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Research Article

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Assessment of Soil and Water Pollution from Organophosphate and Organochlorine Pesticides Linked to Farming Practices in the Binah Prefecture, Togo

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Abstract: In the Binah prefecture of Togo, the growing practice of market gardening during the dry season and the uncontrolled use of unregistered pesticides significantly contribute to soil and water pollution. This study aimed to quantify the residues of banned organophosphate and organochlorine pesticides for the first time in water and soil to assess the extent of contamination. In February 2021, eleven soil and nine water samples were collected from areas near market gardening sites. These samples were analyzed using gas chromatography-mass spectrometry (GC-MS) after extraction through pressurized liquid extraction (ASE) and solid phase extraction (SPE). The results revealed the presence of six organophosphate pesticides (out of the eighteen targeted) in both matrices, with all levels remaining below the standards set by the World Health Organization (WHO). In contrast, organochlorine pesticides were found in higher concentrations. Heptachlor (0.04 µg/L) and Aldrin (0.032 µg/L) exceeded WHO potability standards at the Sirka and Ketao sites. Among soil samples, Pagouda was the most contaminated site, with a total concentration of 74.42 µg/kg, followed by Boufale at 23.98 µg/kg and Kemerida at 15.24 µg/kg. This study underscores the need to address the risks posed by banned persistent pesticides and calls for global strategies to mitigate these issues.

Keywords: Banned pesticides, SPE, ASE, Kara Region, Agricultural Area

1. Introduction

The 2008 food crisis in the world in general [1, 2], and in Togo in particular, prompted the Togolese government to focus on the profitability of agricultural activities to ensure food security and improve the standard of living of its population. Food security and dietary diversity declined significantly from 2007 to 2008 while prices rose [3]. Planned Agricultural Development Zones (ZAAP), each covering at least 100 hectares, have been established nationwide and in Binah to contribute to developing Togolese agriculture. With the continuous support of the Technical Support Advisory Institute (ICAT) in these ZAAPs, a significant increase in productivity has been observed. The Binah prefecture, known as the granary of the Kara region during the study period, is seeing an increase in the use of pesticides for market gardening and cash crops, leading to a growing demand for phytosanitary products. These findings are in line with the national statistics. The total importation of agricultural pesticides increased from 2,141.899 tons to 6,841.564 tons, representing a 68.7% increase, between 2008 and 2022. the official import quantities of hazardous pesticides decreased by 95.9%, from 1,243 tons to 51 tons during the same period (FAOSTAT statistics).

In light of the strong demand for plant protection products, establishing a parallel network for supplying pesticides is raising concerns in the country. Specifically, the porous borders with neighbouring countries [4] facilitate the uncontrolled entry of various agricultural inputs, whether or not they come with usage instructions showing the challenges in pesticide risk communication [5]. As a result of this source of supply, a wide variety of formulations are being used on market garden crops in Togo, uncontrolled and often with a complete lack of knowledge of good practice in the use of these products, which are known to be dangerous. This has led to an increase in the use of agricultural inputs, particularly pesticides. The consequence of this widespread use is an increase in pest resistance to pesticides, leading to an increase in the doses used and, therefore, probably more significant impacts on the health of producers, consumers, and the environment [6].

Pesticides include all products used to combat undesirable plant species and organisms considered harmful. Whether pesticides are authorized today or were authorized in the past, there are concerns about their possible effects on human health and, more broadly, the environment [7]. Several factors affect the infiltration and movement of pesticides into groundwater, including rainfall intensity, land slope, soil texture and type, vegetation, soil water content, irrigation practices, and the physicochemical properties of pesticides, such as water solubility and stability [8]. The synthetic pesticides used in modern agriculture are xenobiotics (manufactured) designed to control insects and weeds. However, these pesticides are harmful to humans and the environment in general [9, 10]. Organophosphate pesticides (OPs) toxic chemical compounds are often neurotoxic and disrupt the nervous system by blocking cholinergic transmission. The toxicity of these compounds can be linked to either acute or chronic exposure [11] and is thought to be responsible for many cases of poisoning worldwide [6]. Studies have shown that these compounds can cause brain damage in children, even at low levels of exposure. The use of organophosphates is globally significant due to their low cost, adsorption potential, and broad spectrum of activity. A recent study indicates a significant association between the consumption of well water contaminated with organochlorine pesticides and a high prevalence of Alzheimer's and Parkinson's diseases linked to neurological disorders [12] in an agricultural area in Akkar, Lebanon. Few previous in Togo have shown the use of organochlorine pesticides and the presence of their residues in environmental compartments [4, 13–15].

To mitigate risks related to pesticide use, the Togolese government has implemented measures for the rational management of chemical pesticides. Aware of the dangers associated with using plant protection products, the government of Togo has adopted several regulations to control their commercialization and use to protect human health and ecosystems. Internationally, these include conventions such as the Stockholm and Rotterdam Conventions, the FAO Code of Conduct on Pesticide Management, and the Basel Convention, among others [13, 16]. Nationally, there is the phytosanitary regulation law in Togo, Law No. 96-007/PR of July 3, 1996, concerning plant protection, which prohibits the importation, repackaging, storage, experimentation, use, or marketing of any pesticide product that is not approved. Additionally, Decree No. 98-099/PR of September 30, 1998, outlines the application of this law, along with various ministerial orders [6]. However, the Togolese market is saturated with counterfeit products and underdosed or overdosed pesticides, which lead to a loss of effectiveness, insect resistance phenomena, and increased potential risks to human health and the environment [17]. The occurrence of organochlorine pesticide residues in biological and environmental matrices has been revealed in Africa and the remote territories of the Kerguelen Islands [18–20] in subantarctic shallow water samples showing their import on faraway territories.

There is no relevant data on organophosphorus and organochlorine pesticides in the Binah prefecture. This work aimed to assess the presence of these compounds in the soils and waters near the market gardening areas of the Binah prefecture in Togo.

2. Materials and Methods

Geographical location of the study area

The Binah prefecture is one of the 07 prefectures of the Kara region. It has a surface area of 480 km^2, representing 4.1% of the surface area of the Kara region. It is between the Kabyè Mountain (Atakora range) and the Benino-

Togolese plain. To the North, it is bordered by the Doufelgou Prefecture, to the south by the Kozah Prefecture, to the East by the Republic of Benin, and to the West by the Kozah and Doufelgou Prefectures. It is located at 9°45’0’’N and 1°16’60’’E. The study covered 09 sites located in the nine (09) cantons (Ketao, Sirka, Kemerida, Solla, Boufale, Pagouda, Pitikta, Lama-dessi (Farende) and Pessare) of the Binah prefecture (**Figure 1**) [21]. The prefecture enjoys a Sudanian climate characterized by a dry season from November to April, influenced by the Harmattan, with cumulative rainfall of less than 50 mm. January and February record no rainy episodes and are characterized by thermal peaks of around 31°C. The rainy season extends from May to October and is dominated by Monsoon, with average temperatures of 26°C and maximum rainfalls in July and October [22]. The population is estimated at 84,199, corresponding to 8.5% of the population of the Kara region according to the fifth census of the Togolese population [21]. The main agricultural activities are the production of sorghum, millet, rice, soybeans, beans, voandzou, cassava, yams, cotton (during the rainy season), and market gardening (during dry season) based on tomatoes, peppers, okra, spinach, ademè (Corchorus olitorius), onions, cabbage and eggplant production.

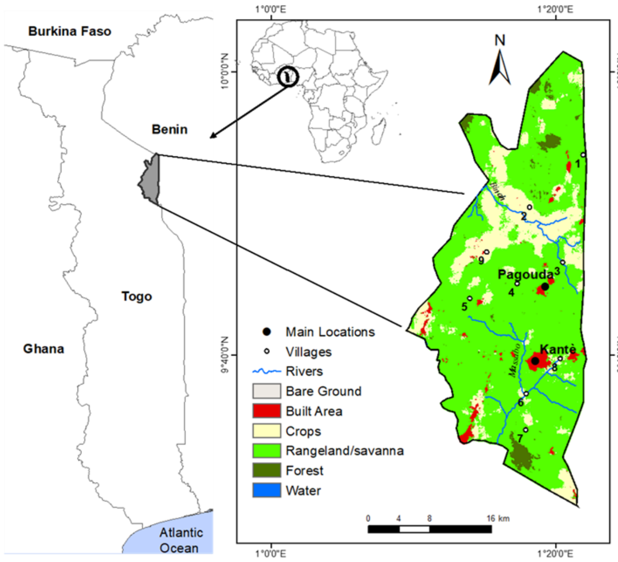


Figure 1: Map of sample sites

Table 1: Description of sampling sites

			Localization for soil sampling sites		Localization for water sampling sites	
Number	**Canton**	**Village**	**X**	**Y**	**X**	**Y**
1	SOLLA	TININGOU	1.36454	9.90023	1.36555	9.90195
2	BOUFALE	LAMATESSI 1	1.30272	9.84027	1.30272	9.84027
	BOUFALE (cotton culture)	LAMATESSI 1	1.302495	9.840706		
3	PAGOUDA	PAGOUDA	1.34135	9.74679	1.34146	9.77531
4	PITIKTA	KATAARA	1.28772	9.74991	1.28824	9.75032
5	LAMA-DESSI (FARENDE)	FARENDE	1.23294	9.73291	1.23274	9.73296
6	KETAO	BOMDE	1.298036	9.62174	1.298743	9.62084
7	SIRKA	SANGAYLAO	1.298298	9.578404	1.29820	9.578236
	SIRKA (cotton culture)	SANGAYLAO	1.297931	9.579571		
8	KEMERIDA	GBLIDE	1.337125	9.661464	1.338579	9.662104
9	PESSARE	TCHADE	1.252648	9.788301	1.25255	9.78747

The sampling campaign took place in February 2021. A total of 11 soil samples against 09 water samples were taken **(Table 1)**, nine from soils with market gardening activities in the nine cantons (Boufale, Pitikta, Sirka, Solla, Lama-dessi, Kemerida, Ketao, Pagouda, Pessare) [21] of the Binah prefecture. In comparison, two soil samples were named from soils with cotton-growing activities (Boufale 1 and Sirka 1). Water reservoirs used as drinking water during agricultural periods were sampled near market gardening sites and used as drinking water during agricultural activities

Soil sampling method

Soil sampling was conducted according to a protocol described elsewhere [23]. Briefly, on a study site, the selected area is first gridded into a perimeter of 100 m² (10 x 10). A soil sample is taken to a depth of 15 cm below the crop surface in each grid using an auger. The samples are then carefully mixed to produce a composite sample representative of the study area. A 1 kg quantity of this composite sample is air-dried for 24 hours, then coarsely crushed by hand to reduce aggregates. The sample is then stored at 4°C in an amber glass bottle.

Sampling method for water catchment areas

Water samples were taken in 1.5-liter amber glass bottles. After being sealed and marked with the identification code, the bottles were covered with aluminum foil, placed in a cooler, and transported to the laboratory, where they were kept at 4°C.

Pesticides investigated

Standard organophosphate pesticide (**Table 2**) solutions were purchased from RESTEK (reference 32563; multi-residue solution of 16 compounds, each at 100 µg/mL in toluene) and LGC standards (Profenofos, 10 µg/mL in cyclohexane and triazophos, powder). Organochlorine pesticide (**Table 3**) solutions were also purchased from RESTEK, where concentrations in these solutions were 200 mg/L for each OCP (organochlorine pesticides AB no. 1, Restek no. 32291) and LGC standards (Chlordecone, 10 µg/mL dans 10 mL dans Isooctane). These standard solutions were used to prepare an external calibration ranging from 10 ng/mL to 250 ng/mL for each compound in dichloromethane (DCM) using at least 5 standards (R^2> 0.999). The limits of detection (LOD), limits of quantification (LOQ), and other quality control (accuracy, precision, and reproducibility) of the analytical techniques were performed as mentioned in [9]. LOD and LOQ were estimated based on the background noise following equations 1 and 2.

$$LOD=3*h\_max*R \quad (1)$$

$$LOQ=10*h\_max*R \quad (2)$$

R represents the ratio of high calibration curve concentration (250 µg/L) to the average of the corresponding areas for each compound.

h_max is the average of the maximum amplitudes recorded around the retention time for each compound for the blank solution 0 µg/L of DCM.

The solubility in water (S), octanol-water partition coefficient (Log Kow), and soil adsorption coefficient (Koc) values are gathered in **Table 2**. S (mg/L) considered for values <= 50, in the range 50 - 500, > 500 is low, moderate, and high, respectively. Additionally, Log Kow is considered low, moderate, and high hydrophobicity with a high level of bioaccumulation for values < 2.7, in the range 2.7 – 3 and > 3.0. Koc measures the mobility of a substance in the soil. High values (>75) indicate that the substance is strongly adsorbed to soil particles and organic matter, limiting its movement within the soil. Conversely, a low value (< 15) suggests that the substance is highly mobile in the soil. Koc is a crucial parameter for estimating a chemical substance’s environmental distribution and exposure levels.

Table 2: List of organophosphorus pesticides studied and some physicochemical properties [24]

N°	Compound	Number CAS	Class	MW (g/mol)	RT (min)	Products ions (m/z)				Log Kow	Koc (ml/g)	Solubility at 20°C (mg/L)	LOD (µg/L)	LOQ (µg/L)	LOD (µg/kg)	LOQ (µg/kg)	WHO (µg/L) in drinking water
1	Diazinon	333-41-5	Insecticide	304.35	21.36	179	137	152	-	3.7	609	60	0.00137	0.00457	0.00055	0.00183	NA
2	Isazophos	42509-80-8	Insecticide	313.74	23.88	161	97	119	-	3.1	155	69	0.00219	0.00731	0.00088	0.00293	NA
3	Chlorpyrifos-methyl	5598-13-0	Insecticide	322.53	25.27	186	125	288	-	4	4645	2.74	0.00101	0.00336	0.0004	0.00135	30
4	Pirimiphos-methyl	29232-93-7	Insecticide fumigant	305.33	25.8	290	276	305	-	4.2	1100	11	0.00078	0.00259	0.00031	0.00103	NA

5	Chlorpyrifos	2921-88-2	Insecticide	350.59	26.31	97	197	199	-	4.7	5509	1.05	0.00089	0.00297	0.00036	0.00119	30
6	Fénitrothion	122-14-5	Insecticide	277.23	26.39	277	125	109	-	3.3	2000	19	0.00142	0.00472	0.00057	0.00189	NA
7	Pirimiphos-ethyl	23505-41-1	Insecticide	333.39	26.57	333	318	304	-	4.8	300	93	0.00089	0.00295	0.00035	0.00118	NA
8	Quinalphos	13593-03-8	Insecticide	298.3	27.58	146	157	156	-	4.4	1465	17.8	0.00362	0.01207	0.00145	0.00483	NA
9	Profenofos	41198-08-7	Insecticide	373.63	28.248	337	339	208	139	1.7	2016	28	0.27985	0.93283	0.11194	0.37313	NA
10	Triazophos	24017-47-8	Insecticide	313.31	30.11	161	77	97	162	3.5	358	35	0.04005	0.13349	0.01602	0.0534	NA
11	EPN*	2104-64-5	Insecticide	323.3	31.43	157	169	63	-	5.02	4000	0.5	0.00557	0.01858	0.00223	0.00743	NA
12	Pyridafenthion	119-12-0	Insecticide	340.33	32.4	77	97	125	-	3.2	7211	100	0.03925	0.13084	0.0157	0.05234	NA
13	Phosalone	2310-17-0	Insecticide	367.81	32.6	182	121	97	367	4.0	2063	1.4	0.00839	0.02797	0.00336	0.01119	NA
14	Pyrazophos	13457-18-6	Fungicide	373.36	33	221	232	237	-	3.8	646	4.2	0.01318	0.04393	0.00527	0.01757	NA
15	Pyraclofos	77458-01-6	Insecticide	360.8	34.03	139	194	360	-	3.8	2095	33	0.01702	0.05674	0.00681	0.0227	NA
16	Azinphos-methyl	86-50-0	Insecticide	317.32	34.08	160	77	132	-	2.9	1112	28	0.00588	0.0196	0.00235	0.00784	NA
17	Azinphos-ethyl	2642-71-9	Insecticide	345.38					-	3.2	1500	4.5	0.00588	0.0196	0.00235	0.00784	NA
18	Phosmet	732-11-6	Insecticide	317.32	34.87	160	77	93	-	2.8	3534	15.2	0.01175	0.03918	0.0047	0.01567	NA

*EPN: (O-Ethyl O-(4-nitrophenyl) phenylphosphonothioate)

Table 3: List of organochlorine pesticides studied and some physicochemical properties [9, 24, 25]

N°	Compound	Number CAS	Class	MW (g/mol)	RT (min)	Products ions (m/z)				Log Kow	Koc (ml/g)	Solubility at 20°C (mg/L)	LOD (µg/L)	LOQ (µg/L	LOD (µg/kg)	LOQ (µg/kg)	WHO (µg/L) in drinking water
1	Alpha-BHC	319-84-6	Insecticide	290.83	19.72	181	183	219		3,82	1888	2.0	0,00002	0,00006	0,000008	0,000024	NA
2	Gamma-BHC	58-89-9	Insecticide	290.83	22.28	183	181	111		3.5	1270	8.52	0,00002	0,00007	0,000008	0,000028	2
3	Bêta-BHC	319-85-7	Insecticide	290.83	24.28	109	111	181		3,8	2807	0.24	0,00004	0,00014	0,000016	0,000056	NA
4	Heptachlor	76-44-8	Insecticide	373.32	24.45	100	272	274		5.44	24000	0.056	0,00002	0,00006	0,000008	0,000024	0.03 (+ HE)
5	Delta-BHC	319-86-8	Insecticide	290.83	25.34	181	183	219		4.1	2807	31.4	0,00002	0,00005	0,000008	0,00002	NA
6	Aldrin	309-00-2	Insecticide	364.9	25.59	66	263	91		6.5	17500	0.027	0,00031	0,00102	0,000124	0,000408	0.03 (+ dieldrin)
7	Heptachlor epoxide (HE)	1024-57-3	Insecticide	389.3	27.07	81	353	351		4.98	22485	0.2	0,00002	0,00007	0,000008	0,000028	0.03 (+ Heptachor)
8	Cis-Chlordane	5103-71-9	Insecticide	409.78	27.55	375	373	377		2.78	20000	0.1	0,00039	0,00129	0,000156	0,000516	2
9	Trans-Chlordane	5103-74-2	Insecticide	409.78	27.73	375	373	377					0,00009	0,00028	0,000036	0,000112	2
10	Endosulfan I	959-98-8	Insecticide	406.93	27.79	237	239	235		4.75	11500	0.32	0,00017	0,00055	0,000068	0,00022	-
11	4-4' DDE	72-55-9	Insecticide	328.03	28.15	246	248			6.51	50118	0.12	0,00024	0,00082	0,000096	0,000328	1 (+4-4' DDD 4-4' DDT)
12	Dieldrin	60-57-1	Insecticide	380.91	28.32	79	82	81		3.7	12000	0 .14	0,00074	0,00246	0,000296	0,000984	0.03 (+ Aldrin)
13	Endrin	72-20-8	Insecticide	380.91	28.91	81	79	82		3.2	10000	0.24	0,00074	0,00246	0,000296	0,000984	0.6
14	Chlordecone	143-50-0	Insecticide	490.63	29.11	272	274	269		4.5	2500	3.0	0,00003	0,0001	0,000012	0,00004	
15	Endosulfan II	33213-65-9	Insecticide	406.9	29.21	235	237	239		4.75	11500	0.32	0,00017	0,00055	0,000068	0,00022	
16	4-4' DDD	72-54-8	Insecticide	320.04		235	165	237		6.02	131000	0.09	0,00023	0,00077	0,000092	0,000308	1 (+4-4' DDE 4-4' DDT)
17	4-4' DDT	50-29-3	Insecticide	354.49	29.46 29.75	235	237	165		6.91	151000	0.006	0,00023	0,00078	0,000092	0,000312	1 (+4-4' DDD 4-4' DDE)
18	Endrin aldehyde	7421-93-4	Insecticide	380.91	29.957	67	66	347		4.80	4300	0.02	0,00074	0,00246	0,000296	0,000984	NA
19	Endrin Ketone	53494-70-5	Insecticide	380.91	30	67	147	221		4.80	4300	0.02	0,00003	0,0001	0,000012	0,00004	NA
20	Endosulfan sulfate	1031-07-08	Insecticide	422.9	30.41	385	387	389	272	3.66	5194	0.48	0,00031	0,00102	0,000124	0,000408	NA

21	Methoxychlor	72-43-5	Insecticide	345.65	31.43	227	169	152	113	5.83	80000	0.1	0,00003	0,0001	0,000012	0,00004	20

DDE: dichlorodiphenyldichloroethylene; DDD: dichlorodiphenyldichloroethane; DDT: dichlorodiphenyltrichloroethane

Extraction of target compounds from samples

Soil samples were sieved to a particle size of 250 microns, freeze-dried at -80°C (CRYOTEC COSMOS 20K), and finely ground (ULTRA-TURRAX® Tube Drive) before extraction. Target compounds were extracted from the soil matrix using pressurized liquid extraction (ASE) (Dionex ASE 350, Thermo Scientific Inc., Waltham, USA), followed by a solid phase extraction (SPE) step (Autotrace 280, Thermo Scientific Inc., Waltham, USA) of liquid extract. The extraction conditions of liquid samples are presented in **Table 4**. The extraction process for pressurized liquid extraction (ASE) of targeted compounds in soil samples used a sample mass of 2.5 g per cell with a cell volume of 34mL. Dichloromethane was chosen as the solvent, and the extraction was performed at a temperature of 100°C and a pressure of 100 bar. A total of 5 static cycles were conducted, each lasting 4 minutes. The total extracted volume of solvent was 60mL. These conditions are designed to enhance the extraction of compounds from the sample by using both high temperature and pressure, which are known to improve the solubility and efficiency of extraction, especially for more difficult-to-extract compounds. Each extract is then evaporated to dryness under a stream of nitrogen (TurboVap LV system, Biotage AB, Uppsala, Sweden) and recovered in 1 mL dichloromethane for analysis. The water samples were centrifuged for 10 minutes at 4°C at 11,000 rpm to remove suspended solids and then filtered under vacuum through a polyvinylidene fluoride (PVDF) membrane with a pore size of 0.45 µm (Durapore, Merck KGaA, Darmstadt, Germany). The samples were then extracted using solid-phase extraction (SPE) following the conditions described in **Table 4**. Each extract was evaporated to dry under a nitrogen stream (TurboVap LV system, Biotage AB, Uppsala, Sweden) and then reconstituted in 1 mL of dichloromethane for analysis.

Table 4: Conditions for solid-phase extraction (SPE) of targeted compounds

Parameter	Conditions
Cartridge	Oasis HLB 6 cc, 200 mg (Waters)
Cartridge conditioning	5 mL methanol + 5 mL EUP
Volume loaded per cartridge (mL)	500
Loading flow rate (mL min-1)	10
Rinsing	5 mL EUP
Drying	Under N_2 flow (10 mL min-1)
Elution	5,5 mL dichloromethane

Analysis by gas chromatography coupled to a mass spectrometer.

Pesticides were analyzed by gas chromatography (HP 6890, Agilent) coupled to a mass spectrometer (MS 5973, Agilent) (with an ion source in electron impact mode) in Selected Ion Monitoring (SIM) mode. Chromatographic separation was performed on an ultra-inert DB 35-MS column (0.25 mm x 30 m; 0.25 µm, Agilent). The carrier gas was helium at a constant 1 mL/min flow rate. GC temperature was programmed as follows: initial temperature of 90˚C (held for 1.3 min), increased to 125˚C at 15˚C/min, then to 165˚C at 5˚C/min, then to 195˚C at 2.5˚C/min and finally to 280˚C at 20˚C/min (held for 4 min).

3. Results and Discussion

Evaluation of pesticide content in water samples

Organophosphorus pesticides (OP) in water samples

The results indicate variable detection frequencies among the eighteen (18) compounds investigated. Six (6) organophosphorus pesticides were identified in water samples, with the following detection frequencies: diazinon (6 times), fenitrothion (4 times), pyrazophos (4 times), chlorpyrifos (2 times), quinalphos (2 times), and azinphos-methyl (1 time), all at concentrations below the limit of quantification (see **Table 2**). The remaining twelve (12) compounds were not detected. These findings aid in understanding the absence of guideline values proposed by the World Health Organization in the fourth edition [25], considering the concentration level below an eventual health risk value. These guidelines suggest that the contamination of selected pesticides in drinking water occurs at concentrations well below levels of health concern [25]. Among the selected pesticides, only chlorpyrifos has a health-related value of 30 µg/L, which aligns with the annual concentration limit established by the Environmental Quality Standard (EQS) through the Directive 2013/39/UE [26] for inland surface water.

Of these compounds, diazinon was the most frequently detected in 6 of the nine (9) samples, while azinphos-methyl was detected only once in the Kemerida sample.This frequency of diazinon detection confirms that this compound, a broad-spectrum organothiophosphate derivative, is widely used as an agricultural insecticide and veterinary medicinal product [27]. Its presence in water samples could be linked to its recent use and the high solubility (60 mg/L) that facilitates the drift into water [24]. As diazinon [28], all detected organophosphate pesticides are not persistent [24]. Chlorpyrifos was only detected at two sites in Kemerida and Pitikta at concentrations below LOQ, contrary to the results of [7], which found a high detection frequency of 100% in the Middle Part of Mono River Basin in Togo, probably due to a significant input of countless tributaries and intensive use in cotton fields in this part of the country. Previous research has demonstrated the use of pesticides in sub-Saharan African countries and the exposure effects, especially in women during pregnancy, and abnormal neurological development in newborns [29, 30]. Only chlorpyriphos was officially registered in Togo at the study date [31]. However, it has a broad-spectrum effect, from killing the target species to human immunological, neurological development, reproductive, and neurological disorders. Thus, these health risks have led to the ban of chlorpyrifos in many countries, while its use is limited in China and other densely populated South Asian countries [32]. None of these pesticides are approved in the European Union [33].

Organochlorine Pesticides (OCP) in water samples

The global concentration of organochlorine pesticides per site is represented in **Figure 2**. Organochlorine pesticides banned from import and use in Togo by ministerial decrees n° 31/MAEP/SG/DA of 21/09/2004 and n° 0078/18/MAEP/Cab/SG/DPV of 17/05/2018 were also detected in water samples. Fifteen pesticides were detected in water samples. Endrin ketone, endosulfan sulfate, methoxychlor, chlordecone, trans-chlordane, and heptachlor epoxide were below their respective LOD in water samples. Eight (8) organochlorine pesticides were detected one (1) time (**Figure 3**): with alpha-BHC (0.038 µg/L) in Kemerida, heptachlor (0.04 µg/L), delta-BHC (< LOQ), cis-Chlordane (< LOQ), 4-4' DDE (< LOQ), and endrin (0.046 µg/L) in Sirka, 4-4' DDT (0.003 µg/L) in Pessare and Endrin aldehyde (0.018 µg/L) in Pagouda. Other organochlorine pesticides were detected at several times at sampling sites with diverse ranges of concentration: lindane or gamma-BHC (< LOQ - 0.060 µg/L; 8 times), endosulfan II (< LOQ - 0.140 µg/L; 7 times), 4-4' DDD (0.011-0.040 µg/L; 4 times), dieldrin (0.002 - 0.010 µg/L; 3 times) and bêta-BHC (< LOQ -0.08 µg/L; 3 times), aldrin (0.00600 -0.032 µg/L; 2 times) and endosulfan I (0.01000- 0.01600 µg/L; 2 times). Endosulfan II and bêta-BHC (< LOQ - 0.08 µg/L) had the highest concentrations.

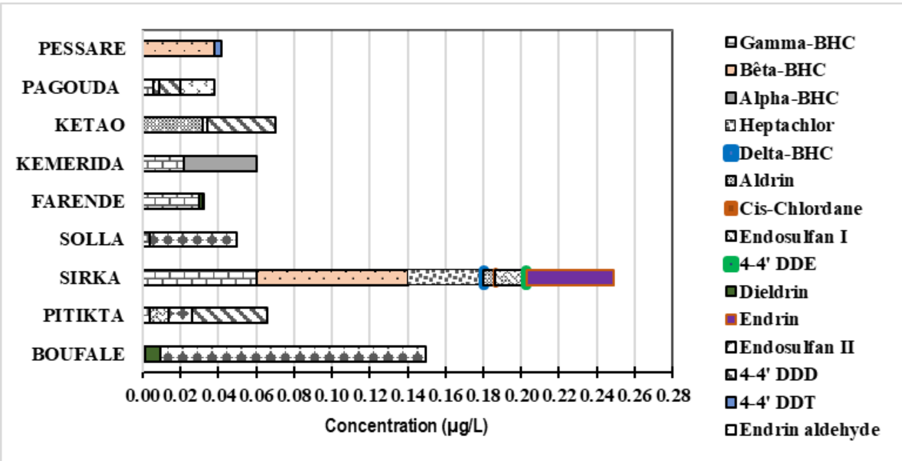


Figure 2: Organochlorine pesticide concentrations (µg/L) in water samples

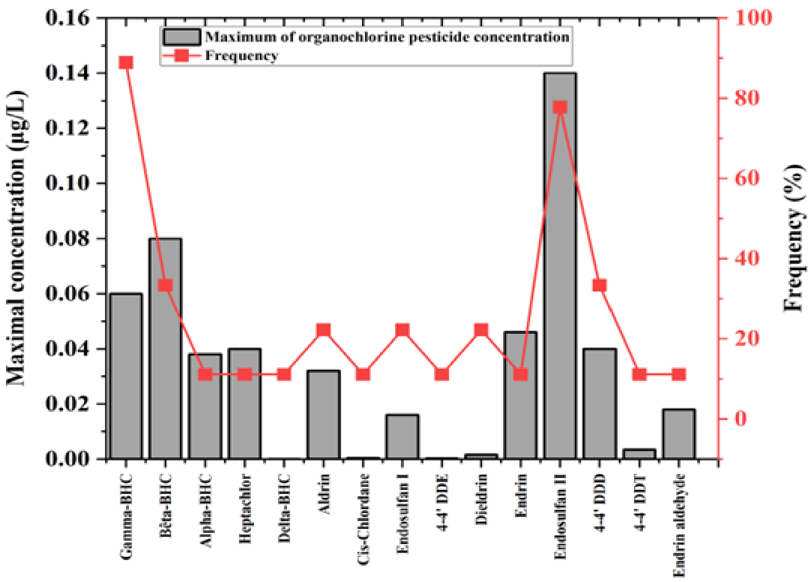


Figure 3: Distribution of organochlorine pesticides (µg/L) and their respective frequency of detection in water samples

Delta-BHC, 4-4' DDE, and cis-Chlordane in the Sirka water sample, bêta-BHC in the Kemerida sample, and gamma-BHC and endosulfan II in the Pessare water sample were detected below their respective LOQ. The detection of technical grade or lindane (gamma-BHC) can be determined by analyzing the ratio of alpha-BHC to gamma-BHC (µg/L). Lindane usage of 99% gamma-HCH or commercial mixtures containing several HCH isomers with alpha-BHC (65%) and gamma-HCH (~12.5%) may result in HCH in water samples. The ratio of alpha-BHC to gamma-BHC serves then as an indicator of the source of HCH. A ratio close to 0 and below 4 indicates the use of lindane [9, 34]. Most ratios are close to 0 (**Table 5**), with the highest detection frequency of gamma-BHC (88.88%) showing the primary use of lindane for agricultural purposes in the past, probably due to the intense use and physicochemical parameters. Indeed, among HCH pesticides, lindane has the lowest value of Log Kow (3.5) and the highest solubility value at 8.52 mg/L.

These results are similar in the tendency to study reports on organochlorine pesticide (OCP) residues in the aquatic environments along the Shaying River in China [35]. However, the authors reported a very low concentration in the order of ng/L in surface and related groundwater samples for HCH, DDT, and metabolites, showing old uses characterized by the high amount of primary compound comparatively to their metabolites [36]. Another study from Asa Dam River in Nigeria showed that the levels of organochlorine pesticides ranged from 0.0036–0.093 µg/kg and 0.001–0.007 µg/L in soil and water samples, respectively [15], confirming that soil samples are more concentrated than water samples in OCPs. All concentrations in water samples are below the WHO guidelines value of 2 µg/L for lindane. DDT and its metabolites, as well as the sum of aldrin and dieldrin and the overall sum of lindane and its isomers (ΣHCH), have values below the WHO recommendation for drinking water except at the Ketao site for aldrin and dieldrin (0.032 µg/L) (**Table 5**). Ultimately, Heptachlor (0.04 µg/L) and Aldrin (0.032 µg/L) present concentrations above WHO potability standards at the Sirka and Ketao sites. However, regarding the quality of water intended for human consumption, European Union Drinking Water Directive 98/83/EC has also adopted a concentration of 0.1 µg/L for a single pesticide and 0.5 µg/L for the sum of pesticides as acceptable values for drinking water [9, 37]. This directive also presents an individual value of 0.030 µg/L for Aldrin and Dieldrin, heptachlor, and heptachlor epoxide and all samples display pesticide concentrations below these guideline values. The results obtained in this study are in line with previous investigations showing that pesticide mixtures are ubiquitous in surface water worldwide, even at lower concentrations [38]. However, the bioaccumulation and biomagnification of total pesticides may lead to cumulative adverse effects in aquatic biota and humans. Globally, pesticide mixtures posed a potential risk to human health, and among the six continents, Africa had the highest proportion of sites at risk from pesticide mixtures [38]. Most organochlorine pesticides that contribute significantly to mixture toxicity have been banned in many countries [32, 33]. Thus, their presence is attributed to their persistence and illegal use, particularly in Africa, where stricter regulations are required. Other factors include insufficient knowledge of safe pesticide use, the unregulated application of banned or expired products, and improper safety protocols among farmers.

Table 5: Average concentrations of active ingredients in water reservoirs per site

Cantons	Detected OPs	Concentration (µg/L)
BOUFALE	Diazinon	< LOQ
PITIKTA	Chlorpyrifos	< LOQ
	Fenitrothion	< LOQ
	Diazinon	< LOQ
SIRKA	Fenitrothion	< LOQ
	Quinalphos	< LOQ
SOLLA	Diazinon	< LOQ
	Pyrazophos	< LOQ
FARENDE	Pyrazophos	< LOQ
	Diazinon	< LOQ
KEMERIDA	Chlorpyrifos	< LOQ
	Azinphos-méthyl	<LOQ
	Pyrazophos	< LOQ
KETAO	Fenitrothion	< LOQ
	Diazinon	< LOQ
PAGOUDA	Diazinon	< LOQ
	Fenitrothion	< LOQ
PESSARE	Quinalphos	< LOQ
	Pyrazophos	< LOQ

Evaluation of pesticide content in soil samples

Evaluation of organophosphate pesticides in soil samples

Analysis of pesticide residues in the eleven soil samples studied showed that six (6) organophosphorus pesticides, all banned by the European Union [37], were found (**Figure 4**) out of the eighteen (18) investigated. These molecules are (**Figure 5**): pyrazophos (< LOQ - 12 µg/kg; 8 times), fenitrothion (0.3 - 1.96 µg/kg; 4 times), chlorpyrifos (0.04 - 1.54 µg/kg; 4 times), isazophos (< LOQ -13.04 µg/kg; 3 times), triazophos (0.4 - 8.7 µg/kg; 2 times) and chlorpyrifos-methyl (0.8 µg/kg; 1 time). Triazophos and chlorpyrifos-methyl were detected in soil contrary to the water sample. Similarly, diazinon, quinalphos and azinphos-methyl with respective solubility of 60 mg/L, 17.8 mg/L and 28 mg/L, were only detected in water samples. Organophosphate pesticide residue determination in water and sediment samples of River Benue in Nigeria reveals similar results, with diazinon present in soil and water but at a concentration lower than in sediment samples, probably due to microorganisms such as Pseudomonas sp, Flavobacterium sp, and Agrobacterium sp activity [10, 28]. On the contrary, fenitrothion was quantified at similar concentrations in sediment and water.

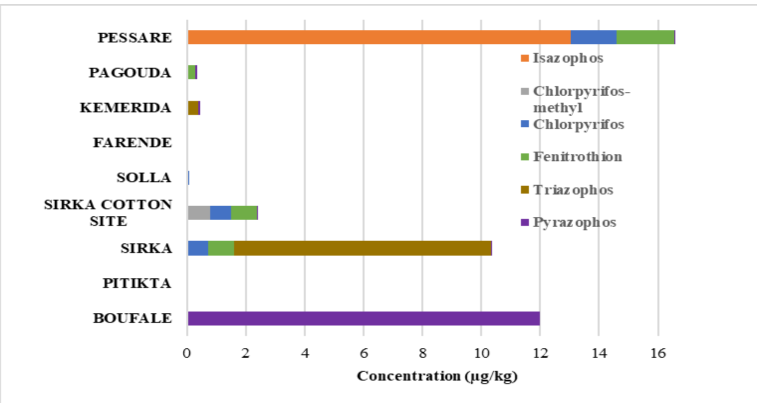


Figure 4: Average concentrations of detected organophosphorus pesticide (µg/kg) in soil per site

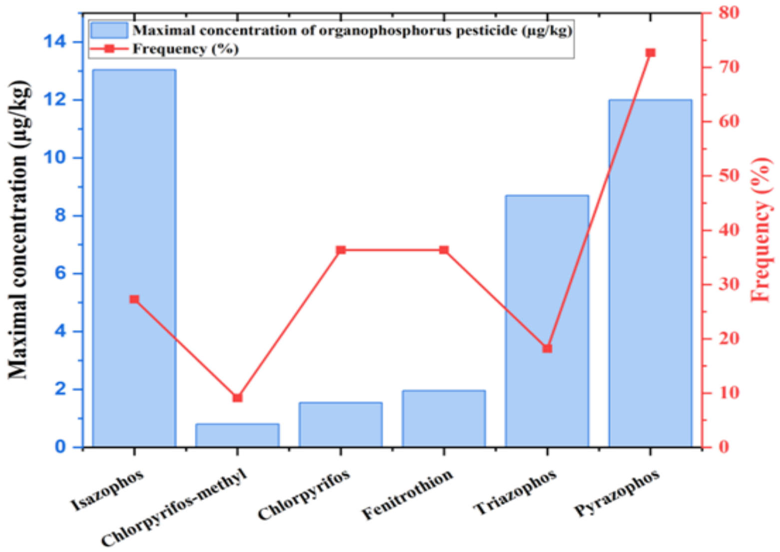


Figure 5: Maximum concentration per detected organophosphate pesticide (µg/kg) in soil and their respective frequency of detection

Table 6: Some groups of specific pesticides and their concentration in water samples at sample sites

Name	BOUFALE	PITIKTA	SIRKA	SOLLA	FARENDE	KEMERIDA	KETAO	PAGOUDA	PESSARE
ΣDDT and metabolites (µg/L)	0.000000	0.040000	0.000240	0.000000	0.000000	0.000000	0.036000	0.011000	0.003400
ΣHCH (µg/L)	0.001400	0.004000	0.140020	0.004000	0.030000	0.060040	0.000000	0.006000	0.038020
Aldrin + dieldrin (µg/L)	0.008000	0.000000	0.006000	0.000000	0.001600	0.000000	0.032000	0.000000	0.000000
Alpha-BHC/Gamma-BHC (µg/L)	0.000000	0.000000	0.000000	0.000000	0.000000	1.727273	NA	0.000000	0.000000

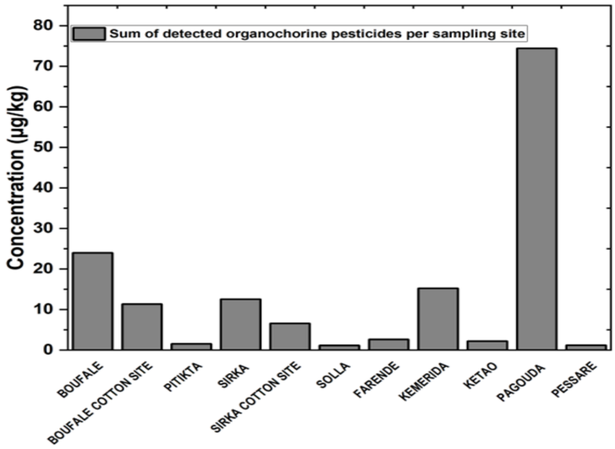
No pesticide residues were found in two (2) of the eleven (11) soil samples: Boufale (cotton site) and Ketao. Pitikta and Farende, each site, have pyrazophos concentration below its LOQ. Sirka is the site with the highest number of compounds detected (5), followed by Pessare (4) and Sirka (cotton site) (4). The compounds with the highest concentrations found were isazophos (LogKow = 3.1) with a concentration of 13.04 µg/kg at Pessare, followed by pyrazophos at Boufale with 12.0 µg/kg and triazophos at Sirka site with 8.70 µg/kg. Low concentrations of chlorpyrifos were found in the samples, ranging from 0.04 to 1.54 µg/kg. In contrast to these results, high concentrations of chlorpyrifos were found in cocoa plantation soil samples in Ghana in 2016, with an average concentration of 30 µg/kg [39], probably due to soil properties, organic matter contents, pesticide uses, and humid climatic conditions. Depending on climate and soil stability, chlorpyrifos' half-life can range from 14 to 380 days, with an average of around 60 - 120 days in the soil [32]. Since 2015, triazophos has been banned in the member states of the Permanent Inter-State Committee for Drought Control in the Sahel (CILSS), of which Togo is a member. Despite this, triazophos residues were detected in the soils of Kemerida and Sirka at concentrations of 0.4 µg/kg and 8.7 µg/kg, respectively, suggesting that this compound was still recently used in significant quantities at some sites even though it is not registered [31]. Soils at Pitikta (<LOQ), Solla (0.1 µg/kg), Pagouda (0.36 µg/kg), and Farende (<LOQ) were the 4 sites with the lowest total pesticide levels of the 11 samples analyzed. In contrast, soils at the Pessare, Boufale, and Sirka sites had the highest values at 16.6 µg/kg, 12 µg/kg, and 10.36 µg/kg, respectively.

Evaluation of organochlorine pesticides in soil samples

The levels of organochlorine pesticides found in the studied soils (**Table 7**) are comparable to those reported in Benin, Mali, Senegal, The Gambia, and Tanzania [13, 36, 40]. This similarity is noteworthy because these countries have crop types, cultivation techniques, and natural conditions similar to Togo [4, 36]. Fifteen pesticides were detected out of the 21 that were searched for in the soil samples (**Figure 6**). Endrin and endrin aldehyde are the most abundant pesticides on the study zone soils. Gamma-BHC, bêta-BHC, and endrin aldehyde have a high detection frequency of 63.63% against endrin and alpha-BHC with 45.45%. Pagouda is the most contaminated site, with a total of 74.42 µg/kg, followed by Boufale and Kemerida, with concentrations of 23.98 µg/kg and 15.24 µg/kg, respectively.

Table 7: Average concentration of organochlorine pesticides in soil samples (µg/kg)

Name	BOUFALE	BOUFALE COTTON SITE	PITIKTA	SIRKA	SIRKA COTTON SITE	SOLLA	FARENDE	KEMERIDA	KETAO	PAGOUDA	PESSARE
Gamma-BHC	< LOQ	0.00	0.00	< LOQ	< LOQ	< LOQ	0.00	1.72	0.00	< LOQ	< LOQ
Bêta-BHC	< LOQ	< LOQ	0.00	< LOQ	< LOQ	0.00	0.00	< LOQ	< LOQ	0.00	< LOQ
Alpha-BHC	1.24	0.2	0.00	0.00	0.64	0.00	0.00	2.94	0.5	0.00	0.00
Heptachlor	0.00	0.00	0.00	< LOQ	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Delta-BHC	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Aldrin	0.00	0.76	0.58	0.00	3.8	0.00	2.5	0.00	1.7	0.00	< LOQ
Heptachlor epoxide	0.00	0.00	0.00	0.00	1.38	0.00	0.00	0.00	0.00	0.00	< LOQ
Cis-Chlordane	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Trans-Chlordane	0.18	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Endosulfan I	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	5.72	0.00
4-4' DDE	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Dieldrin	3.72	0.6	0.00	0.00	0.00	0.00	0.08	2.38	0.00	5.66	0.00
Endrin	< LOQ	2.6	0.00	0.00	0.00	0.00	< LOQ		0.00	19.16	1.1
Chlordecone	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Endosulfan II	0.00	0.00	0.00	0.00	0.00	< LOQ	0.00	0.00	< LOQ	0.00	0.1
4-4' DDD	0.00	< LOQ	< LOQ	< LOQ	0.00	1.18	< LOQ	0.00	0.00	0.00	0.00
4-4' DDT	0.00	0.00	0.00	0.00	0.00	0.00	0.08	0.00	0.00	0.00	0.00
Endrin aldehyde	18.28	6.26	0.00	12.56	< LOQ	0.00	< LOQ	7.24	0.00	43.88	0.00
Endrin Ketone	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Endosulfan sulfate	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Methoxychlor	0.56	0.96	0.96	0.00	< LOQ	0.00	0.00	0.96	0.00	0.00	0.00
ΣDDT and metabolites	0.000	0.000	0.000	0.000	0.000	1.180	0.080	0.000	0.000	0.000	0.000
ΣHCH	1.240	0.200	0.000	0.000	0.640	0.000	0.000	4.660	0.500	0.000	0.000
Aldrin+dieldrin	3.720	1.360	0.580	0.000	3.800	0.000	2.580	2.380	1.700	5.660	0.000
Alpha-BHC/Gamma-BHC	1550000	NA	NA	0.000	800000	0.000	NA	1.709	NA	0.000	0.000
Endosulfan I/Endosulfan II	NA	NA	NA	NA	NA	0.000	NA	NA	0.000	NA	0.000



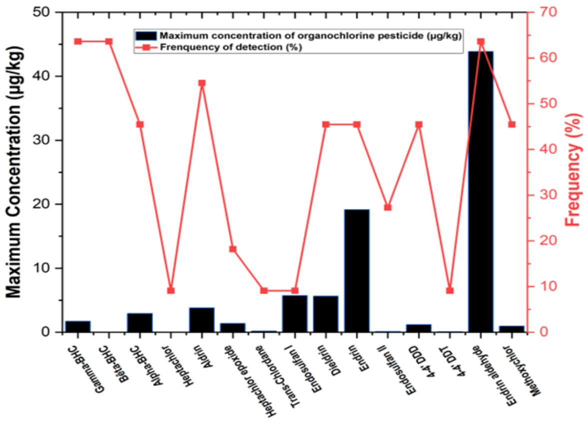


Figure 6: Total concentration (µg/kg) per site (above) and maximum concentration and respective frequency of detection (below) of organochlorine pesticide in soil samples.

The presence of DDE or DDD in the studied soils indicates the metabolic activity of the existing microflora. DDT is metabolized to DDE by microorganisms under aerobic conditions, while it is transformed to DDD under anaerobic conditions [41]. The average concentrations of DDT, DDE, and DDD in the market garden soils of the Binah prefecture vary significantly. DDE was not detected. The parent compound DDT was detected only at one site in Farende at an average concentration of 0.08 µg/kg. The concentrations of DDD detected at five sites range from values below the limit of quantification at Boufale (cotton site), Pitikta, Sirka, and Farende to 1.18 µg/kg at the Solla site. These values are lower than the 4.8 µg/g detected in 2007 in the soils of the market garden site in the Ouémé Valley in Benin [42]. Other studies conducted during the same period indicate average residual concentrations of DDT and its metabolites ranging from 0.1 to 9.7 µg/kg in agricultural soils in Tanzania in 2004 [43] and ranging from 1.79 to 7.93 µg/kg in Senegal in 2003 [44]. Previous studies conducted on agricultural soils of market gardening sites in Togo in 2017 in the Plateaux region indicate an absence of DDT and degradation by-products [14]. The variations in residue concentrations can be attributed to a cessation of the use and sale of products banned by the Stockholm Convention, yet these substances persist in the environment [16].

Aldrin and dieldrin concentrations were generally opposite at the agricultural sites sampled. Aldrin is more frequent (54%) than dieldrin (45%), but has a lower maximum concentration, with values ranging from < LOQ to 2.5 µg/kg vs. 0 to 5.66 µg/kg. The work conducted in the Plateaux region of Togo in 2017 also shows that only Aldrin was detected at average concentrations ranging from 0.04 to 0.93 µg/kg.[14]. The residual values of aldrin detected in the studied soils are not comparable to the 0.49 µg/g observed in the soils of the market gardening site in the Ouémé Valley in Benin [40] with agricultural production contamination. They are relatively high compared to the contamination levels of sites in Senegal at 1.16 µg/kg in the agricultural soils of the Niaye area [44]. Dieldrin was detected at a concentration of 2.15 µg/g in the soils of the vegetable growing site in the Ouémé Valley in Benin [42] and at average concentrations of 0.2 – 0.8 µg/kg in the agricultural soils of the border areas between Senegal and The Gambia [45]. In the soils, due to the rapid conversion of aldrin to dieldrin, there will be higher concentrations of dieldrin than aldrin. Indeed, dieldrin indicates either a direct use of dieldrin or a degradation of aldrin [46]. However, the high logKow value of aldrin at 6.5 compared to that of dieldrin at 3.7 (**Table 3**) may explain the higher detection frequency of aldrin in our soil samples.

Heptachlor epoxide was detected at Pessare and Sirka (cotton site), with respective levels < LOQ at 1.38 µg/kg, and heptachlor, once at a concentration < LOQ at Sirka. This again confirms the use of heptachlor in Togo agriculture and its transformation into heptachlor epoxide by soil biological systems. Aldrin and dieldrin concentrations were generally opposite at the agricultural sites sampled. Aldrin is more frequent (54%) than dieldrin (45%), but has a lower maximum concentration, with values ranging from < LOQ to 2.5 µg/kg vs. 0 to 3.72 µg/kg [47]. The residual values of endrin ranged from below LOQ to 19.16 µg/kg (Boufale, Boufale Cotton Site, Farende, Pagouda, Pessare). These values are higher than those reported in Senegal and the Gambia 0.2 - 1.0 µg/kg [45] and those reported in Togo in the soils of cash crops (cotton, coffee, and cocoa) in the Plateaux Region 1.13 µg/kg to 6.89 µg/kg [15, 47]. The concentrations of endosulfan I (0 – 5.72 µg/kg) and endosulfan II (< LOQ - 0.1 µg/kg) are quantified in the studied soils with detection frequencies of 9.09% and 27.27%, respectively.

Analyses show variability in the concentration levels of BHC and its derivatives, which are below the guideline value. The residual values of alpha BHC range from 0.1 to 1.47 µg/kg, except in Farende, where it was undetected. In contrast to its isomers, Alpha BHC is absent at the sites of Pitikta, Sirka, Pagouda, and Pessare. Methoxychlor intended to replace DDT but has since been banned due to its acute toxicity, bioaccumulation, and endocrine-disrupting activity, was detected across three sites (Boufale, Pitikta, and Kemerida) with residual values between 0.56 and 0.96 µg/kg. Trans-chlordane was detected at Sirka and Boufale, with concentration levels of 0.09 µg/kg at Boufale and below the limit of quantification at Sirka.

4. Conclusion

During this study, chemical pollution by pesticides in the water resources of nine sites used for market gardening sites in the Binah prefecture during the dry season appeared relatively low. No organophosphorus pesticides were detected at levels above the surface water quality limit. On the other hand, conditions conducive to a change in pesticide use practices are emerging. Indeed, the fragile border between Togo and neighbour countries favors the circulation and trading of uncontrolled formulations of pesticides. This results in a more diversified use of products with inadequate doses posing challenges in risk communication. Soil sample measurements from the same market garden sites were also of concern. 06 organophosphate pesticides were detected in soil and water samples despite being banned. Organochlorine pesticides such as endosulfan sulfate, endrin ketone, and chlordecone were not found in any samples. Methoxychlor, heptachlor epoxide, cis-chlordane, trans-chlordane, and delta-BHC were found either in water samples or in soil samples; the remaining contaminants were detected simultaneously in both matrices analyzed. The contamination profile of the samples, particularly in soils, is alarming sometimes, showing historical pollution and the persistence of these compounds in the environment. Therefore, this study recommends establishing monitoring, action programs, and environmental risk assessment. One of the remaining questions is the analysis of agricultural products. The issue of pesticide use deserves the attention of decision-makers. More strict measures must be taken to rationalize the use of plant protection products and thus limit environmental contamination and the risks of exposure. It would be valuable to further follow up on this study by examining groundwater contamination from pesticide residues and the impact on staple vegetables grown in the Binah region. This would provide a comprehensive assessment of resource contamination by pesticides and evaluate the overall health risks involved.

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