{-1}. Among Pyrethroid studied, fenvalerate and deltamethrin are not detected. Lambda cyhalothrin is a most Pyrethroid found in a soils (95%). The other Pyrethroid (fenpropathrin, cyfluthrin and cypermethrin) are found at 50, 40 and 25% frequency, an indicated in Table 4. Our finding are contrary to those of Fosu-Mensah *et al.,* 2016, who found in cocoa farm soils, deltamethrin with a mean value of 0.04±0.01 mg/kg. In 2015, Liu *et al.,* detected in soils samples from Persimmon and Jujube (China), deltamethrin level which varied respectively from n.d.-42.8 µg/kg and n.d.-46.9 µg/kg. In the same study, fenvalerate level found in the soils samples ranged from n.d.–15.9 µg/kg (Persimmon) and n.d.–2.5 µg/kg (Jujube). Mean of lambda cyhalothrin in our study is 82.6±98.2 µg.kg^{-1} and ranged from 0.4-301.4 µg.kg^{-1}. The concentrations of lambda cyhalothrin in the soil samples may be attributed to the use of pesticides containing lambda-cyhalothrin as its active ingredient in the study area. Additionaly, lambda cyhalothrin is an molecule which has high affinity for soil as reported by Tarus Nyambati, Kituyi and Segor Chebii (2007). Mean of the level (0.03±0.00 $mg.kg^{-1}$) of lambda cyhalothrin found in cocao soils by Fosu-Mensah *et al.,* 2016 is lower than our finding. Low level of lambda cyhalothrin (7.3 $\mu g.kg^{-1}$ in Persimmon soil) and (16.0 $\mu g.kg^{-1}$ in Jujube soil) in China were observed by Liu *et al.,* 2016. Cypermethrin and cyfluthin means are respectively 1.2±2.3 $\mu g.kg^{-1}$and 44.4±34.1 $\mu g.kg^{-1}$. Cyfluthrin mean (0.04±0.00 $mg.kg^{-1}$) reported by Fosu-Mensah *et al.,* 2016 is lower our finding while cypermethrin mean (0.03±0.01) is 25 times higher than our finding.

Conclusion

At the end of our study, it appear that different king of pesticide residues are present in vegetable farm soil of South of Benin. Its pesticide residues belong Organochlorine, Oganophosphorus and Pyrethroid pesticide class. Organochlorine residues most found in soil samples were endosulfan I and it metabolite endosulfan II follow by dieldrin and and DDE, respectively metabolite of aldrin and DDT. Despite their presence in vegetable farms soil in high frequency, only endosulfan II and endosulfan I had the highest levels with means respectively 95.2±132.2 μgkg^{-1} 81.3±103.7 μgkg^{-1}. It's import to note that their presence in vegetable farm soils ins't linked with their resent used. Oganophosphorus and Pyrethroid residues most found in soil samples were lambdacyhalothrin, chlorpyrifos and profenofos. The means highest levels of this residues quantified in the soils were 82.6±98.2 μgkg^{-1} for lambdacyhalothrin, 31.6±40 μgkg^{-1} for profenofos and 148.2±191.6 μgkg^{-1}.

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