CRSTRA
Scientific and Technical Research Centre for Arid Areas

المجلة الجزائرية للمناطق الجافة
Journal Algérien des Régions Arides (JARA)
Algerian Journal of Arid Regions

Research Paper

Origins of pesticide residues in agricultural soils in Biskra (South-East Algeria): survey vs. detection

F. BETTICHE[1*], O. GRÜNBERGER[2], W. CHAÏB[1], H. MANCER[1], K. BENGOUGA[1], M. BELHAMRA[1]

1. Scientific and Technical Research Centre for Arid regions (CRSTRA), Biskra, Algeria.
2. LISAH, Univ. Montpellier, IRD, INRA, Montpellier SupAgro, Montpellier, France.

Received: 06 November 2019 ; Accepted: 18 November 2019 ; Published: December 2019

Abstract

In southern Mediterranean countries, little is known about pesticide inputs under greenhouses, and use is rarely compared with the actual content in the soil. The contamination of agricultural soils by pesticides in the Ziban region (Algeria) was studied by comparing field surveys (farmers, sellers) and direct observations (pesticides packaging) with results of a soil multi-residue analysis using chromatography (liquid and gas) and mass spectrometry detection. Twelve soil samples (six locations at two depths) under tomato greenhouses in six localities (M'Ziraa, Sidi Okba, Ain Naga, El Ghrous, Doucen, and Lioua) were analyzed. The number of active ingredients reported by respondents and observations reached 71 AIs belonging to 34 chemical groups. Despite the wide range of AIs declared to be used by farmers, four other (unexpected) AIs have been identified and quantified by chromatographic analyzes: azinphos-methyl (AZM) (organophosphate), methyl-parathion (organophosphate), and the metabolites p,p'-DDE (organochlorine) and desethylatrazine (triazine). Only AZM and p, p'-DDE were detected in all samples. After examining the possible sources, AZM could be drifting from neighboring regions. The origin of p,p'-DDE was likely the historical use of DDT in locust control or the recent use of dicofol. In conclusion, phytosanitary surveys alone are not sufficient to identify all the AI that can circulate in an agricultural landscape; periodic multi-residue analyses are required to monitor soil contamination. Also, maximum limits for each active ingredient in the soil must be established at the national level to classify a « pollueted » soil.

Key words: Surveys, Multi-residue analysis, GC/MS, LC/MS, azinphos-methyl (AZM), p,p'-DDE, Soil, greenhouses, Biskra.

---

* *Corresponding author : **BETTICHE Farida***
E-mail address: farida.bettiche@gmail.com, bettiche.farida@crstra.dz

e-ISSN: 2676-2226, Published by: Scientific and Technical Research Centre for Arid Areas (CRSTRA)
CRSTRA

1. Introduction

Surveys to list pesticides active substances employed by farmers have been conducted in the Mediterranean region (e.g., Jaradat (2009) and Al-sa'ed et al. (2011) in Palestine; Marucci et al. (2011) in Italy; Ozkan et al. (2004) in Turkey; Fekkoul et al. (2013) and Merouane (2014) in Morocco), including in Algeria (e.g., Siouane (2012) in the north-central area (Blida); Ayad-Mokhtari (2012) in the northwest (Oran, Mostaganem, and Tlemcen); and in the southeast, Ramdani et al. (2009) and Belhadi et al. (2016) in Biskra and Ben Abdelhamid (2016) in El Oued). These surveys identified active substances used for different crops and production systems in different soils and climatic conditions. Nevertheless, only a very few studies, particularly in developing countries, performed simultaneous analyses of the soil matrix in the considered fields (e.g., Ayad-Mokhtari (2012) and Merouane (2014)); Moreover, these last studies (2012 and 2014) considered a limited number of active substances: 2 and 5 respectively, by Gaz Chromatography (GC) or Liquid Chromatography (LC) method.. Discrepancies between molecules employed and detected are explained by the lack of materials and standards according to the first study and are due to the low use of these compounds in the zone and the partial or total transformation of the molecules according to the second one.

The 2015 Algerian index (Downloadable on this site: http://www.minagri.dz/pdf/ Dpvct/INDEX _PRODUITS_PHYTO_2015.pdf) of agricultural phytosanitary products (DPVCT 2015) lists the authorized ingredients (insecticides, acaricides, fungicides, and herbicides), with more than 200 synthetic organic chemical active ingredients that enter into the formulations of almost 900 commercial products. In the past, more than 480 pesticides were registered in Algeria, according to Ayad-Mokhtari (2012). According to Bouziani (2007) in Siouane (2012) and in Louchahi (2015), approximately 400 active ingredients were in approximately 7000 specialties in 2007. Thus, in 10 years, the number of registered AIs decreased by half following the decrease observed in the European Community (EC) in the 90's; however, the Algerian list maintained a few AIs that are prohibited in the EC. Moreover, Ould El Hadj et al. (2011) referred to the use of other AIs that are not included in the 2015 or 2007 Algerian phytosanitary index, including the organo-chlorinated persistent organic pollutants (POPs): aldrin, dieldrin, toxaphene and Hexachlorocyclohexane (HCHs), which were used in locust control in the Algerian Sahara.

A traditional sylvo-agro-pastoral agriculture with large areas of date palms characterizes the Ziban area, near the city of Biskra (Bouziane and Labadi 2009). Currently, the area dedicated to phoeniculture is approximately 42 000 hectares (Directorate of Agricultural Services -DSA-Biskra 2009 in Absi 2013), with more than 4 million palms (Bouammar 2010; Absi 2013). In more recent years, a massive development of market gardening under plastic greenhouses has occurred (Hartani et al. 2015). In 2012, according to the Directorate of Agricultural Services (2013), Biskra represented the highest national level (≈37%) of this development, with a greenhouse area of 3581 ha (Bettiche 2017). Cerruto and Emma (2010) report that pesticide application is a practice closely associated with greenhouse production to improve the quality and quantity of agricultural products. Additionally, pollutants are contained in a fixed space with little chance for degradation under natural conditions (Chen et al. 2013).

The aims of this study were the following: first, list the active ingredients used by greenhouse farmers; second, compare surveys and analytical results for the presence of pesticides commonly used in the greenhouse environment in the Ziban region; and third, question the possible origins of some pesticides when detected in soil.

2. Materials and Methods

2.1. Study area

The study site was the Ziban region (Biskra), located in the north-eastern part of Algerian Sahara (Fig. 1). The study included six localities (Table 1): three in the Ziban East and three in the Ziban West. The six districts, ranked by decreasing vegetable production under greenhouses, are El Ghrous (EG), Ain Naga (AN), Doucen (D), Sidi Okba (SO), Lioua (L) and

M'Ziraa (MZ). These districts represented 65% of the land dedicated to greenhouse crops during the campaign in 2011/2012 (DSA, 2013). The soil was sampled at the six sites (1 sample per locality). Of the protected cultivation areas, 89% are occupied by Solanaceae (tomato, chili, pepper) of which over 50% are dedicated to the cultivation of tomato (2011/2012 campaign). In five sites, farmers chose three tomato greenhouses (400 m2), whereas in the M'Ziraa site, a single, large greenhouse (1 ha) was chosen.

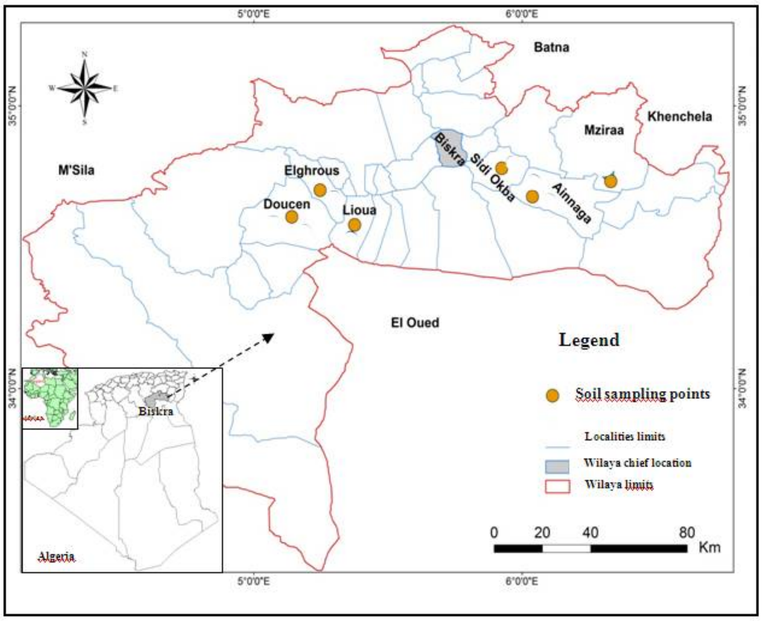

Figure 1: Geographic location of the study area and the sampling points.

2.2. Surveys

Surveys were conducted to determine the pesticides used on greenhouse crops in the Biskra district (Fig.1). These surveys were conducted between 2011/2012 and 2014/2015 in a campaign aimed toward greenhouse farmers (63) and pesticide providers (12) in face-to-face surveys. Surveyed farmers and sellers of chemical inputs were located in the six localities involved in the study. Direct questioning was performed asking the following: "What are the pesticides you use/sell for treatments of diseases and pests on protected crops in greenhouses?" Other non-systematic means of investigation included observations of used pesticide packaging indoors or around greenhouses during our field trips.

2.3. Soil Sampling

Sampling was performed in the 6 tomato greenhouse soils at two depths, (0-5 cm) and (5-30 cm), in April 2014 (end of the tomato cultivation cycle). The 12 samples covered the six districts including MZ, AN, SO, EG, D and L (Fig. 1). At the AN, SO, EG, D and L districts, each sample was composed of fifteen soil subsamples from three tomato greenhouses. Subsamples from each depth of 0-5 and 5-30 cm were collected randomly using a graduated stainless spoon and an Edelman auger at the first and second depths, respectively. For the site of M'Ziraa, the soil sampling was conducted in one Canary type tomato greenhouse (1 ha) with more subsampled points. Because the soils of the large greenhouses were covered with black plastic mulch, the plastic cover was cut through to sample. The subsamples were bulked to form 12 composite samples (2/site, one from each depth). All samples (more than 1 kg) were placed into polyethylene labeled bags, transported to the CRSTRA laboratory, air-dried at room temperature and then passed through a 2 mm sieve. Almost 500 g of each sample was transferred into plastic boxes and stored in a freezer at -18 °C until chromatographic analysis.

Table 1: Data from the six localities and principal cropping systems practiced.

Localities	SAU[1] (ha) (ANAT, 2006)	SUG[2] (ha)	Main cropping systems
M'Ziraa	6941 ha, 4583 ha irrigated: (66.02%)	176	Greenhouses, field crops, irrigated cereals (Naouri et al. 2015), date palms.
Ain Naga	25 060 ha, 11 363 ha irrigated: (45.34%)	554	
Sidi Okba	9625 ha, 8439 ha irrigated: (87.67%)	277	Greenhouses, date palms, fruit trees (in monoculture), field crops, cereals + sheep and cattle breeding (Hamamouche et al. 2015).
El Ghrous	6862 ha, 3103 ha irrigated: (45.22%)	680	Greenhouses, date palms, field crops + arboriculture (for self-consumption) (Bouammar 2012).
Doucen	7462 ha: 6744 ha irrigated: (90.37%)	363	Greenhouses, potato and melon-watermelon, cereal and forage crops (Bouammar 2010), Arboriculture (apricot tree) (Benaziza and Bentchikou 2012), date palms, sheep and cattle breeding.
Lioua	5556 ha, 4923 ha irrigated: (88.60%)	253	Greenhouses, date palms, field crops, cereals.

Notes: [1]Useful agricultural Area; [2]Surface under greenhouses; 1 ha = 100 are.

2.4. Pesticide Multi-Residue Analysis

The samples were analyzed in October 2014 at the organic pollutants unit of the Labocea laboratory according to a specific internal method described as follows. Sample extraction was conducted with acetone in an acid medium. A mixture of internal standards was added to the sample, which was stirred for 15 min after ultrasound, followed by mechanical agitation for 30 min. The acetone extract was then filtered on a folded filter. For molecules analyzed by LC, the filtered acetone extract was diluted 1/100 in Evian water acidified with formic acid. For analysis by Solid phase extraction/triple quadrupole detector (SPE/TQD) (HPLC system and mass spectrometer), 1 ml of extract was injected. For molecules analyzed by GC, an aliquot of the filtered acetone extract was purified on Florisil eluting with methanol. Samples were concentrated by evaporation to 250 µL and then analyzed by GC/MS. The identification of molecules using GC and LC was performed by using an internal method for the determination of organic pollutants in sediment/soil: the yields were corrected by the use of internal standards added immediately before extraction.

2.4.1. GC/MS System

GC separations were performed with a capillary column (60 m, 0.25 mm × 0.25 µm). The flow rate was 1.5 ml/min with an oven temperature of 50 °C and an injection volume of 1 µl in splitless mode. The carrier gas was helium with a pressure of 1.6 psi. The temperature of the oven was programmed as follows: the initial temperature of 50 °C maintained for 1 min, followed by a first temperature gradient of 40 °C/min to 90 °C and a second gradient of 6 °C/min up to 320 °C for 15 min.

3. Results and Discussion

3.1 Survey Results: List of Active Ingredients Used Under Greenhouses in Biskra

The active ingredients (AIs) were directly mentioned by the sellers or identified according to the trade names provided by the farmers, referring to the phytosanitary index, versions 2007 and 2015, published by the Plant Protection and Technical Inspection Department (DPVCT). These AIs were subsequently classified according to chemical group using the pesticide properties database (PPDB) and the bio-pesticides database (BPDB).

When the data collected from the different parties (farmers and sellers) and the various means of investigation (surveys and observations) were combined, the number of AIs reached 71 (including one herbicide) entered into the formulation of more than 100 trade specialties including 34 chemical classes (with one inorganic class). Of these AIs, fungicides were more than half (51%), whereas insecticides and acaricides were slightly less than half (48%), with herbicides representing 1% (only one AI; Table 2). A farmer and/or a seller cited these substances at least once. Belhadi et al. (2016) studied the greenhouse environment during the 2011/2012 campaign in the same 3 localities in the Ziban West and detected 187 AIs, which are not listed entirely in their paper.

With regard to Algerian legislation, 5 (≈8%) of the 60 AIs reported for use by farmers in the 2013/2014 cycle were withdrawn from approval at the end of 2013, including methomyl, zineb, dinocap, amitraz, endosulfan and dicofol. Regarding European rules, 9 AIs (15%) of these 60 AIs were not approved for use. They included the Algerian AIs withdrawn (except methomyl) and four additional AIs (carbendazim, diazinon, hexaconazole and thiocyclam-hydrogen-oxalate). According to the PPDB website (last update 22/02/2017), carbendazim remains in use in Austria, Spain, Poland, Portugal, Romania and the United Kingdom, whereas endosulfan remains in use in Spain, particularly in the Almeria region, the largest greenhouse growing area in the Mediterranean basin dedicated to intensive vegetable production (Gallardo et al. 2013 in FAO 2013). The Sixth meeting of the Stockholm Convention on Persistent Organic Pollutants (POPs) recommended listing endosulfan in Annex A (eliminated POPs), with exemptions. Although approved for use in Europe, only the following countries continue to use methomyl: Bulgaria, Cyprus, Greece, Spain, Hungary, Italy, Malta, Portugal and Romania.

Regarding the health risk and according to the World Health Organization (WHO, 2010), only one active ingredient from the list (ethoprofos) (table 2) has been identified as extremely hazardous (class Ia), and 4 others (abamectin, methomyl, oxamyl and tefluthrin) are highly hazardous (class Ib). The primary 10 chemical groups to which most farmer-reported AIs belonged are the following: avermectins, neonicotinoids, triazoles, carbamates, synthetic pyrethroids, organophosphates, organochlorines, oxadiazines, benzimidazoles and strob ilurins.

Table 2 : List of all chemical substance groups, active ingredients, and the types of pesticide uses mentioned under greenhouses by chemical input providers and farmers between 2011 and 2015, in addition to direct observations of pesticide empty containers around or inside greenhouses.

Substance group	**Active ingredient (AI) (Algerian and European status)**	**Surveyed parts and survey periods (campaign)**				**Direct observations**	**Substance group**	**Active ingredient (AI) (Algerian and European status)**	**Surveyed parts and survey periods (campaign**				**Direct observations**
		Providers (s)			**Farmers**				**Providers (s)**			**Farmers**	
		N=1 (2011/2012)	**N=5 (2012/2013)**	**N=6 (2013/2014)**	**N=63 (2013/2014)**	**(2013/015)**			**N=1 (2011/2012)**	**N=5 (2012/2013)**	**N=6 (2013/2014)**	**N=63 (2013/2014)**	**(2013/015)**
Insecticides							**Fungicides**						
Anthranilic diamide	Chlorantraniliprole (1a, 2a)		+	+	+		Acetamide	Cymoxanil ((1a, 2a)		+	+	+	+
Avermectin	Abamectin (1a, 2a)	+	+	+	+	+	Anilinopyrimidine	Cyprodinil (1a, 2a)				+	
	Emamectin benzoate (1a, 2a)	+	+	+	+		Benzamide	Zoxamide (1a, 2a)				+	
Carbamate	Methomyl (1w, 2a)	+	+	+	+	+	Benzimidazole	Carbendazim (1a, 2na)		+	+	+	
	Oxamyl (1a, 2a)				+			Methylthiophanate (1a, 2a)		+	+	+	
Diacylhydrazine	Tebufenozide (1a, 2a)				+		Carbamate	Mancozeb (1a, 2a)		+	+	+	+
Neonicotinoid	Acetamiprid (1a, 2a)	+	+	+	+	+		Maneb (1a, 2a)		+	+	+	
	Imidacloprid (1a, 2a)	+	+	+	+	+		Propamocarb HCl (1a, 2a)		+	+	+	+
	Thiacloprid (1a, 2a)				+			Propineb (1a, 2a)		+	+	+	+
	Thiamethoxam (1a, 2a)		+	+	+			Zineb (1w*, 2na)				+	
Organochlorine	Endosulfan (1w*, 2na)	+	+	+	+		Chloronitrile	Chlorothalonil (1a, 2a)		+	+	+	+
Organophosphate	Acephate (1w*, 2na)		+				Dicarboximide	Iprodione (1a, 2a)				+	

	Chlorpyrifos-ethyl (1a, 2a)		+	+	+	+	Dicarboximide	Iprodione (1a, 2a)				+	
	Chlorpyrifos-methyl (1a, 2a)		+	+			Dinitrophenol	Dinocap (1a, 2na)				+	
	Diazinon (1a, 2na)		+	+	+	+	Organophosphate	Fosetyl-aluminum (1a, 2a)		+	+	+	+
	Ethoprophos (1a, 2a)				+		Oxazole	Hymexazol (1a, 2a)		+	+	+	
Oxadiazine	Indoxacarb (1a, 2a)	+	+	+	+	+	Phenylamide	Metalaxyl (1a, 2a)		+	+	+	
Synthetic Pyrethroid	Beta-cypermethrin (1a, 2p)					+		Metalaxyl-M (1a, 2a)		+	+	+	+
	Bifenthrin (1a, 2a)		+	+		+	Phenylpyrrole	Fludioxonil (1a, 2a)				+	
	Cypermethrin (1a, 2a)		+	+	+	+	Phenylpyrrole	Fludioxonil (1a, 2a)				+	
	Deltamethrin (1a, 2a)				+		Phthalimide	Folpel/Folpet (1a, 2a)		+	+		+
	Lambda cyhalothrin (1a, 2a)		+	+	+		Pyrimidine	Fenarimol (1a, 2na)		+			
	Tefluthrin (1a, 2a)				+		Quinoline	Quinozol/ Quinosol (1a, 2a)				+	+
Spinosyn	Spinosad (1a, 2a)				+		Strobilurin	Azoxystrobin (1a, 2a)	+	+	+	+	
Unclassified	Buprofezin (1a, 2a)		+	+		+		Kresoxim-methyl (1a, 2a)				+	
	Thiocyclam-hydrogen-oxalate (1a, 2na)		+	+	+	+		Pyraclostrobin (1a, 2a)				+	
Acaricides													
Benzilate	Bromopropylate (1a, 2na)		+					Trifloxystrobin (1a, 2a)				+	
Carboxamide	Hexythiazox (1a,				+		Triazole	Bromuconazole				+	

	2a)							(1a, 2a)					
Amidine (1)	Amitraz (1w*, 2na)	+	+	+	+			Difenoconazole (1a, 2a)		+	+	+	
Organochlorine	Dicofol (1w*, 2na)		+	+	+	+		Epoxiconazole (1a, 2a)				+	
Pyrazolium	Fenpyroximate (1a, 2a)		+	+				Hexaconazole (1a, 2na)	+	+	+	+	+
Pyrethroid	Acrinathrin (1a, 2a)				+			Myclobutanil (1a, 2a)				+	
Sulfite ester	Propargite (1a, 2na)		+	+		+		Penconazole (1a, 2a)	+	+	+	+	+
Tetrazine	Clofentezine (1a, 2a)				+			Tebuconazole (1a, 2a)				+	
Herbicides													
Phosphonoglycine	Glyphosate (1a, 2a)					+		Triadimenol (1a, 2a)	+	+	+	+	+
Total: 34 groups; 71 AIs: 36 Fs (50.7%), 25 Is (35.21%), 9 As (8 + abamectin) (12.67%) and 1 H (1.4%).							Inorganic compounds	Copper (1a, 2a)				+	
								Copper oxychloride (1a, 2a)				+	+
								Sulfur (1a, 2a)				+	+

Notes: +: AI mentioned, 1 Algerian status, 2 European Community status, a: Approved, na: Not approved, w: Withdrawn recently, p: Pending, * December 2013.

Table 3: Number and type of AIs analyzed by GC and LC (identified and not identified by the surveys) in the soil.

Analysed compounds		PAHs[1]	Pesticides	PCB[2]	PBDEs[3]	Various	Total
By GC		**16**	**57**	**11**	**10**	**10**	**104**
By LC		**-**	**41**	**-**	**-**	**-**	**41**
Identified by survey	**< DL**	**-**	**10**	**-**	**-**	**-**	**10**
	> DL	**-**	**5**	**-**	**-**	**-**	**5**
Not identified bysurvey	**> DL**	**9**	**4**	**-**	**-**	**6**	**19**
	< DL	**7**	**79**	**11**	**10**	**4**	**111**
Total		**16**	**98**	**11**	**10**	**10**	**145**

Notes: DL: detection limits. [1] Polycyclic aromatic hydrocarbons, [2] Polychlorinated biphenyl, [3] Polybrominated diphenyl ethers.

Table 4: Concentrations of active substances (µg.kg^{-1} of dry weight) not reported by the surveys and detected in soils under tomato greenhouses.

Active ingredient (AI)	Substance groups	Type	Depth (cm)	Locations						DL (µg.kg^{-1} dw)
				MZ	**AN**	**SO**	**EG**	**D**	**L**	
Azinphos-methyl (AZM)	**Organo-phosphate**	I	0-5	11.1	**8.4**	11.0	22.1	**51.1**	11.5	<2
			5-30	14.8	**36.5**	16.7	**5.5**	24.8	7.8	
Parathion-methyl		I	0-5	<DL	<DL	<DL	**4.59**	<DL	<DL	<2
			5-30	<DL	<DL	<DL	<DL	<DL	<DL	
p,p'-DDE	**Organo-chlorine**	I	0-5	0.89	0.75	0.76	**0.98**	0.85	0.78	<0.1
			5-30	0.91	**0.73**	0.79	0.97	0.84	0.76	
Desethylatrazine (DEA)	**Triazine**	H	0-5	<DL	<DL	<DL	<DL	<DL	<DL	<0.5
			5-30	<DL	<DL	**1.38**	<DL	<DL	<DL	

Notes: I: Insecticide, H: Herbicide, DL: Detection limits, dw: dry weight.

3.2 Analytical Results: Pesticides and Other Organic Micro-Pollutant Detection

Among all AIs identified by the surveys, only 15 were analyzed in the soil, and only 5 were above the detection limit (DL). The content of approximately 60 survey-identified pesticides were under limits of detection for the analytical method. Finally, 4 unexpected active ingredients (3 insecticides and 1 herbicide) were quantified, although they were not recorded in the surveys. In addition to the pesticide AIs, among the non-reported survey organic compounds that exceeded the DL, 9 PAHs and 6 various compounds (benzene derivatives and phthalates, among others) were identified (Table 3). In the analysis of polychlorobiphenyls (PCBs) or polybrominated diphenyl ethers (PBDEs), no compound exceeded the corresponding DL (Table 3). However, only pesticides and those not identified by surveys will be discussed in this paper. Table 4 shows concentrations of the 4 unexpected AIs not on the survey list but detected (at least once) in the soil samples, namely, azinphos-methyl, parathion methyl, p,p'-DDE (DDT-metabolite), and desethylatrazine (atrazine-metabolite).

Because DEA and parathion-methyl were detected only in one site each and at one depth, these compounds were considered accidental. Nevertheless, because the number of sampling points was small, the detection of each could represent a minor but significant practice, which we will discuss as unexpected detections. Either these single detections resulted from analytical errors or possibly from occasional incorrect practices, including use of counterfeit material (reasons from 1 to 4 in figure 2).

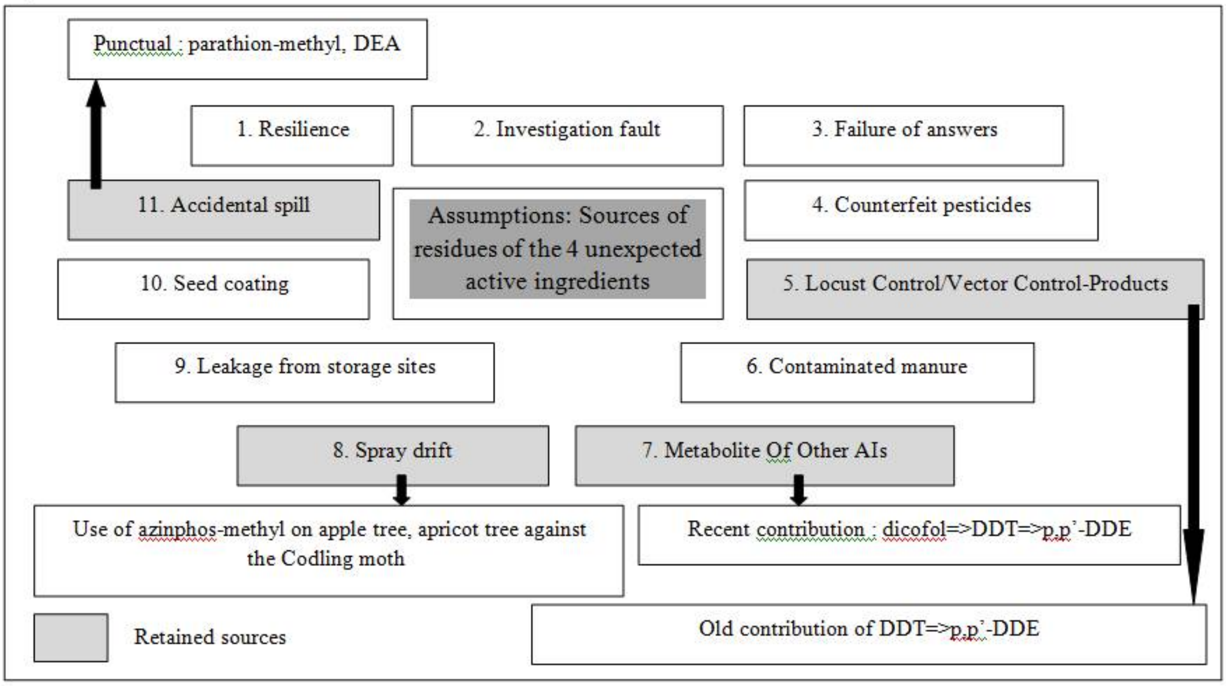

Figure 2: Possible causes of the presence of unsuspected active ingredients in agricultural soils.

However, DEA, a soluble and leachable metabolite from atrazine herbicides, was unexpectedly detected in this context, in which atrazine should not be used. Nevertheless, as Shomar et al. (2006) related, “In spite of being banned, farmers of Gaza use atrazine more than any other pesticide because it is highly effective and less expensive compared to other herbicide currently available.” Thus, atrazine use might have occurred in one of the 6 greenhouses sampled. Detection in the deep layer and the relative persistence of the metabolite (DT50 > 150 days) suggest treatment before plowing, indicating that the treatment may have been applied before the greenhouse was established, which could explain the absence of mention of the AI from the surveys.

Parathion methyl is an obsolete insecticide with a rapid degradation dynamic (DT50 <20 days), and the application was detected on the soil surface. These two elements suggest that past use was not detected, and therefore, the detection was either an analytical error or of illegal use. Note that parathion methyl is classified extremely hazardous (Ia) by the WHO (2010).

Azinphos-methyl and p,p'-DDE were detected systematically in all our samples and at each depth. These active substances were unexpected, and their origins require further discussion. For each unexpected ingredient, we propose 11 hypotheses (Fig. 2) that could explain their presence in greenhouse soils.

Concentrations of AZM in soils of the 3 eastern sites were highest in the deep samples (5-30 cm) unlike those of the 3 western sites, in which contents were highest at the soil surface (0-5 cm). However, these concentrations were all above the detection limit. Although p,p'-DDE concentrations were also above the detection limit, the difference in depths was less pronounced than that for AZM.

3.2.1 Azinphos-methyl (AZM)

Azinphos-methyl (phosphorodithioic acid, S-3,4-dihydro-4-oxo-1,2,3-benzotriazin-3-ylmethyl O,O-dimethyl phosphorodithioate) is an organophosphorus non-systemic pesticide widely used to control a variety of insects on food and non-food crops, ornamentals and forest trees (Flocco et al. 2004); AZM is one of the most heavily applied pesticides in the United States (NCFAP 1997 in Shulz et al. 2003). In the present study, AZM was detected in all studied soils, with the lowest and highest detected concentrations of 5.5 and 51.1 µg.kg-1 for dry weight in the El Ghrous and Doucen soils, respectively (Ziban West). In East Ziban, in Ain Naga, the lowest and highest concentrations ranged from 8.4 to 36.5 $\mu g.kg^{-1}$ dry weight. In Algeria, with the absence of standards for soil pollution, discussion about the levels of soil content for AIs is difficult, because no reference is provided to trigger recommendation, decontamination or depollution actions. In the Khaled-Khodja (2016) study, AZM was one of the pesticides analyzed by "Labocea" in water and sediments of Oueds (rivers) near the Gulf of Annaba (northeastern Algeria, a wilaya/state that produces industrial tomatoes, cereals and citrus) during October 2009, February 2010, May 2010 and August 2010; however, this substance was never detected (maybe because of the rapid degradation ability (DT50 <30 days)). AZM soil concentrations in this study were 2 to 20-fold greater than those found in plum orchard soil in South Africa by Reinecke and Reinecke (2007). According to Oliver et al. (2003), limited data are available about the behavior of AZM in soils around the world. Notably, Reinecke and Reinecke (2007) showed that around a plum orchard in Western Cape, South Africa, AZM concentrations in non-target areas were consistently higher than those in the target areas, leading to the conclusion that AZM was carried by wind to adjacent areas by drift during treatment. According to Quaghebeur et al. (2004) in Lamprea-Maldonado (2009), for typical wind speeds of 3 and 5 $m.s^{-1}$, pesticides can travel distances of 250 and 500 km per day, respectively, in the atmosphere. In the Ziban region during the year, two types of winds occur: northern winds that blow during the winter and spring with a maximum moisture content of 85% (Masmoudi 2009), and the south and southwesterly winds named "siroccos" characterized by the training of sands that are common in March, April and May; over 23 years (1988-2010), the wind maximum monthly average speed (59 $m.s^{-1}$) at Biskra was recorded during the month of April. Greenhouse plastic film is removed every year at the end of the crop cycle, leaving the soil without any cover. Greenhouse locations may change at a maximum every 3 years (for tunnels), giving way to land fallow or field crops (market gardening, cereals, and fodder). Considering these elements, the AZM might have been used before the installation of a new greenhouse or be the result of drifting. This drifting may result from treatments of palm trees; for example, in Israel, since the 1960s, date growers have used various organophosphates for the control of fruit pests (Blumberg 2008). Malathion, which provided the most satisfactory control of nitidulids, was replaced during the early 1980s with AZM, which was more effective against sap beetles. However, the use of this compound has been banned according to the regulations published in 2002 by European Trading Companies (Blumberg 2008). Soroker et al. (2005) describe treatments of red date palm weevil (*Rhynchophorus ferrugineus*) along the northern coast of the Dead Sea. Tree and trunk branches are treated during the period from May to July twice per month with AZM (0.2%) or with diazinon (0.3%). However, we have no evidence of similar use of AZM on date palm in Algeria or in Biskra. Drift might come from the cultivation of other fruits; for example, before the EU ban of AZM, this insecticide was applied against the codling moth *Cydia pomonella* (L.) (Lepidoptera: Tortricidae) in Spain (Rodriguez et al. 2012). In Algeria, AZM is authorized for use against the codling moth on apples, including in the nearby wilaya of Batna (Brahim 2010; DSA-Batna, 2013 in Abdesselam 2016), one of the wilayas bordering (northeast) Biskra (Fig.1). Codling moth targets pome fruits such as apples and pears, in addition to stone fruits and walnuts (Dunley and Welter 2000). AZM has been used on a variety of crops; however, the primary use has been on tree crops, including pome and stone fruit and nut crops (Lewis 2004). In Doucen (western Ziban), where the highest concentration of AZM was detected, fruit arboriculture is represented primarily by apricot trees (Table 1). The flight of the first generation of C. pomonella in the Batna region occurred between April and May from 2004 to 2013 (Regional Station for Plant Protection (SRPV) of Ain Touta 2013 in Abdesselam 2016). This period implies that corresponding treatments are performed during the maximum winds coming from the north and from the south on Biskra. As a consequence, a real drift from the Batna or Doucen fruit tree production zones might explain the AZM in Ziban sites. Temporary drift and the influence of wind sequence and soil contamination before or after tilling might explain the differences in content within different soil layers.

Another concern that will not be addressed here but is relevant to note is that AZM is classified as highly hazardous (Ib) by the WHO (2010). AZM residues can contaminate the food chain through ingested soil by farm animals that graze on the rest of the crops (including roots), and in Doucen, for example, sheep and cattle are raised, which represent a risk for the population through consumption of contaminated meat and/or milk.

3.2.2 p,p'-DDE

Unlike AZM, the concentrations of p,p’-dichlorodiphenyldichloroethylene (p,p'-DDE or 4,4’-DDE) were uniform between depths and among sites. The highest and lowest concentrations were detected at El Ghrous and Ain Naga, respectively, at 0.98 and 0.73 µg.kg^{-1} of dry weight. The direct application of technical DDT or dicofol can introduce DDT into the environment (Wang et al. 2011). In a survey on phytosanitary uses, Ayad-Mokhtari (2012) noted the current use of prohibited products such as DDT and malathion in the region of Oran (northwest Algeria). p,p'-DDE is a metabolite of DDT degradation. Metabolites may be at higher levels in the soil than those of the parent pesticide (Andreu and Pico 2004), because metabolites are more stable and persistent than the parent compound (Quensen et al. 1998 and Yao et al. 2006 in Wang et al. 2007). DDEs can persist in soil for a very long time, potentially hundreds of years (ATSDR 2002 in Kihampa et al. 2010). DDT-related products strongly adhere to soils and therefore remain in the surface layers but may be found in surface and underground waters.

The use of DDT in agriculture was banned in most developed countries in the 1970s (banished by WHO since 1972), and generalized interdiction was effective during the 1980s. Finally, the Stockholm agreement proposed production prohibition and elimination of stocks in the 1990s. However, the WHO recommended DDT for indoor use at its third meeting in May 2007, and the Conference of the Parties of the Stockholm Convention lifted the ban for this use, concluding that the necessity for use of DDT for disease vector control will be evaluated every two years (WHO 2011). Therefore, DTT remains a widely used insecticide in India and African countries to fight malaria (Yu 2014). Malaria ranks first in the Algerian list of infectious diseases; in southern Algeria, 300 cases of malaria were reported between 1980 and 2007 (Hammadi et al. 2009). Algeria is also the most affected country in the Mediterranean and Maghreb by leishmaniasis (Cherif 2014). The number of cases reported in Algeria in 2005 was 30,227 (Guemache et al. 2007), with Biskra first among the wilayas affected that year (2005). In prophylaxis against this transmission, (Guemache et al., 2007) it is advised to kill sandflies, which are the disease vector, with a wide spraying of all the houses before the end of April. Insecticides that could be used are organochlorines, organophosphorus compounds, carbamates and pyrethroids (Rorbet and Chandre 2009).

Similar to all African countries, Algeria is affected by migratory locust invasions. Currently, the organochlorines formerly used in locust control tend to be replaced by other groups such as synthetic pyrethroids that are far less persistent in the environment. According to Ould El Hadj (2011), during a large-scale locust invasion, particularly in the Saharan environment, and in the search for a radical treatment, even pesticides banned in most countries (organochlorines) are used anarchically. However, even in the case of old treatments using DDT, degradation products can always be present.

- **Recent use: DDT contained in dicofol**

Dicofol was ranked first on the list of acaricides sold by the sellers of plant protection products (Bettiche et al. 2017) and was also on the list of active substances reported by sellers and farmers in the present study. Bellamine et al. (2012) suggested in their study in rural Kenitra (west of Morocco) on intensively farmed soil that the source of DDT could be dicofol, which produces DDT as a metabolite. Additionally, Li et al. (2006) found that a DDT impurity in dicofol may be responsible for the recent DDT input into soil in the Pearl River Delta (region of southern China). Dicofol has also been identified by Turgut et al. (2009) in Turkey as a contributing factor to the continuous DDT contamination of soil and water. According to Strandberg et al. (1998) in Benbakhta et al. (2014), the ratio of DDT metabolites, p,p'-DDT/p,p'-DDE, provides a useful index of whether DDT is recent or old (historical). A value <0.33 usually indicates an older entry of DDT. In this study, although p,p'-DDT was analyzed for, it was not detected, suggesting that with p,p'-DDT/p,p'-DDE <0.33, the DDT had undergone a total transformation to p,p'-DDE. Thus, the source of DDT was old. However, this calculation assumes an initial production of DDT and not of dicofol.

Conclusions

Despite the many AIs (71) identified by the various surveys, we did not identify all the AIs that can circulate and contaminate the agricultural soils of the Ziban area. Four unexpected pesticide AIs were identified with the multi-residue analysis technique, which led us to question an approach that was established only on recent surveys and direct observations. The actual trend in analytical methods is that more substances can be investigated on the same soil sample in a few analytical runs, reaching currently approximately 230 ingredients in some European labs at a commercial cost of approximately 1€ per substance. However, this number must be confronted against the fact that potential pesticide pollutants may include the 200 AIs of the actual authorized list in addition to the 200 AIs that were removed from the authorized list, the 12 POPs, and at least 12 metabolites (3 HCHs (α-/β-/γ-), 3 endosulfans (α-/β-/-sulfate), 4 DDTs (o,p'-DDT, p,p'-DDT, p,p'-DDD and p,p'-DDE) and 2 atrazines: deethylatrazine (DEA) and deisopropylatrazine (DIA)). For example, after the study, the possibility remains to detect other AIs of the 255 pesticide AIs that were not identified (-71) by survey and/or analyzed (-98) and have been used in Algeria.

Nevertheless, particularly in southern countries, research or environmental administrations should compare evidence from surveys with analytical facts and specify regular and complementary multi-point monitoring of practices and soil contents. Indeed, soil sampling and analysis will be necessary more often to reflect amounts of AIs and metabolites to prevent a misunderstanding of the contamination situations. This work lists 11 situations that may cause a discrepancy from the surveys (Fig. 2), and we could not imagine a process other than analytical measurements to identify the content of problematic pesticides and metabolites in soil that escape survey listings. The limited number of soil samples analyzed (lack of repetitions) was a limiting factor of the scope of the present work; for example, questions were raised about the two unexpected substances that were measured in two greenhouses soil at two different depths (DEA, parathion methyl) that could not be answered.

All soil samples were contaminated with azinphos-methyl (AZM) and p,p'-DDE. For AZM, the most likely sources were the drifting transport from other regions (bordering wilayas) in which the AI is used in apple orchards against the codling moth. For p,p'-DDE, a metabolite of DDT, two possible sources of non-agricultural and agricultural origin were suspected. On one hand, the resilience of the metabolite from technical DDT formerly used (before 1980) in locust control and/or in agricultural use (before 1960) may produce residues because of the significant half-life; on the other hand, the DDE may be a metabolite of dicofol, which is currently used as an acaricide for greenhouse crops and was reported in our farmer interviews.

In the absence of national or international standards for soil pesticides residues (except for a few countries), the concentrations recorded cannot be used to determine pollutant status. However, contamination is effective, because certain substances reside in the soil beyond the treatment target (depth and time).

Acknowledgments

Part of this study was funded by the Scientific and Technical Research Centre for Arid Areas CRSTRA (National Research Fund, FNR).The authors thank all the staff of « LABOCEA » Labratory (Plouzané-Bretagne, France) who dealt with the analytical part. We are thankful to Dr. D. HEDDADJ and Dr. F.LAKHDARI for their scientific support.

References

Abdesselam Z (2016) Utilisation Des Sucres et Virus de La Granulose Pour La Lutte Contre Le Carpocapse (Cydia Pomonella L.) (Lepidoptera, Tortricidae) En Verger de Pommier Situé Dans La Région d'Inoughissen (Wilaya de Batna). Magister Thesis, University of Batna, Batna, 126p.

Absi R (2013) Analyse de La Diversité Variétale Du Palmier Dattier (Phoenix Dactylifera L.): Cas Des Ziban (Région de Sidi Okba). Magister Thesis, University of Biskra, Biskra, 105p.

Al-sa'ed R, Ramlawi A, Salah A (2011). A Survey on Utilization of Agricultural Pesticides in Selected Palestinian Districts. International Journal of Environmental Studies, 68: 519–529. https://repository.najah.edu/handle/20.500.11888/9677.

Andreu V, Picó, Y (2004) Determination of Pesticides and Their Degradation Products in Soil: Critical Review and Comparison of Methods. Trends in Analytical Chemistry, 23(10–11) : 772–789. DOI: 10.1016/j.trac.2004.07.008.

Ayad-Mokhtari N (2012) Identification et Dosage Des Pesticides Dans l'Agriculture et Les Problèmes D'environnement Liés. Magister Thesis, University of Oran, Oran, 87p.

Belhadi A, Mehenni M, Reguieg L, Yakhlef H (2016) Pratiques Phytosanitaires Des Serristes Maraichers de Trois Localités de L'ouest Des Ziban et Leur Impact Potentiel Sur La Sante Humaine et L'environnement. Revue Agriculture, 1: 9–16. http://revue-agro.univ-setif.dz/documents/numero-special/Session1/Belhadi-et-al.pdf.

Bellamine K, Elabidi A, Benbakhta B, Hajjaji N (2012) Residues of Organochlorines Pesticides in Soil of the Rural Zone of Kenitra (Gharb of Morocco). Continental Journal of Water, Air and Soil Pollution, 3(2) : 20–30. DOI: 10.5707/cjwasp.2012.3.2.20.30.

Ben Abdelhamid F (2016) Evaluation de L'impact Des Produits Phytosanitaire Sur l'Environnement (Cas Des Pesticides Dans La Wilaya d'El-Oued).MasterII Thesis, University of Ghardaïa, Ghardaïa, 126p.

Benaziza A, Bentchikou MM (2012) Recherche Sur La Période D'échantillonnage Des Feuilles D'abricotier Cultivé Dans La Région de Biskra (Algérie). Courrier Du Savoir, 13(avril) : 53–61. http://revues.univ-biskra.dz/index.php/cds/issue/view/49.

Benbakhta B, Khallaf M, Fekhaoui M, Abidi A, Dussauze J, Yahyaoui A (2014) Organochlorine Pesticides in Sediments from the Atlantic Coast of Morocco. International Journal of Innovation and Applied Studies, 6(4) : 1129–1137.

Bettiche F (2017). Usages Des Produits Phytosanitaires Dans Les Cultures Sous Serres Des Ziban (Algérie) et Évaluation Des Conséquences Environnementales Possibles. Phd Thesis, University of Biskra, Biskra, 327p.

Bettiche F, Grunberger O, Belhamra M (2017) Contamination Des Eaux Par Les Pesticides Sous Systeme De Production Intensive (Serres), Cas De Biskra, Algérie. Courrier Du Savoir, 23(Juin) : 39–48. http://revues.univ-biskra.dz/index.php/cds/issue/view/166.

Blumberg D (2008) Date Palm Arthropod Pests and Their Management in Israel. Phytoparasitica, 36(5) : 411–448. DOI: 10.1007/BF03020290.

Bouammar B (2010) Le Développement Agricole Dans Les Régions Sahariennes. Phd Thesis, University of Ouargla, Ouargla, 296p.

Bouammar B (2012) L'agriculture Saharienne et Sécurité Alimentaire En Algérie. La sécurité alimentaire et l'agriculture saharienne. Pp. 99–105. https://manifest.univ-ouargla.dz/documents/Archive/Archive Faculte des Sciences de la Nature et de laVie et des Sciences de la Terre et de l'Univers/La-securite-alimentaire-et-lagriculture-saharienne-fevrier2012-/Bouammar_Boualem.pdf.

Bouziane MT, Labadi A (2009) Les Eaux Profondes de La Région de Biskra (Algérie). European Journal of Scientific Research, 25(4) : 526–537. http://www.eurojournals.com/ejsr.htm.

Brahim I (2010) Étude Du Comportement de Ponte Du Carpocapse (Cydia Pomonella L.) (Lepidoptera, Tortricidae) Sur Deux Variétés de Pommier. Magister Thesis, University of Batna, Batna, 192p.

Cerruto E, Emma G (2010). Exposure to Pesticides: A Survey on Sicilian Greenhouse Operators." Pp. 236–43 in International Conference Ragusa SHWA2010 - September 16-18, 2010 Ragusa Ibla Campus- Italy Work Safety and Risk Prevention in Agro-food and Forest Systems. http://www.ragusashwa.it/CD_2010/lavori/TOPIC3/orale/10SHWA4-Safety greenhouse workers.pdf.

Chen Y, Huang B, Hu W, Weindorf DC, Yang L (2013) Environmental Assessment of Closed Greenhouse Vegetable Production System in Nanjing, China. Journal of Soils and Sediments, 13(8) : 1418–1429. DOI: 10.1007/s11368-013-0729-8.

Cherif K (2014) Etude Éco-Épidémiologique de La Leishmaniose Cutanée Dans Le Bassin Du Hodna (M'Sila). Phd Thesis, University of Setif, Setif, 197p.

DPVCT (2015) Index Des Produits Phytosanitaires À Usage Agricole 2015. Direction de la protection des végétaux et des contrôles techniques, 217p. http://www.minagri.dz/pdf/Dpvct/INDEX_PRODUITS_PHYTO_2015.pdf.

Dunley JE, Welter SC (2000) Correlated Insecticide Cross-Resistance in Azinphosmethyl Resistant Codling Moth (Lepidoptera: Tortricidae). Journal of Economic Entomology, 93(3) : 955–962. DOI: 10.1603/0022-0493-93.3.955.

FAO (2013) Good Agricultural Practices for greenhouse vegetable crops. Principles for Mediterranean climate areas.Book, 640p. http://www.fao.org/3/a-i3284e.pdf.

Fekkoul A, Zarhloule Y, Boughriba M, Barkaoui A, Jilali A, Bouri S (2013). Impact of Anthropogenic Activities on the Groundwater Resources of the Unconfined Aquifer of Triffa Plain (Eastern Morocco). Arabian Journal of Geosciences, 6 : 4917–4924. DOI: 10.1007/s12517-012-0740-1.

Flocco CG, Carranza MP, Carvajal LG, Loewy RM, Pechén De D'Angelo AM, Giulietti AM (2004) Removal of Azinphos Methyl by Alfalfa Plants (Medicago Sativa L.) in a Soil-Free System. Science of the Total Environment, 327(1–3) : 31–39. DOI: 10.1016/j.scitotenv.2003.08.024.

Guemache S, Zellegui H, Hamdi chérif M (2007) Epidémiologie de La Leishmaniose Cutanée. SEMEP CHU Setif , PPT, 27p. www.snapo.org/files/leishmaniose%205.ppt.

Hamamouche MF, Kuper M, Lejars C (2015) Émancipation Des Jeunes Des Oasis Du Sahara Algérien Par Le Déverrouillage de L’accès À La Terre et À L’eau. Cahiers Agricultures, 24(6) : 412–19. DOI: 10.1684/agr.2015.0777.

Hammadi D, Boubidi SC, Chaib SE, Saber A, Khechache Y, Gasmi M, et al (2009) Le Paludisme Au Sahara Algérien. Bulletin de La Societe de Pathologie Exotique, 102(3) : 185–192. DOI: 10.3185/pathexo3356.

Hartani T, Naouri M, Kuper M (2015) L’Entrée Des Jeunes Dans L’Agriculture: Cas Du Maraîchage Sous Serre Dans Les Ziban (Algérie). Alternatives Rurales, 69–78. http://alternatives-rurales.org/wp-content/uploads/HSJeunes/AltRurHSJeunesHartaniEtAlLectEcran.pdf.

Jaradat KMA (2009). Adsorption and Desorption Characteristics of Endosulfan Pesticide in Three Soils in Palestine. Degree Of Science, An-Najah National University, Nablus, 100p. DOI: 10.2462/09670513.1003.

Khaled-Khodja S (2016) Évaluation de La Qualité Physico–chimique Des Rejets Anthropiques (Urbains, Agricoles et Industriels) Au Golfe d’Annaba. Phd Thesis, University of Anaba, Anaba, 186p.

Kihampa Ch, Ram Mato R, Mohamed H (2010) Residues of Organochlorinated Pesticides in Soil from Tomato Fields , Ngarenanyuki , Tanzania. Journal of Applied Sciences and Environmental Management, 14(3) : 37–40. www.bioline.org.br/ja.

Lamprea-Maldonado DK (2009) Caractérisation et Origine Des Métaux Traces, Hydrocarbures Aromatiques Polycycliques et Pesticides Transportés Par Les Retombées Atmosphériques et Les Eaux de Ruissellement Dans Les Bassins Versants Séparatifs Péri-Urbains. Phd Thesis, Ecole centrale de Nantes, Nantes. https://tel.archives-ouvertes.fr/tel-00596847/file/doc00005291.pdf.

Lewis CM (2004) Azinphos-Methyl (Guthion) Risk Characterization Document (Revision No.1).Report, 166p. DOI: 10.1007/s13398-014-0173-7.2

Li J, Zhang G, Qi S, Li X, Peng X (2006) Concentrations, Enantiomeric Compositions, and Sources of HCH, DDT and Chlordane in Soils from the Pearl River Delta, South China. Science of the Total Environment ,372(1) : 215–224. DOI: 10.1016/j.scitotenv.2006.09.023

Louchahi MR (2015) Enquête Sur Les Conditions D’utilisation Des Pesticides En Agriculture Dans La Région Centre de L’algérois et La Perception Des Agriculteurs Des Risques Associés À Leur Utilisation. Magister Thesis, Ecole Nationale Superieure d’Agronomie, Alger, Alger, 103p.

Marouane B (2014) Transfert Des Nitrates et Des Pesticides Dans Les Sols de La Région Du Gharb- Etude À L’échelle de La Parcelle. Phd Thesis, Mohammed V University, Rabat, 147p. http://toubkal.imist.ma/handle/123456789/10003.

Marucci A, Campiglia E, Colla G, Pagniello B (2011) Environmental Impact of Fertilization and Pesticide Application in Vegetable Cropping Systems under Greenhouse and Open Field Conditions. Journal of Food, Agriculture & Environment, 9(3 - 4) : 840–846.

Masmoudi R (2009) Etude de La Fiabilité Des Systèmes de Distribution D’eau Potable En Zones Arides Cas de La Région de Biskra. Phd Thesis, University of Biskra, Biskra, 183p. thesis.univ-biskra.dz/998/1/Hydrau_d1_2009.pdf.

Naouri M, Hartani T, Kuper M (2015) Mobilités Des Jeunes Ruraux Pour Intégrer Les Nouvelles Agricultures Sahariennes (Biskra, Algérie). Cahiers Agricultures, 24(6) : 379–386. DOI: 10.1684/agr.2015.0778.

Oliver DP, Kookana RS, Salama RB (2003) Land Use Effects on Sorption of Pesticides and Their Metabolites in Sandy Soils. I. Fenamiphos and Two Metabolites, Fenamiphos Sulfoxide and Fenamiphos Sulfone, and Fenarimol and

Azinphos Methyl. Australian Journal of Soil Research, 41(5) : 847–860. DOI: 10.1071/SR02094.

Ould El Hadj MD, Bouziane N, Minami A, Ould El Hadj-Khelil A (2011) Problèmes de La Lutte Chimique Au Sahara Algérien: Cas Des Acridicides. Algerian Journal of Arid Environment, 1(1) : 77–83. https://dspace.univ-ouargla.dz/jspui/bitstream/123456789/6684/1/E010109.pdf.

Ozkan B, Kurklu A, Akcaoz H (2004) An Input-Output Energy Analysis in Greenhouse Vegetable Production: A Case Study for Antalya Region of Turkey. Biomass and Bioenergy, 26(1) : 89–95. DOI: 10.1016/S0961-9534(03)00080-1.

Ramdani N, Tahri N, Belhadi A (2009) Pratiques Phytosanitaires Chez Les Serristes Maraîchers Des Localités de Tolga et de Sidi-Okba (Wilaya de Biskra). Journal Algérien Des Régions Arides, 8 : 73–80. DOI: 10.1017/CBO9781107415324.004.

Reinecke SA, Reinecke AJ (2007) The Impact of Organophosphate Pesticides in Orchards on Earthworms in the Western Cape, South Africa. Ecotoxicology and Environmental Safety, 66(2) : 244–251. DOI:10.1016/j.ecoenv.2005.10.006.

Rodríguez, M.A., Bosch,D., & Avilla, J (2012) Azinphos-Methyl and Carbaryl Resistance in Adults of the Codling Moth (Cydia Pomonella (L.), Lepidoptera: Tortricidae) from Northeastern Spain. Pesticide Biochemistry and Physiology, 103(1): 43–48. DOI: 10.1016/j.pestbp.2012.03.002.

Rorbet V, Chandre F (2009) Pesticides et Lutte Antivectorielle Vecteurs Du Paludisme et Des Arboviroses. P. 41 in Colloque de restitution des travaux du 1er plan d'action ORP (Observatoire des Résidus de Pesticides) 2006-2008 Paris, Maison de la Chimie, 11 & 12 mars 2009. https://docplayer.fr/13312341-Pesticides-et-lutte-antivectorielle.html.

Schulz R, Hahn Ch, Bennett ER, Dabrowski J.M, Thiere G, Peall SKC (2003) Fate and Effects of Azinphos-Methyl in a Flow through Wetland in South Africa. Environmental Science & Technology, 37(10) : 2139–2144. https://www.ncbi.nlm.nih.gov/pubmed/12785519.

Shomar BH, Müller G, Yahya A (2006) Occurrence of Pesticides in Groundwater and Topsoil of the Gaza Strip. Water, Air, and Soil Pollution, 171 : 237–251. DOI: 10.1007/s11270-005-9038-1.

Siouane MR (2012) Comparaison Des Pratiques Phytosanitaires Dans Différents Systèmes de Culture. Application À La Plaine de Metidja En Algérie. Master of Science, Institut Agronomique Méditerranéen de Montpellier, Montpellier, 96p.

Soroker V, Blumberg D, Haberman A, Hamburger-Rishard M, Reneh S, Talebaev S, et al (2005) Current Status of Red Palm Weevil Infestation in Date Palm Plantations in Israel. Ecotoxicology and Environmental Safety, 66(1) : 244–251. DOI: 10.1007/BF02980931.

Turgut C, Gokbulut C, Cutright TJ (2009) Contents and Sources of DDT Impurities in Dicofol Formulations in Turkey. Environmental Science and Pollution Research, 16(2) : 214–217. DOI: 10.1007/s11356-008-0083-3.

Wang F, Jiang X, Bian YR, Yao FX, Gao HJ, Yu GF, et al (2007) Organochlorine Pesticides in Soils under Different Land Usage in the Taihu Lake Region, China. Journal of Environmental Sciences, 19(5) : 584–590. DOI: 10.1016/S1001-0742(07)60097-7.

Wang Y, Guo S, Xue R, Qi S, Xu Y, Xue B, et al (2011) Organochlorine Pesticides in the Soil of a Karst Cave in Guilin, China. Environmental Monitoring and Assessment, 180 : 489–500. DOI 10.1007/s10661-010-1801-0.

WHO (2010) The Who Recommended Classification of Pesticides By Hazard and Guidelines To Classification 2009. http://www.who.int/ipcs/publications/pesticides_hazard/en/.

WHO (2011) Global Malaria Programme. The Use of DDT in Malaria Vector Control WHO Position Statement, 16p. http://apps.who.int/iris/bitstream/handle/10665/69945/WHO_HTM_GMP_2011_eng.pdf;jsessionid=1B6A2597D26B13C6B0CB734F516A5D0D?sequence=1.

Yu SJ (2014) The Toxicology and Biochemistry of Insecticides, Second Edition. CRC Press. https://books.google.dz/books?hl=fr&lr=&id=4023BgAAQBAJ&oi=fnd&pg=PP1&dq=Yu+SJ.+2014.+The+Toxicology+and+Biochemistry+of+Insecticides&ots=fXMnFWDL1d&sig=OzyDyYngmHF6jE69jTorPxqejVM&redir_esc=y#v=onepage&q=Yu SJ. 2014. The Toxicology and Biochemistry of Insecticides&f=false.