

DETERMINATION OF TRACE RESIDUES LEVEL OF PESTICIDES IN SOME VEGETABLES GROWING IN ALGERIA BY GC/ μ ECD AND GC/MS

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ABSTRACT

An analytical multiresidue method for the simultaneous determination of various classes of pesticides in vegetables (tomato, pepper, hot pepper and potato) was developed. Vegetable samples are extracted with acetonitrile.

Final determination was made by gas chromatography with μ -Electron Capture Detection (μ -ECD) for organochloride pesticides. Organophosphorus, pyrethrinoids and other pesticides analysis was carried out by gas chromatography coupled with mass spectrometry in the selected ion monitoring (SIM) mode. The identification of compounds was based on retention time and on comparison of the primary and secondary ions. Recovery studies were performed at various fortification levels of each compound and the recoveries obtained ranged from 70% to 127 % with relative standard deviations lower than 8%. The method showed good linearity over the range studied and the detection and quantification limits for the pesticides studied varied from 0.015 to 0.030 $\mu\text{g}\cdot\text{g}^{-1}$ and 0.05 to 0.1 $\mu\text{g}\cdot\text{g}^{-1}$, respectively. The proposed method was used to determine pesticides levels in peppers, hot pepper and tomatoes grown in multichapelle greenhouses at Biskra region and potato grown at Setif region in Algeria.

Keywords: Pesticides residue; Hot pepper, Pepper; Tomato; Potato; GC/ μ ECD; GC/MS.

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1. INTRODUCTION

Pesticides called also agrochemicals are substances or mixture of substances intensely worldwide used in agriculture field in order to protect plants from pests, weeds or diseases, allowing an increased crop yield, and then preserve food security [1]. Also re used in public health protection programs, they can prevent humans from vector-borne diseases, such as malaria, dengue fever, and schistosomiasis [2].

Pesticides have been associated with both health and environmental issues [3-5], and the agricultural use of certain pesticides has been abandoned [4]. Exposure to pesticides can be through contact with the skin, ingestion, or inhalation. The type of pesticide, the duration, exposure voice, and the individual health status are the main factors in the possible health outcome. Also, in a human or animal body, pesticides may be metabolized, excreted, stored, or bioaccumulated in body fat [3-4, 6]. Many negative health effects associated with heavy use of pesticides include, among others, dermatological, gastrointestinal, neurological, carcinogenic, respiratory, reproductive and endocrine effects [3-4, 7-10]. In addition, high occupational, accidental, or intentional exposure to pesticides can result in hospitalization and death [3, 11].

Residues of pesticides can be found in a great variety of everyday foods and beverages, including for instance cooked meals, water, wine, fruit juices, refreshments, and animal feeds [12-19]. Furthermore, it should be noted that washing and peeling are not sufficient to remove completely the residues of pesticides, which represents an order concentration of traces or ultra-traces in the foodstuff considered.

In the most cases, the concentrations do not exceed the legislatively determined safe levels [16, 19, 20-21]. However, these “safe limits” may underestimate the real health risk as in the case of conjointly exposure to two or more chemical substances, which occurs in real-life conditions and may have synergistic effects [3, 22]. Pesticides residues have also been detected in human breast milk samples, and there are concerns about prenatal exposure and health effects in children [6, 23-25].

Over the last 40 years, various sample preparation methodologies have been developed to extract these compounds from foods, although they are generally time-consuming and

laborious. In 2003, Anastassiades, Lehotay, Štajnbaher and Schenck introduced a methodology for pesticide extraction that is applicable to fruits and vegetables. This method, which has been described as quick, easy, cheap, effective, rugged and safe (QuEChERS), has been proven to have several advantages due to its excellent capacities of recovery, enrichment and extraction of the analyte of interest [26-27].

Gas chromatography (GC) and high-performance liquid chromatography (HPLC) are normally the techniques used for pesticide separation in foods [28-29]. The coupling of those systems with mass spectrometers (MS) makes possible the identification and monitoring of pesticides in several matrices [30-31]; besides the possibility of operating in selective ion monitoring (SIM) mode, currently named as single reaction monitoring (SRM), or extracted ion (EI) monitoring mode, and multiple reaction monitoring (MRM) mode (this last for tandem system) that improves the selectivity and sensitivity, and decrease the limits of detection and quantification (LOD and LOQ, respectively) [30-32], these latter are used for all classe of pesticides. However, other techniques use appropriate detectors are specific to restricted families such as for organochlorines, a μ -ECD detector [33], specific for organochlorine pesticides, or an FPD detector specific for organophosphate pesticides is used [34].

In this study, we are interested in determining the level of residues in traces of pesticides in tomato, pepper, hot pepper and potato.

Tomato, hot pepper and hot pepper samples were collected in multi-chapel greenhouses located in the Biskra region (South-eastern Algeria) and potato samples were collected in the Sétif region (North-eastern from Algeria)

2. RESULTS AND DISCUSSION

Tables 1 and 2 group together the concentrations of trace pesticides respectively, organochloride and organophosphorus pesticides in tomatoes, peppers and hot peppers. While, the Table 3 concern organochloride, organophosphorus and other pesticide concentration level in potato

Figure 1 and 2 show respectively, GC/MS chromatogramm of organochlorides pesticides

standard etalon and GC/MS chromatogramm of organophosphorus pesticides standard etalon.

Table 1. Oranochlorides pesticide concentration level in tomato, pepper and hot pepper

Compounds	Retention time (minute)	LD (ppm)	LQ (ppm)	Concentration (ppm)
α -HCH	09.90	0.015	0.050	< L.D
HCB	10.98	0.015	0.050	< L.D
Lindane	11.46	0.015	0.050	< L.D
Heptachlore	11.98	0.022	0.075	< L.D
Aldrine	12.24	0.022	0.075	< L.D
Heptachlore Epoxide	13.05	0.022	0.075	< L.D
α -Endosulfan	13.71	0.022	0.075	< L.D
<i>p,p'</i> -DDE	14.77	0.022	0.075	< L.D
Endrine	15.20	0.020	0.066	< L.D
<i>p,p'</i> -DDD	15.35	0.020	0.066	< L.D
<i>p,p'</i> -DDT	15.47	0.022	0.075	< L.D

Table 2. Oranophosphorus pesticide concentration level in tomato, pepper and hot pepper

Compounds	Retention time (minute)	LD (ppm)	LQ (ppm)	Concentration (ppm)
Dichlorovos	06.27	0.020	0.066	< L.D
Ethoprophos	09.08	0.030	0.10	< L.D
Dimethoate	09.95	0.030	0.10	< L.D
Diazinon	10.34	0.030	0.10	< L.D
Chlorpyrifos-methyl	11.33	0.015	0.05	< L.D
Pirimiphos-methyl	11.74	0.030	0.10	< L.D
Malathion	11.89	0.030	0.10	< L.D
Parathion	12.23	0.020	0.066	< L.D
Ethion	15.01	0.020	0.066	< L.D

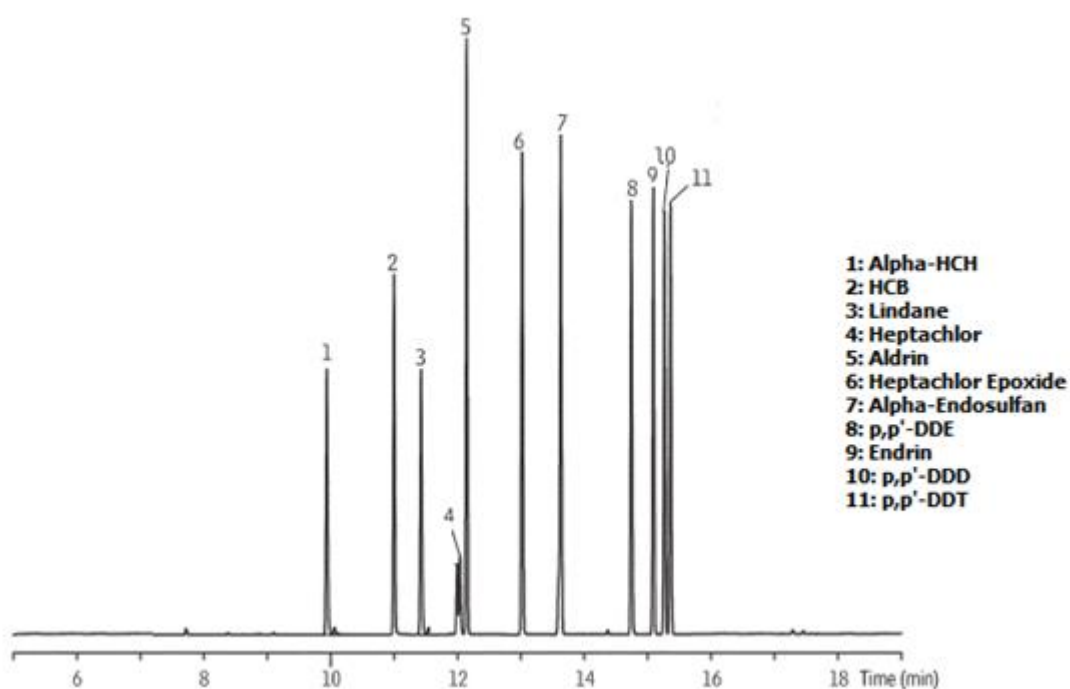


Fig.1. GC/μ-ECD chromatogram of organochlorides pesticides standard etalon

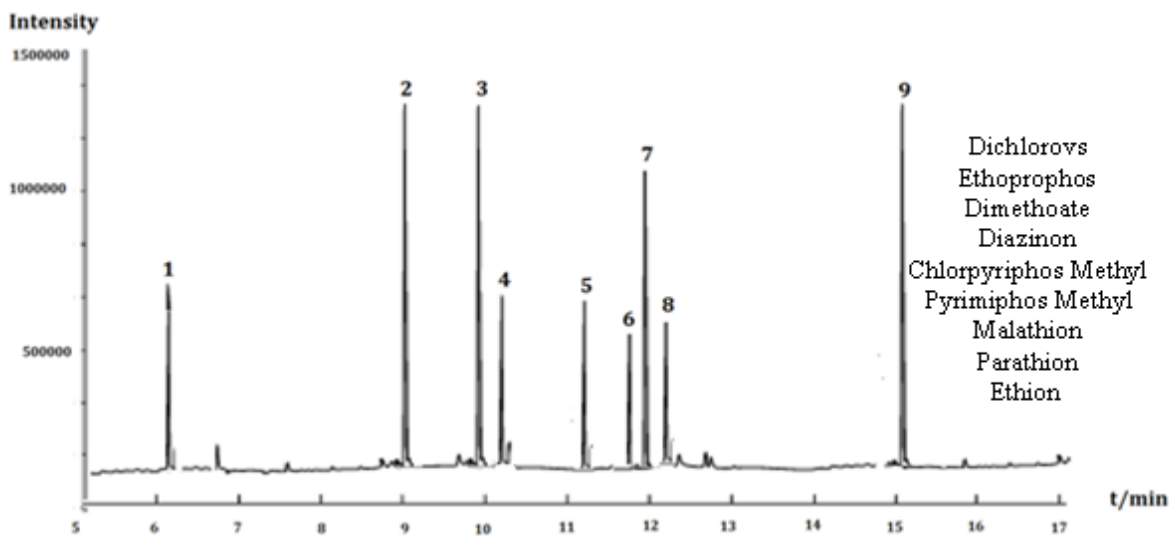


Fig.2. GC/MS chromatogram of organophosphorus pesticides standard etalon

According to the Table 1 and 2, eleven organochloride and nine organophosphorus pesticides were detected at trace level in tomato, pepper and hot pepper. The detection limit varied from 0.01 to 0.02 ($\mu\text{g.g}^{-1}$), and the quantification limit varied 0.05 to 0.1 ($\mu\text{g.g}^{-1}$) for the organochlorides pesticide, however the detection limit varied from 0.01 to 0.03 ($\mu\text{g.g}^{-1}$), and the quantification limit varied 0.05 to 0.15 ($\mu\text{g.g}^{-1}$) for the organochlorides pesticide. The twenty pesticides concentration recorded in tomato, pepper and hot pepper were below the quantification limit.

Table 3 illustrates the concentration of residues in traces of organochlorine, organophosphorus pesticides, pyrethroids and other family of pesticides detected in potato samples.

Sixteen organochlorides, ten organophosphorus, three pyrethroids and five other pesticides were detected in potatoes samples. The detection limit of organochlorides pesticides was 0.015 and the quantification limit was 0.05 for all organochlorides. For the organophosphorus and pyrethroids pesticides, the limit of detection and quantification varied respectively between 0.015 and 0.022 $\mu\text{g.g}^{-1}$ and between 0.05 and 0.075 $\mu\text{g.g}^{-1}$. However, the limit of detection for the other pesticides was 0.015 $\mu\text{g.g}^{-1}$ and the quantification limit was 0.05 $\mu\text{g.g}^{-1}$. All pesticides (organochlorides, organophosphorus, Pyrethroids and other pesticides) detected in all samples (tomato, pepper, hot pepper and potato) were below the quantification limit, since that the presence of organochlorides in all sample even though below the detection limit pose a serious problem, when these latter are classified and listed by Stockholm convention as persistent organic pollutants and banned from any use [35].

However, the organochloride pesticide remains always in foodstuff, especially fruits and vegetables in many countries around Africa, since we find them in atmospheric air in Algeria with high concentration levels [36]. The fact of their presence in atmospheric air necessarily implies their circulation and transfer between different environmental matrix (soil, water and biosphere). The organophosphorus pesticides, pyrethroids and other pesticides detected in this study are sometimes less than or equal to the limit equal to the maximum residue limit (MRLs) reported in the literature for a finished product [37]. It should be noted that the limit of detection and quantification reported in this study can be further improved with other extraction and analysis techniques such as pressure liquid extraction (PLE) with

chromatography in gas phase at atmospheric pressure (APGC) [38], which will make it possible subsequently to quantify the residues of pesticides at the trace scale, even see ultratraces and this makes it possible to determine whether or not we have exceeded the maximum limit residue (MLR). Finally, this work has just been added to the literature in order to increase the data from the analysis of residues in trace amounts of pesticides in fruits and vegetables cultivated in Algeria.

Table 3. Organochlorides, organophosphorus and other pesticide concentration level in potato

Compounds	LD (ppm)	LQ (ppm)	Concentration (ppm)
1- Organochlorides pesticides			
α -HCH	0.015	0.05	< L.Q.
γ -HCH	0.015	0.05	< L.Q.
4,4'-DDE	0.015	0.05	< L.Q.
2,4'-DDD	0.015	0.05	< L.Q.
2,4'-DDT	0.015	0.05	< L.Q.
4,4'-DDD	0.015	0.05	< L.Q.
4,4'-DDT	0.015	0.05	< L.Q.
Aldrin	0.015	0.05	< L.Q.
Alachlor	0.015	0.05	< L.Q.
Butachlor	0.015	0.05	< L.Q.
Dieldrin	0.015	0.05	< L.Q.
Endrin	0.015	0.05	< L.Q.
Endosulfan sulfate	0.015	0.05	< L.Q.
α -Endosulfan	0.015	0.05	< L.Q.
Hexachlorocyclopentadiene	0.015	0.05	< L.Q.
Heptachlor Epoxide Isomère B	0.015	0.05	< L.Q.
Heptachlor	0.015	0.05	< L.Q.
Metolachlor (dual)	0.015	0.05	< L.Q.
2- Organophosphorus pesticides			

Chlorpyrifos-ethyl	0.015	0.05	< L.Q.
Dichlorovos	0.022	0.075	< L.Q.
Disulfoton	0.022	0.075	< L.Q.
Ethoprophos	0.015	0.05	< L.Q.
Fenitrothion	0.022	0.075	< L.Q.
Parathion-methyl	0.015	0.05	< L.Q.
Ronnel	0.015	0.05	< L.Q.
Tokuthion	0.015	0.05	< L.Q.
3-Pyriethrinoids pesticides			
Bifenthrin	0.015	0.05	< L.Q.
Cypermethrin	0.022	0.075	< L.Q.
λ -Cyhalothrin	0.015	0.05	< L.Q.
Others pesticides			
Atrazine	0.015	0.05	< L.Q.
Simazine	0.015	0.05	< L.Q.
Flopet	0.015	0.05	< L.Q.
Fosethyl-Al	0.015	0.05	< L.D.
Cymoxanil	0.015	0.05	< L.D.

3. EXPERIMENTAL

3.1. Sampling sites, periods and locations

3.1.1. Tomato, pepper and hot pepper

The choice of samples was made at several sampling points, distributed in a homogeneous manner; the samples taken are: tomato, pepper and hot pepper, the equidistance between them has been respected; to obtain representative samples; these have almost equal weights; and in its raw state (direct sample in the greenhouse).

The tomato, pepper and hot pepper samples were collected in multi-chapel greenhouses located in the wilaya of Biskra (South-East of Algeria), where today, with 130,000

greenhouses covering more than 5,000 hectares, the collection period was in spring 2017 (see Fig.3.). The samples were transported in the raw state in new containers, in good storage conditions.

3.1.2. Potato

The choice of samples was made at several sampling points, distributed in a homogeneous manner; the samples taken are potato, the equidistance between them has been respected; to obtain representative samples; these have almost equal weights; and in its raw state.

The potato samples were collected in the Sétif region in Winter 2019. this region has a cultivated area representing 85% of the total area. the potato is a widely consumed vegetable in Algeria, for example in 2013 we recorded a production of approximately 5000 ton/year (see Fig.3.). The samples were transported in the raw state in new containers, in good storage conditions.

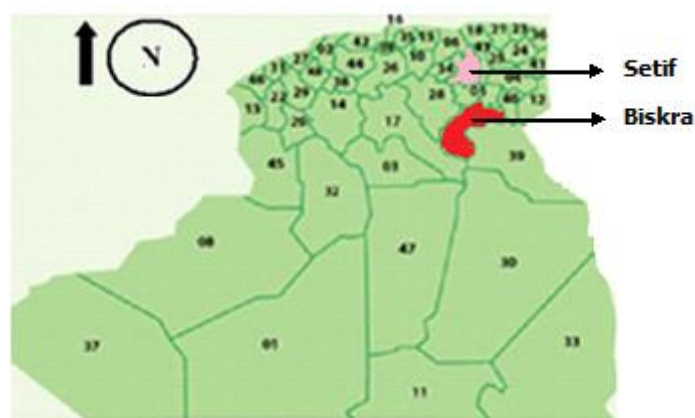


Fig.3. Sampling sites

3.2. Description of the experimental protocol

The protocol used is that appearing on the kits purchased from Supelco (Saint Quentin Fallavier, France) which is thus described. 10 ± 0.1 g of sample, well homogenized and accurately weighed, are introduced into a 50 ml centrifuge tube to which 10 ml of acetonitrile are then added. The tube tightly closed is then vigorously shaken, manually or with a vortex for 1 min. We then add the salt mixture consisting of 4 g of anhydrous magnesium sulfate, 1 g of sodium chloride, 1 g of trisodium citrate dihydrate and 0.5 g of disodium hydrogen citrate

sesquihydrate. The well-screwed tube is shaken vigorously a second time for 1 min then centrifuged at 3000 rpm for 5 min.

An aliquot of 6 mL of supernatant is transferred to a polypropylene tube containing 150 mg of primary secondary amine (PSA), 900 mg of magnesium sulfate and 45 mg of carbon black (Envi-CARB). The tube is shaken vigorously for 2 min then centrifuged at 3000 rpm for 5 min. A 4 mL aliquot of the purified and acidified extract with formic acid to 5% in acetonitrile (10 μ L per ml of extract) is introduced into a pill box and concentrated to dry under a weak stream of nitrogen. The dry residue is taken up in 1 ml of hexane and analyzed by GC/ μ ECD or GC/MS.

3.3. Instrumental Analysis

Organochlorides pesticides were analyzed with an HP GC model 6890 series (Hewlett-Packard Corp., Palo Alto, CA) equipped with a μ ECD set at 300 °C and a Phenomenex ZB-5 widebore capillary column of dimensions 30 m \times 0.53 mm \times 1.25 μ m (Phenomenex, Torrance, CA). The carrier gas was ultra high pure Helium at a rate of 1.1 mL/min and a constant pressure of 6.5 psi. The makeup gas, 5% argon/ methane, was supplied at such a rate that the total column plus makeup flow was constantly equal to 60 mL/min. 4 μ L of the final extract was injected in a splitless mode, at a temperature of 280 °C. The following oven temperature program was used: from 120 to 200 °C at a rate of 5 °C/min with a hold time of 5 min, then from 200 to 240 °C at a rate of 2 °C/min with a final time of 1 min, and finally from 240 to 290 °C at a rate of 5 °C/min. Peak identification was performed by the HP ChemStation calibration table set up with a relative retention time window of 0.65%. Organophosphorus pesticides were analyzed using an HP GC chromatograph coupled with 7000B triple quadrupole mass spectrometer (Agilent Technologies, USA), using Mass Hunter software. Where, the mass ionization was carried out using electron ionization mode at + 70 eV, with a sample injection volume of 1 μ L.

Chromatographic separations were carried out using HP-5 MS Ultra Inert column (30 m \times 0.25 mm, 0.25 μ m) which obtained from Agilent Technologies (USA). A pure Helium gas (> 99.999%) was used as carrier gas with a constant flow rate of 1.83 mL/min. Back flush parameters were installed as follows; holding for 3 min; inlet pressure of 1 psi; three way

splitter pressure of 40 psi; at oven temperature of 280 °C. The oven temperature program was initially held for 1 min at 70 °C, 40 °C/min to 150 °C, and 6 °C/ min to 250 °C. Finally, the oven temp was programed to 315 °C by 15 °C/min (hold for 1 min).

The identification of compounds was carried out by comparing the relative retention times with those of authentic standards and, for MSD analyses, the ion trace ratios of peaks. For quantitative purposes, each compound was referred to the internal standard in the chromatogram.

3.4. Quality assurance and quality control

Quantitative data were kept as reliable when the resulting concentrations lied within the detector operative ranges, i.e., 3.3 up to ~1000 times the respective detection limits LODs; these latter were set equal to 3 times the noise. Considering that ca. 2 kg of sample and final solutions were 10 or 200 µL in volume, the LODs ranged from 0.015 to 0.022 µg.g⁻¹ for organochlorides pesticides, 0.015 to 0.030 µg.g⁻¹ for organophosphorus pesticides, 0.015 to 0.022 µg.g⁻¹ for pyrethrinoids pesticides, and 0.015 µg.g⁻¹ for other pesticides (Atrazine, Simazine, Flopet, Fosethyl-Al and Cymoxanil). The percent recoveries ranged from 70% to 127% (±8%).

4. CONCLUSION

This work allowed us to detect more than 40 pesticides including organochlorine, organophosphorus, pyrethrinoids and other pesticides in the samples of tomato, pepper, hot pepper and potato cultivated in two regions in Algeria, in this case the Sétif and Biskra region. Eleven organochlorides and nine organophosphorus pesticides were recorded in tomato, pepper and hot pepper samples.

Eighteen organochloride, eight organophosphorus, three pyrethrinoids and five other pesticides were detected in potato samples.

All pesticides detected in tomato, pepper, hot pepper and potato were below the quantification limit, even though many pesticides detected were equal or less equal the maximum limit residue (MLR).

Organochlorides pesticides were detected in all samples, although they have been banned for

years and at the same time they are classified as persistent organic pollutants by the Stockholm convention, hence there is a need to monitor their presence in foodstuffs given the major risk it presents to human health

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