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Determination of four pesticide residues in market garden soils at three major vegetable production sites in Abidjan and surroundings, Côte d'Ivoire

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Abstract

The widespread use of pesticide leads to environmental contamination, and excessive use of pesticide in market gardening could leave potentially harmful residues in soil, air and water. This study aimed at determining cypermethrin, glyphosate, carbendazim and profenofos residue concentrations in market garden soils in order to assess their possible contamination. A total of thirty five soil samples were collected from three major market garden production sites. Analyses of soil samples were performed using high performance liquid chromatography (HPLC-UV). Physicochemical properties of these soils were also characterized. The results obtained showed that four pesticide residues were detected in 79.67% of soils samples. They also indicated that the concentration of profenofos was higher than other pesticide residue concentrations. A proportion of 80% of soils contained more than one pesticide residue and glyphosate residues represented 26.95% in soils, while 25.21% of soils contained cypermethrin residues.As for profenofos, it showed high levels at a percent of 24.34%. Glyphosate and cypermethrin displayed respectively average concentrations ranging from 0.07 mg/kg±0.006 to 0.015 mg/kg ±0.012 and 0.028 mg/kg±0.02 to 0.062 mg/kg±0.053.23.47% of soil samples were contaminated with carbendazim residues at concentrations ranging from 0.007mg/kg ±0.003 to 0.012 mg/kg±0.007. From these study outcomes, known toxicity of aformentimed molecules justifies removal of pesticide residues from market garden soils using biodegradation method.

Keywords: Pesticide residues, market garden, soil contamination, HPLC-UV

Introduction

Pesticides are used for crops' protection, and to get rid of weeds. Two decades ago, pesticides use increased significantly worldwide (Baishya *et al.*, 2015). As such, they contributed tremendously to improving agricultural productivity (Camard *et al.*, 2010). To illustrate, on global scale, vegetables crops played a crucial role in human nutrition. In addition, they improved family incomes, especially in developing countries (FAO, 2012). Nevertheless, vegetables production is hampered by pressure from pests, weeds, disease attacks which limit their productivity (Nasr *et al.*; 2014; Yarou *et al*., 2017). In order to avoid high crops losses and improve productivity, vegetable producers apply pesticides in high doses(Giroux *et al*., 2004; Harrison *et al*., 2013; Mohammed *et al*., 2018). Excessive pesticides use, raised many concerns related to demonstrated toxicity of chemicals used, and their negative effects on environment and human health (Biego *et al*., 2009). Despite beneficial advantages provided to agriculture, their negative impact on vegetable quality, health, and environment brought about many concerns (Abagale *et al.*, 2019). Soil organic matter plays a pivotal role in the retention of pesticides (Guigon *et al*., 2006). As a matter of facts, use of pesticides leads to soil pollution, because frequently found in soil and water streams, and uptaken by foodstuff, therefore creating health hazards (Ashraf *et al*., 2014; Buvaneswari *et al*., 2018). Given the adverse effects and potential risks related to pesticides that could negatively impact the environment and public health, it is necessary to constantly monitor residues content in vegetable and market garden soils.

Additionally, rapid urbanisation of Abidjan and surroundings in Côte d'Ivoire, led to a high demand of vegetables. This situation encouraged development of market gardening around cities with a side effect of using considerable amounts of chemical pesticides. From this perspective, many studies were carried out on pesticides residues in vegetables (Yao *et al.*, 2016; Diarra *et al*., 2018; Kpan *et al*., 2019) and fruits (Biego *et al.*, 2009), but studies on the determination of pesticides residues in market garden soils are limited. For reasons aforementioned, this study aimed at investigating levels of cypermethrin, glyphosate, carbendazim, and profenofos contamination in market garden soils.

Materials and Methods

Soil sampling

The study was carried out on three biggest vegetables production sites (Port-Bouët, Songon and Bingerville). These sites were selected and described according to early investigation Mambé-Ani *et al.*, (2019). Coordinates of study sites are recorded in Table 1.

Table 1: coordinates of study sites

Study site	situation	latitude	longitude
Port-Bouët	South-East of Abidjan	5ºNorth	4º West
Bingerville	West of Abidjan	5º19 North	4º 15 West
Songon	North of Abidjan	5º21 North	3º 54 West

A total of 35 soil samples were collected one month after last pesticide application. The sampling procedure involved collecting a composite sample from five sub-samples following the procedure described by Kouakou *et al*. (2019). Soils were sampled randomly to form a composite sample of 1kg. The resulting composite sample was thoroughly mixed. The sampling depth was 0 to 30 cm. Samples were collected in plastic bags, labelled and placed in an ice box containing ice packs. They were sent to laboratory for analysis. Each soil sampling location referenced using GPS.

Physico-chemical analysis

It was estimated that contents and toxicity of pesticide in soilwas influenced by many physicochemical parameters (Savadogo *et al*., 2008; Chaplain *et al*., 2011). Yet, characterization of these parameters for market garden soils had been rarely studied in Ivorian scientific works. Soil physicochemical properties measured were pH, moisture, water activity (aw), organic matter (OM) and carbon (C), nitrogen (N), calcium (Ca), chloride (Cl), magnesium (Mg), texture and C/N. pH was measured using a pH meter

(Mettlertoledo, France), moisture was determined using a drying oven (P Selecta, France) and balance (Adam Nimbus, South Afrca), total nitrogen was determined by the Kjeldahl (1883) method, texture was established by pipette Robinson method, organic carbon was determined according to Walkley and Black (1934) titration method, the organic matter was calculated by the following formula: OM% = Carbon x 1,724. C/N was deduced from carbon and total nitrogen values. Calcium, magnesium and chloride were determined by standard methods described by Petard (1993). All tests were carried out in triplicate.

Pesticides extraction and HPLC/UV analysis

All samples were treated and analysed at the central laboratory of Agrochemistry and Ecotoxicology of the National Laboratory in support of Agricultural development (LANADA). Experiments were conducted as follows: soil was air dried and sieved (2 mm) before extraction, then pesticides residues were chosen based on commonly used pesticides at selected market gardening sites. According to previous work by Mambé-Ani *et al*., (2019), pesticides selected for this study were glyphosate, cypermethrin, carbendazim and profenofos.

To extract glyphosate residues in soils, a weight of 25 g mixed with 25 ml of bi-distilled water was centrifuged for 20 min at 5000 rpm after 15 min shaking. Then, the supernatant was filtered on Whatman paper, and1 ml of the filtrate was collected in another centrifuge tube with 1ml of sodium tetraborate solution and 1 ml of fluorenylmethoxy-carbonyl. The latter mixture was shaken for 30 minand separated by centrifugation for 5min at 5000 rpm. The new supernatant was collected in vials for quantification. Cypermethrin, carbendazim and profenofos residues were extracted with the same procedure as above with some modifications. 25g of soil was extracted using 50 ml of dichloromethane by vortex mixing for 30 min at 400 rpm. After centrifugation at 5000 rpm for 5 min, the supernatant was evaporated to dryness using a rotary vacuum evaporator (Buchi, Switzerland). Dried extracts obtained were re-dissolved in 5 ml of methanol and placed in vials for analysis. The extracts were analysed using HPLC method. The HPLC system (Shimadzu, Japan) was equipped with a detector UV/VIS SPD-20A, a nucleosilC18 (Shim pack VP-ODS 250 L x 4.6 mm ID) column, a pump LC-20AT, a source of temperature CTO-20A (40ºC), a degasser DGU-20A5. The analysis was performed in isocratic mode with a flow rate of 0.5 ml/min. The retention times and other analytical parameters are listed in Table 2. The chromatograms are extracted at different wavelength as mentioned in table 2. The conditions used for the analysis are summarised in Table 2. The eluent used for the present investigation was a mixture of water and acetonitrile. The different injection volumes were 10 µL for glyphosate, cypermethrin, carbendazim and 20 µl for profenofos. Data collection was performed using a computer with LC solution software. Samples and standard peak areas were used to quantify pesticide residues. Pesticide residue concentrations were finally determined using the following formula:

$$C_p = \frac{S_c \times C_e \times V_2 \times V_f \times F}{S_e \times M_e \times V_1}$$

Where

Cp: concentration of the active pesticide molecule (mg/kg)
Sc: peak area of the sample
Se: peak area of the standard
Ce: concentration of the standard (mg/L)
V1: total volume of extract (L)
V2: volume of purified extract (L)
Vf: final volume (L)
Me: mass of the sample (Kg)
F: dilution factor

Table 2: Characteristics of chromatographic analysis

Active ingredients	Wavelength (nm)	Column type	Injection volumes (µl)	Retention times (min)	Solvents	
					Water (%)	Acetonitrile (%)
Glyphosate	205	Nucleosil C18	10	3.31	50	50
Cypermethrin	230	Nucleosil C18	10	4.94	10	90
Carbendazim	210	Nucleosil C18	10	6.73	30	70
profenofos	254	Spherisorb S50 D	20	6.24	10	90

Statistical analysis

Data analysis was performed using software R version.3.6.1. Significant differences and similarities between different parameters were figured out added up to correlation between pesticide residues and soil properties, using a one-way ANOVA and HSD tests. Finally, statistical significance tests were carried out at 5 % confidence level ($p < 0.05$) along with Excel 2013 to draw graphs.

Results and Discussion

Physicochemical soil parameters

The present investigation focused on the detection of pesticide residues in market garden soils in Abidjan, and its surroundings, Songon and Bingerville. To better understand soil state, physicochemical parameters were determined. These physicochemical soil parameters are displayed in Table 3. Soil pH is a parameter which influences the bio-availability and transport of pesticides in soils (Fosu-Mensah *et al*., 2016). The pH measured in soils ranged from 5.44±1.1 to 6.46±0.36, indicating that the soils were moderately acidic. This acidity could be explained by the repeated and excessive use of pesticides, organic amendments and the permanent practice of market gardening on these soils (Touré *et al*., 2010). These results are in agreement with studies carried out in Port-Bouët (Côte d'Ivoire) by Kouakou *et al*. 2019, and N'djili CECOMAF Kinshasa (RD Congo) (Muliele *et al*., 2017) on market garden soils.

Outcomes from investigation showed that, Bingerville had the lowest organic matter values (0.47±0.23%) while Songon had the highest (3.12±2.31%). Soil samples from the three study sites were generally low in organic matter. There were significant differences ($p < 0.05$) in means concerning organic matter in Bingerville and Songon, however there were no significant differences in organic matter between Bingerville and Port-Bouët. The low organic matter contents would be due to permanent fertilisation and intensive tillage (Ben Hassine *et al*., 2008). The organic matter values obtained in our study are lower than those found in market gardening soils by Temgoua *et al*. (2015) in Cameroon.

Table 3: Physicochemical analysis results of soil samples

	Study site		
Parameters	Bingerville	Port-Bouët	Songon
pH	$5.44±1.15^{b}$	$6.45±0.35^{a}$	$6.12±0.90^{ab}$
Moisture	$30.20±11.06^{a}$	$11.80±4.90^{b}$	$3.56±0.79^{c}$
Aw	$0.981±0.001^{b}$	$0.983±0.001^{a}$	$0.983±0.001^{a}$
%OM	$0.47±0.23^{b}$	$1.04±0.99^{b}$	$3.12±2.31^{a}$
C/N	$5.04±3.35^{b}$	$9.07±7.67^{b}$	$28.35±25.2^{a}$
Ca^{2+} (cmole.kg^{-1})	$0.49±0.22^{a}$	$0.45±0.18^{a}$	$0.57±0.24^{a}$
$Mg2^{+}$ (cmole.kg^{-1})	$1.03±0.39^{a}$	$1.16±0.53^{a}$	$1.09±0.44^{a}$
Chloride (cmole.kg^{-1})	$1.53±0.44^{a}$	$3.10±2.57^{a}$	$2.70±2.24^{a}$

On the same row, values with same letter (a,b, c) are not significantly different at the 5% threshold

The C/N values of all samples analysed were very high and ranged from 5.04±3.35 to 28.35±25.20. C/N levels in Songon was higher (28.35±25.20) than Bingerville and Port-Bouët, and ranged from 5.04± 3.35 to 9.07±7.67 respectively. The C/N ratios revealed that there was significant difference ($p < 0.05$) between soil samples of Bingerville and Songon as well as Port-Bouët and Songon; however, there was no significant difference ($p > 0.05$) in mean C/N of soils of Bingerville and Port-Bouët. The carbon/nitrogen (C/N) ratio is an indicator of the quality of soil organic matter (Genot *et al*., 2009). The high C/N value obtained in songon site was 28.35, reflecting a slow rate of mineralisation of the organic matter at this site.

Concentrations of calcium, magnesium and chloride ions in soil samples were generally low. Studies by Muhinda *et al*. (2009) indicated that calcium values between 2 and 4 cmole/kg are low; yet our results ranged from 0.45 to 0.57 cmole/kg, suggesting a nutrient deficit for the crops. The low concentrations obtained in our studies could be explained by the acidity and the low organic matter contents in the analised soils (Aikpokpodion *et al*., 2010). The results obtained were higher than those of Kouadio *et al*. (2018) and Voko *et al*. (2013) in Côte d'Ivoire but lower than those of Akotto *et al*. (2014); Akassimadou *et al*. (2014) and Ballot *et al*. (2016) in Côte d'Ivoire and Centrafrique. Differences between measured values might be due to the different soil and climatic conditions in which the trials were conducted.

Results of texture analysis of soil samples is presented in figure 1. Results showed that the soil of Port-Bouët had a high sand content (92.77±2.17%). Sand values measured in soils of the three study sites were statistically different. Indeed, there was a significant difference ($p < 0.05$) between proportions of sand in soils of Port-Bouët and Songon sites (50.21±17.05%), as well as for proportions of sand in Port-Bouët and Bingerville sites (41.43±18.08 %).Values measured of clay in Songon soils (26.85±9.97%) were statistically higher than those measured in Port-Bouët site (3.4± 0.31%). However, silt contents indicated that proportions of silt in the Songon and Bingerville soils are statistically identical ($p > 0.05$). In general, the particle size distribution revealed that textural class of soils was mainly sandy and sandy-clay loam. These results are similar to those conducted in Port-Bouët Abidjan (Côte d'Ivoire) by Kouakou *et al*. (2019) on market garden soils.

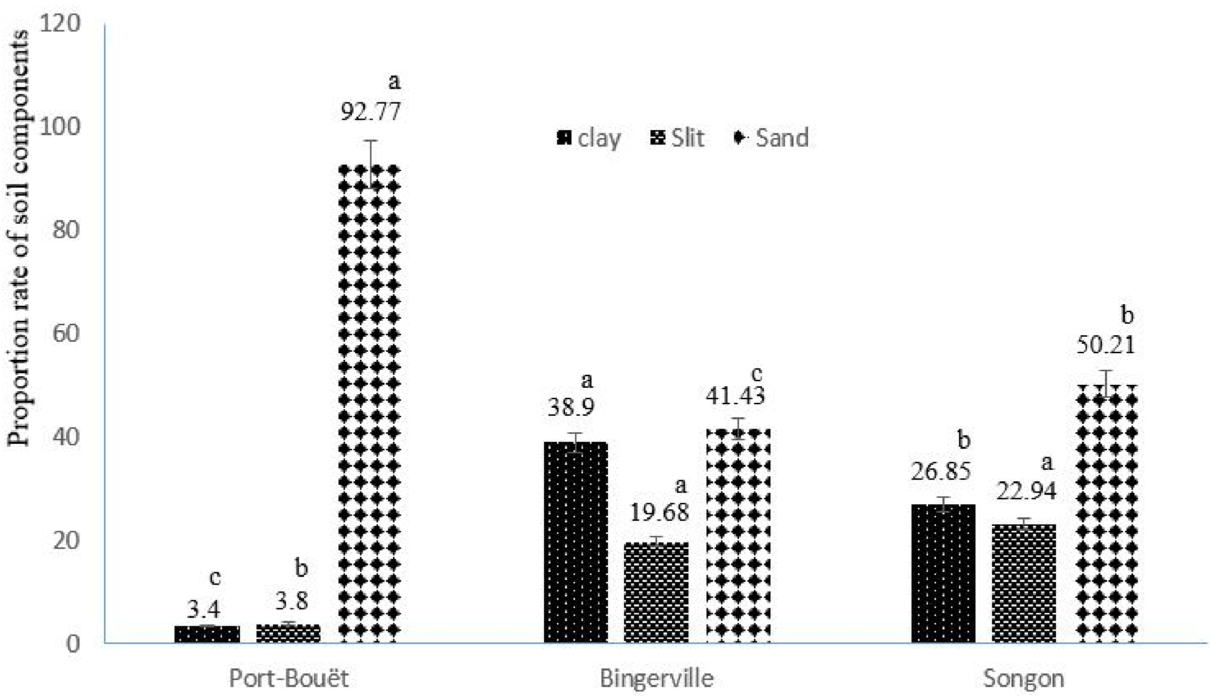

Figure 1: Soils granulometric proportions in Port-Bouët, Bingerville, and Songon
Color bars of the same letter are statistically identical at 5 % threshold (test HSD)

Pesticide residue concentrations

Table.4 presents the concentrations of pesticide residues measured in soil samples. A proportion of 79.67% of soil samples analysed showed presence of four (4) pesticide residues. On the contrary, percentage of contaminated soil reported in this study was lower than the 100% value reported by Kihampa *et al*. (2010) in Tanzania.Glyphosate was detected in 26.95% of the soil samples analysed at a mean concentration ranging from 0.015 ± 0.012 to 0.07 ± 0.006 mg/ kg. Although glyphosate was the most detected in the crop soils, the concentrations of glyphosate residues were the lowest. This can be explained by rapid biological or photochemical degradation of this molecule. Studies of Vreeken *et al*. (1998) and Pkan *et al*. (2018) corroborate our results. In fact, these authors showed that the low levels of glyphosate in soils were due to its degradation into aminomethylphosphonic acid (AMPA), its major metabolite. The mean concentrations of glyphosate reported in this study was as low as the mean value (6.9 µg) reported in soils in Galicia, North-West Spain (Veiga *et al*., 2001).

Table 4: Pesticide residue concentrations in studied soils (mg/kg)

	Study site		
Parameters	Bingerville	Port-Bouët	Songon
Cypermethrin	0.049±0.015[a]	0.062±0.053[a]	0.028±0.02[a]
Glyphosate	0.015±0.012[a]	0.007±0.006[a]	0.007±0.01[a]
Carbendazim	0.012±0.007[a]	0.008±0.01[a]	0.007±0.003[a]
Profénofos	0.498±0.27[a]	0.068±0.067[c]	0.256±0.163[b]

On the same row, values with same letter (a,b, c) are not significantly different at the 5% threshold

Due to its high toxicity, search for cypermethrin residues is of great importance (Garoiaz *et al*., 2012). Cypermethrin was detected in 25.21% of soil samples analysed at a mean concentration ranging from 0.028 ±0.02 to 0.062±0.053 mg/kg. Cypermethrin levels in soil samples indicated a low level of cypermethrin contamination. The mean concentrations of cypermethrin recorded in this study were lower than mean concentrations of 4,45 mg/kg reported by Onuwa *et al*. (2017) in market garden soils in Nigeria. In contrast, studies conducted by Mahugija *et al*. (2017) on tomato soil samples in Tanzania showed no cypermethrin residues.

Proportions of 23.47% and 24.34% of soils were contaminated with carbendazim and profenofos respectively at means ranging from 0.007±0.003 to 0.012±0.007 mg/kgand 0.068±0.067 mg/kg to 0.498±0.27 mg/kg (table 4 and Figure 2). There were no statistically significant sites difference ($p > 0.05$) in mean concentrations of the detected glyphosate, cypermethrin and carbendazim residues in soil samples. However, there was a significant difference ($p > 0.05$) in mean concentrations of profenofos residues in different sites.The soils of Bingerville had the highest residue levels of profenofos (0.498±0.27 mg/kg) followed by Songon (0.256±0.163 mg/kg) and Port-Bouët (0.068±0.067 mg/kg). The presence of pesticide residues in soil samples suggested previous and current use of chemical pesticides. Among the different pesticide residues detected in soil samples, profenofos was found with the highest concentration of 0.498±0.27, 0.068±0.067 and 0.256±0.163 in Bingerville, Port-Bouët and Songon respectively. These results can be attributed to unsafe practices such as excessive pesticide use, non-compliance with recommended rates of pesticide and repeated pesticide use. These poor agriculture practices led to exposures to pesticide residues in soils and vegetables, which were the main source of pesticide residues levels detected in soils. These findings were like those observed in studies conducted in market gardens in Abidjan (Côte d'Ivoire) (Tano *et al*., 2012) and in Kouka and Toussiana (Burkina Faso) (Son *et al*., 2018), where these authors highlighted poor agricultural practices. Profenofos was present in large quantities in soil samples because it is commonly associated with other pesticide molecules. The concentrations of profenofos obtained in Port-Bouët were lower than those reported (89.79 µg/kg) in the studies by Abagale *et al*. (2019), but were higher than those by Rafique *et al*. (2016).The high levels of profenofos could be explained by its excessive and continuous use and low degradation. The concentration of carbendazim obtained were lower than those found by Raheem *et al*. (2017) in Basra (Irak). The low levels of carbendazim obtained could be attributed to biodegradation, photodegradation and hydrolysis of this molecule. Concentrations of pesticide residues obtained in our study might be due to the fact that these pesticides were applied at a higher level than the recommended dose (Bhandari *et al*., 2020).

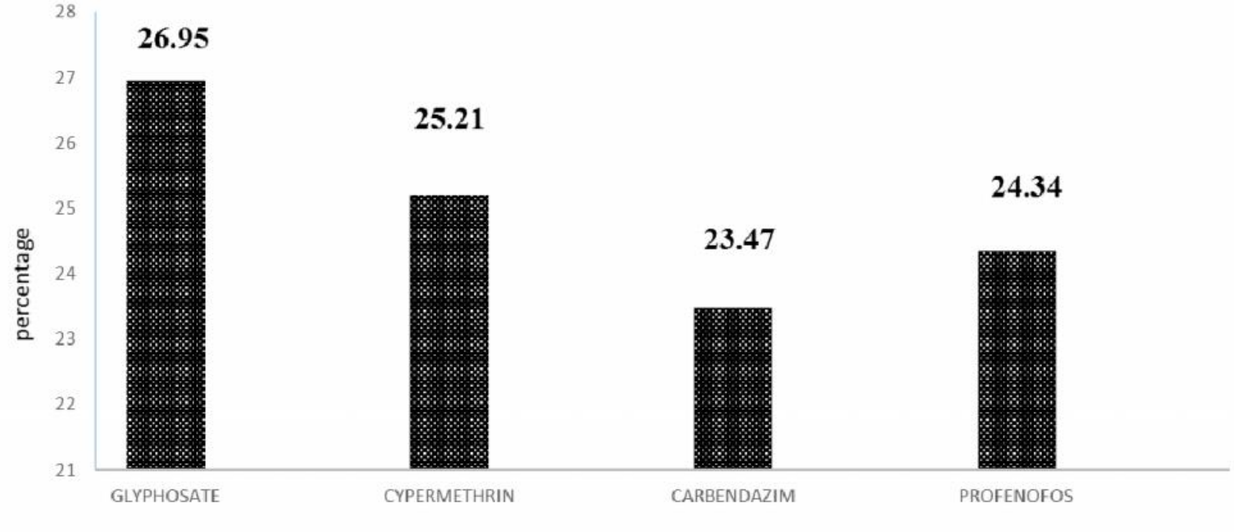

Figure 2: Positivity Percentage of soils for each active molecule

Relationship between soil physicochemical properties and pesticide residues detected

Table 5 gives an overview of the relationship between soil physicochemical properties and pesticide residues. The correlation test established a correlation between certain physicochemical parameters and pesticide residues in soil samples with a correlation coefficient r = 0.80 at 5 % confidence level ($p < 0.05$). Results of the correlation test indicate that, overall, parameters studied are weakly correlated, except for a few parameters such as organic carbon and percentage of organic matter, which are perfectly correlated (r=1). A positive correlation was observed between organic matter and pesticide residues such as cypermethrin and carbendazim, which indicated that the pesticide residue levels in soils are associated with organic matter content. This findings are similar to a study by Stoleru *et al*. (2015) which reported a significant correlations between organic matter and pesticides residues measured in vegetable soils in Romania. A negative correlation is observed between glyphosate and all the physicochemical parameters except for calcium, chloride ion and moisture. Similarly, a negative correlation was observed between cypermethrin and pH, magnesium and calcium, which indicate that an increase in the levels of these soil parameters resulted in a decrease in the pesticide residue levels in the soils. Soil parameters influence the fate of chemical compounds in soil. Thus, an increase in soil parameters such as pH, moisture, water activity, Magnesium, chloride ion resulted an increase in the concentrations of cyperméthrin and glyphosate. These results are comparable to those of Bentum *et al*. (2006) who reported significant negative correlations between pesticide residues with soil pH.

Table 5: Correlation between soil content and pesticide residues

	Cypermét	Carbendaz	Profenofos	glyphosate	Ca	cl	%CO	C/N	%.MO	Azote	Mg	Humidité	Aw	pH
Cypermét	1.00000													
Carbenda	0.49824	1.0000												
Profenofos	0.00265	0.2527	1.00000											
glyphosate	-0.01010	0.4082	0.21788	1.0000										
Ca	-0.01021	0.1001	0.23261	0.0686	1.00000									
cl	0.15458	-0.1395	-0.23604	0.1377	0.14860	1.0000								
%CO	0.42129	0.1470	-0.29692	-0.2804	0.02907	0.3438	1.0000							
C/N	0.17238	-0.0378	-0.22893	-0.2802	-0.08687	0.1822	**0.8737**	1.0000						
%MO	0.42129	0.1470	-0.29692	-0.2804	0.02907	0.3438	**1.0000**	**0.8737**	1.0000					
Azote	0.06354	0.0832	-0.13211	-0.0134	0.18740	0.1838	0.0350	-0.2928	0.0350	1.00000				
Mg	-0.35015	-0.2145	-0.04117	-0.2329	0.16168	-0.0349	-0.0904	-0.0750	-0.0904	0.16835	1.0000			
Humidité	-0.23615	0.0543	0.08816	0.3064	-0.26941	-0.2995	-0.4520	-0.3978	-0.4520	-0.00491	-0.1773	1.00000		
Aw	0.14424	0.0573	0.07506	-0.2769	-0.00293	0.0276	0.1286	0.1668	0.1286	-0.19848	-0.0873	-0.39756	1.00000	
pH	-0.02803	0.0406	0.22883	-0.0211	0.16728	-0.0146	0.0591	0.0929	0.0591	0.13100	0.0700	-0.24519	0.48646	1.0000

Conclusion

The results of this study show that the soils of the three production areas have many similarities except for some parameters such as texture and C/N ratio. The diagnosis of the market garden soils indicate that 79.67% of soil samples are contaminated with the four pesticide residues studied. The presence of these pesticide residues suggested the extensive use of these pesticides in the study area. Pesticide residues detected in soils even in small quantities pose hazards to environment (soil, water, air) and health human due to bioconcentration phenomena. These findings and consequences of the presence of pesticides in soils, biological technology such as biodegradation to eliminate and/or reduce these pesticide residues from the soil should be implemented in order to protect the environment and the health of the population.

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