Journal of Agricultural Chemistry and Environment, 2026, 15(1), 63-73
https://www.scirp.org/journal/jacen
ISSN Online: 2325-744X
ISSN Print: 2325-7458

Quantitative Study of Some Pesticides in the Sediments of the Alibori River

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How to cite this paper: Kouazounde, B.J., Chitou, N.E. and Chouti, K.W. (2026) Quantitative Study of Some Pesticides in the Sediments of the Alibori River. *Journal of Agricultural Chemistry and Environment*, **15**, 63-73.
https://doi.org/10.4236/jacen.2026.151005

Received: December 14, 2025
Accepted: February 10, 2026
Published: February 13, 2026

Abstract

Many pollutants are likely to compromise the quality of waterways. The Alibori River, located in the cotton basin of Benin, is subject to being compromised by organic pollutants from pesticides used in cotton production. To assess the contamination of the sediments of the Alibori River by pesticides, they were sampled, packaged in aluminum foil, and stored in the laboratory. The presence and content of Cypermethrin, Lambda-cyhalothrin, and Acetamiprid were determined on 5 g of sediment. Extraction was performed using 1% of acidified acetonitrile after purification before chromatographic analysis using an HPLC chromatograph. According to the results, only cypermethrin was detected in the river sediments with concentrations between 2708 $\mu g \cdot kg^{-1}$ and 2732 $\mu g \cdot kg^{-1}$ during the dry season. This appearance of cypermethrin could be explained by its strong adsorption to soil particles and its low mobility. Gas chromatography (GC) is more suited to determining lambdacyhalothrin, which could explain its absence. It would therefore be appropriate to continue analyzing these molecules in surface water and groundwater by adopting several analysis methods.

Keywords

Cypermethrin, Lambda-Cyhalothrin, Acetamiprid, Alibori River

1. Introduction

The Alibori River, one of the tributaries of the Niger River in the Cotton Basin of Benin, is subject to agricultural pressure which has continued to increase for years. The water from this river is used for various daily uses, including washing, watering, and drinking due to the shortage of water supply in the riverside villages.

A recent study conducted by [1] [2] on the physicochemical characterization of the waters and sediments of the Alibori river showed that they are polluted by metals, phosphates, and nitrites from the intensive use of fertilizers and pesticides in the agricultural sector.

Pesticides are chemical substances intended to destroy or repel organisms unwanted (insects and grass). However, they are harmful to human health and the environment. More than 97% of pesticides used for agricultural purposes are found in environmental compartments, notably water and soil, with consequences on humans (toxicological effects) and on other living organisms (ecotoxicological effects) [3].

Currently, the pesticides used in Benin are mainly herbicides to combat weeds and insecticides to combat pests [4]. The active ingredients likely to be found in its pesticides are lambda-cyhalothrin, chlorpyrifos, indocarb, acetamiprid, spinetoram, cypermethrin, bifenthrin, dimethoate, profenofos, emamectin benzoate, glyphosate, atrazine, etc. [5] [6]. They can have consequences on humans and living organisms other than humans. The present objective is to evaluate qualitatively and quantitatively in river sediments of Alibori some active ingredients of insecticides used in the cotton basin of Benin.

2. Materials and Methods

2.1. Geographic Location

The chosen study area is the Alibori River located in the Sudanian zone between 10˚30' and 12˚ North latitude and 1˚32' and 3˚50' East longitude in the Commune of Banikoara located in the north west of the Alibori department, Republic of Benin. It originates at approximately 410 m altitude, in the granite massif of Kita on the flank of the Atacora range in the commune of Pehunco with a length of 427 km [1] [2].

2.2. Collection of Data on Pesticides Used

Data on the different phytosanitary products used, the frequency of use and the quantity used per hectare were collected from a survey of 200 cotton producers who consumed the most phytosanitary products and a few resource persons in villages located on the banks of the river "Banikoara, Founougo, Goumori, Gomparou, Kokey, Kokiborou, Ounet, Sompérékou" in the commune. They were supplemented by information collected through direct and participatory observation of producers' practices during the phases of preparation of the mixture and spraying of cotton in the field.

2.3. Qualitative Pesticide Analyzes

The analysis of the various surveys carried out made it possible to identify the different pesticides most used in the commune of Banikoara by farmers to protect cotton, corn, and other crops (**Table 1**). According to **Table 1**, these pesticides are made up of herbicides and insecticides. The active ingredients likely to be found in herbicides are Atrazine, glyphosate, diuron, butachlor, nicosulfuron, prometryn, fluometuron, haloxyfop-r-methyl. Those of insecticides are cypermethrin, abamectin, emamectin benzoate, emamectin, acetamiprid, endosulfan, c-ethyl.

Table 1. The different types of pesticides used in cotton cultivation in northern Benin [5].

Herbicides		**Insecticides**	
Usual name	**Active ingredient**	**Usual name**	**Active ingredient**
Atraherb	Atrazine	Vizier 92 CE	Cypermethrin 72g/L Abamectin
Kalach III H	Glyphosate 360 g/L	Thalis	Emamectin benzoate Emamectin Acetamiprid
Herbicide action	Diuron (800 g/l)	Lambda	Lambda-cyhalothrin
Butaforce	Butachlor	Sharp shooter	Chlorpyrifor 40% Cypermethrin 4%
Nicosulfuron	Nicosulfuron	Cotofan	Endosulfan
Callifor G III	- Glyphosate 60 g/L - Fluometuron 250 g/L - Prometryn 250 g/L	Best cypermethrin	Cypermethrin
Nicomaize	- Nicosulfuron 40 g/L - Atrazine	EMA star 112 EC II	- Emamectin benzoate 20 g/L - Acetamiprid 64 g/L
Cotochem560g III	- Prometryn 280 g/L - Fluometuron 280 g/L	Dursuban	- Cyfluthrin (T) - Chlorpyrifos-Ethyl (T)
Killer 480SL	Glyphosate 480 g/L		
Atrazila 80Wi	Atrazine		
Atrazine 50% SC	Atrazine		
Sunphosate 360SL	Glyphosate 360 g/L		
Glyphosate 41%SL	Glyphosate		
Glyphader 480 III H	Glyphosate 480 g/L		
Kabaherb 720 g/L	2,4D amine salt		
Last more	Diuron		
Herbextra 720SL II	Glyphosate(T)		
Malick 180 EC	Haloxyfop-r- methyl 108 g/L		

Table 2 shows that more than 38% of herbicides contain glyphosate, 22% contain atrazine, 11% contain diuron, and 11% nicosulfuron. Glyphosate and diuron are weakly mobile and persistent, atrazine and nicosulfuron are mobile but weakly persistent.

More than 38% of insecticides contain cypermethrin, 25% acetamiprid, 13% lambda-cyhalothrin and 13% abamectrine. Lambdacyhalothrin and cypermethrin are immobile and highly persistent.

Table 2. Characteristics of some active ingredients of pesticides used in cotton cultivation in northern Benin.

Name of material	The number of herbicides that contain it	Koc (INERIS (Edite, 25/09/19))	Classification	LogKoc INERIS (Edite, 25/09/19))	DT50 INERIS (Edite, 25/09/19))	Persistence
Herbicides						
Glyphosate	38%	1435	Poorly mobile	3.15	69-stable	Big
Atrazine	22%	100	Mobile	2	44d	Average
Diuron	11%	1990	Poorly mobile	3.30	220d	Big
Nicosulfuron	11%	347.5	Fairly mobile	2.54	18.7d	small
Insecticide						
Cypermethrin	38%	202,900	Motionless	5.31	750 d	Big
Acetamiprid	25%	106.5	Fairly mobile	2.03	Stable	Big
Endosulfan	13%	12,400	TF mobile	4.10	120 days	Big
Abamectin	13%	1400	Poorly mobile	3.15	89 d	Big
Lambda-Cyhalothrin	13%	157,000	Motionless	5.20	Stable	Big

2.4. Determination of Mobility

The mobility of the pesticide was determined using the log Koc formula with Koc and the Adsorption coefficient (organic carbon) (Table 3).

Table 3. Classification according to mobility [7].

Log Koc	Classification
<1	Very mobile
1 - 2	Mobile
2 - 3	Fairly mobile
3 - 4	Poorly mobile
4 - 5	Very weakly mobile
>5	Motionless

2.5. Sampling and Pesticide Analysis

2.5.1. Choice of Molecules

Insecticides are designed to kill insects, so they can also affect the human body, which justifies the choice of insecticides. The choice of molecules is based on persistence and frequency of use. Research into the characteristics of the molecules (lambda-cyhalothrin, acetamiprid, cypermethrin) reveals that these molecules are highly persistent. The surveys recorded in the table... showed that 38% of the insecticides used contain acetamiprid, 25% contain cypermethrin, and 13% contain lambda-cyhalothrin. Given the proportion of insecticides containing these molecules, their persistence, and their toxicity, we decided to quantify these three molecules in river sediments. In addition, the results obtained by [1] [2] which showed a high concentration of metallic trace elements in the sediments and a high concentration of nitrite and nitrate in the waters at the level of these sites. The choice was based on the position of the crop fields with this water course, the different anthropogenic activities, and the uses of the river water.

2.5.2. Sampling and Conservation Method

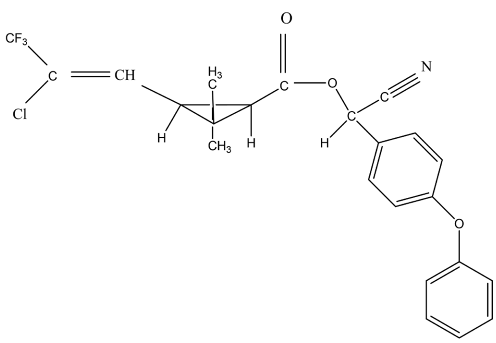
Six sediment samples were collected, three upstream and three downstream at different sites along the river using a stainless steel Schipeck grab. At each site, sediment was collected at three points across the width of the river to obtain one sample per site. Each triplet of sediment along the river was mixed to produce one representative sample upstream and downstream for each season.

The sediments were sampled in aluminum foils previously cleaned with pentane and 95% ethanol. The collected sediments were placed in coolers where they were kept cold using ice blocks. In the laboratory, the sediments were stored in a freezer at −18˚C until the pesticide analysis step.

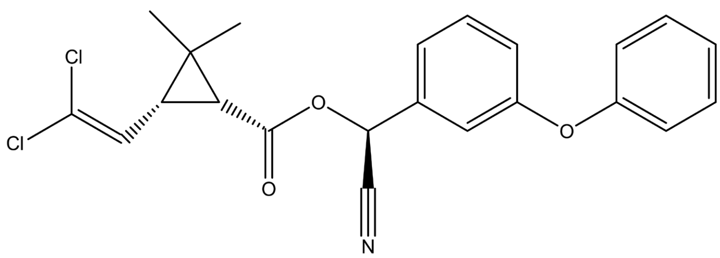
2.5.3. Sediment Analysis

For the rest of the work, only the most used and most persistent insecticides were determined. To determine the presence and content of these insecticides, (Lambda-cyhalothrin, acetamiprid, cypermethrin of respective Formulas 1, 2, and 3) the internal laboratory method was adapted and consisted of weighing 5 g of sediment was taken in a polyethylene bottle of 50 mL to which 10 mL of distilled water was added and shake for minutes. After a break of 30 min, 10 mL of acetonitrile acidified to 1% with acetic acid was added. The mixture was stirred with a shaker. After stirring, 4 mg of magnesium sulfate ($MgSO_4$) and 1 mg of sodium acetate (CH_3COONa) were added to the mixture obtained.

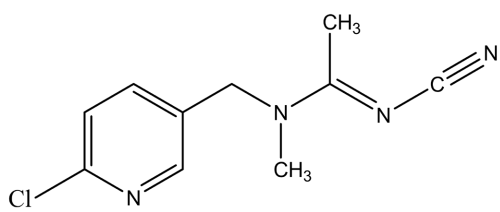
The mixture thus obtained was centrifuged at 5000 rpm using a centrifuge for 5 min to recover the supernatant. One milliliter (1 mL) of the supernatant was taken into the Epindof tube containing 150 mg of magnesium sulfate ($MgSO_4$), 25 mg of PSA (Primary secondary Amine), and 25 mg of C18. The mixture was stirred and the supernatant was collected for analysis of the desired compound by HPLC using a C18 reverse phase column, an acetonitrile/water mobile phase in an elution gradient with a flow rate of 1 ml and a UV detector at 230 nm.



[1]-Lambda-cyhalothrin



[2]-cypermethrin



[3]-Acetamiprid

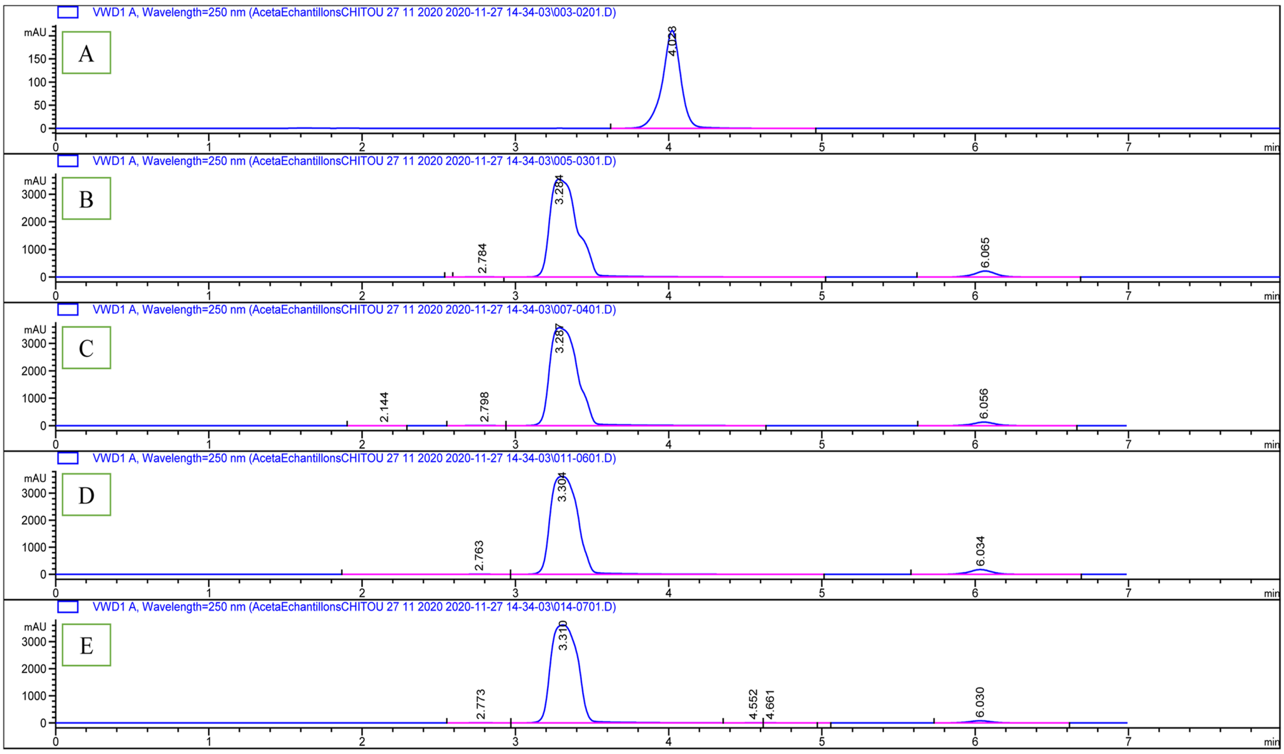
DOI: 10.4236/jacen.2026.151005

3. Results

Quantitative Analyzes

- **Acetamiprid**

Figure 1 shows the chromatogram of acetamiprid, part A shows the peak of acetamiprid in the standard with a retention time of 4 min. The other parts B, C, D, and E present the sediment results upstream and downstream of the river during the rainy season (RS) and dry season (DS). These four peaks come out before 4 min and other peaks after 4 min. This confirms the absence of acetamiprid in the sediments of the Alibori River during both seasons.

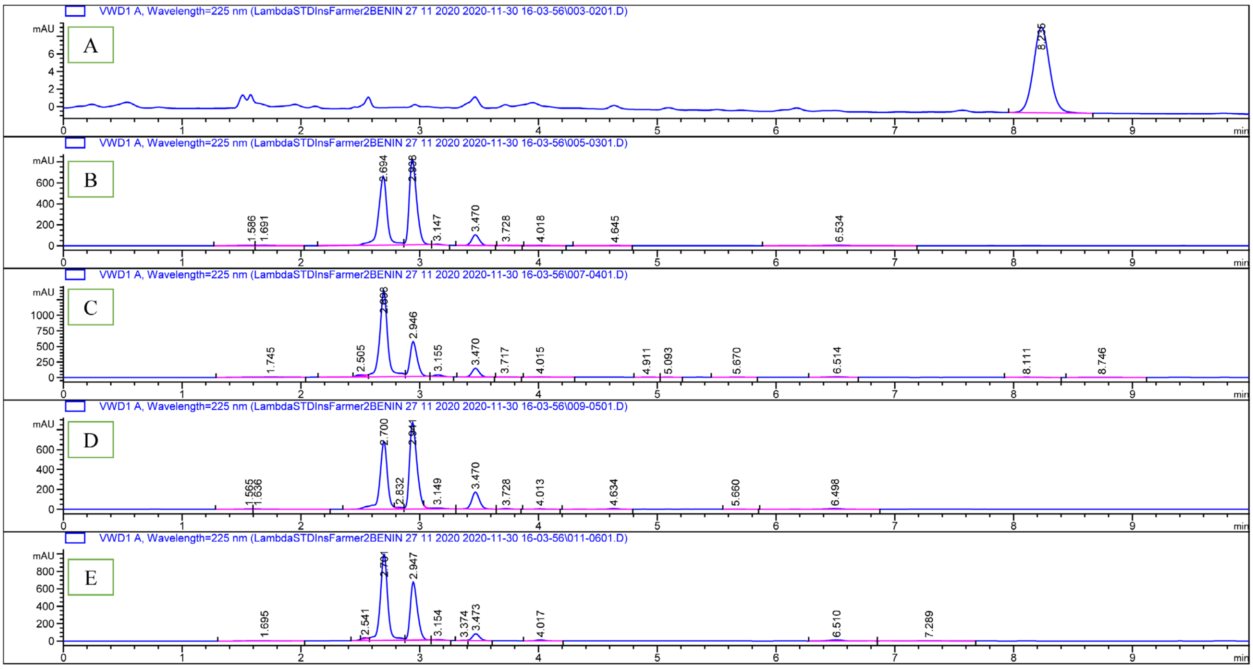


(A): standard chromatogram; (B): Chromatogram obtained from the analysis of the sediment upstream of the river during the dry season; (C): Chromatogram obtained from the analysis of the sediment downstream of the river during the dry season; (D): Chromatogram obtained from the analysis of the sediment upstream of the river during the rainy season; (E): Chromatogram obtained from the analysis of the sediment downstream of the river during the rainy season.

Figure 1. Acetamiprid chromatography.

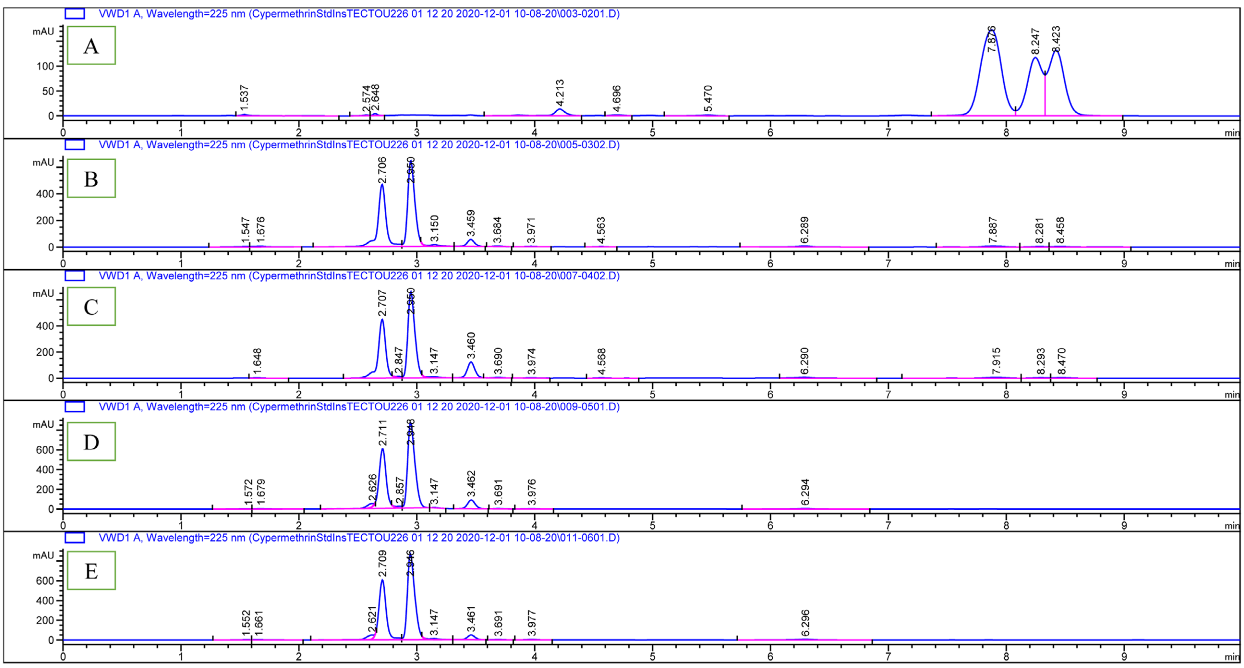
- **Lambda-cyhalothrin**

Figure 2 shows the chromatogram of lambda-cyhalothrin. Chromatogram A in the figure shows the Lambda-cyhalothrin peak in the standard at 8.16 min. Chromatograms B, C, D, and E present the results of sediments upstream and downstream of the river during the rainy season (RS) and dry season (DS). These four chromatograms show peaks between 2.5 min and 3.7 min. The peaks obtained in the sediments of the Alibori River do not correspond to the lambda-cyhalothrin peak.



(A): standard chromatogram; (B): Chromatogram obtained from the analysis of the sediment upstream of the river during the dry season; (C): Chromatogram obtained from the analysis of the sediment downstream of the river during the dry season; (D): Chromatogram obtained from the analysis of the sediment upstream of the river during the rainy season; (E): Chromatogram obtained from the analysis of the sediment downstream of the river during the rainy season.

Figure 2. Lambda-cyhalothrin chromatography.



(A): standard chromatogram; (B): Chromatogram obtained from the analysis of the sediment upstream of the river during the dry season; (C): Chromatogram obtained from the analysis of the sediment downstream of the river during the dry season; (D): Chromatogram obtained from the analysis of the sediment upstream of the river during the rainy season; (E): Chromatogram obtained from the analysis of the sediment downstream of the river during the rainy season.

Figure 3. Cypermethrin chromatography.

- **Cypermethrin**

Chromatogram A in **Figure 3** shows the cypermethrin peaks in the standard at 7.87 min; 8.24 min and 8.42 min. Chromatograms B, C, D, and E present the results of sediments upstream and downstream of the river during the rainy season (RS) and dry season (DS). Chromatograms B and C show a peak at the same level as the standard with triplets. This shows the presence of cypermethrin in the sediments of the Alibori River during the dry season from upstream to downstream. The D and E chromatogram shows no sign at this date, which explains the absence of cypermethrin during the rainy season in the sediments.

Table 4 shows the content of cypermethrin residues in the river sediments during the dry season. These contents vary from 2708 μg·kg^{-1} to 2732 μg·kg^{-1} from upstream to downstream of the river.

Table 4. The content of cypermethrin residues.

Site	Measure	Residue content of cypermethrin in μg/kg
Upstream	1	2632
	2	2784
	M	2708
	Ecartype	107,480
Downstream	1	2724
	2	2740
	M	2732
	Ecartype	11,314

4. Discussion

The active ingredients of phytosanitary products can be found in different compartments of the environment after use, depending on their properties and the physicochemical conditions of the environment.

In the sediments of the Alibori River, no residue of the desired pesticide (Acetamiprid, Lambda-cyhalothrin, and cypermethrin) was detected during the rainy season. On the other hand, during the dry season, only cypermethrin was detected in the river sediments with concentrations between 2708 μg·kg^{-1} and 2732 μg·kg^{-1}. This appearance of cypermethrin could be explained by its strong adsorption to soil particles and its low mobility [8]. In addition, the presence of cypermethrin in the sediment of the Alibori River in the dry season contrary to the results of [8] in the Agbado River could be due to the frequency of use of these products (pesticide) depending on cotton production (Alibori (41%), Atacora (18%)) and its intensive use in recent years. The absence of cypermethrin in the sediments during the rainy period could be explained by its presence in the water. In addition, this result corroborates with that of [9] which stipulates the absence of Pyrethroids in the sediments of the Agbado River in the rainy season, but their presence in the same season. At neutral or acidic pH, cypermethrin is stable [10]. The pH of the river sediments is between 4.59 and 6.78; this explains the long lifespan of cypermethrin in sediments and its low degradation. The other peaks observed on the cypermethrin chromatogram could be explained by the presence of the degradation products of cypermethrin (a carbamoyl derivative of cypermethrin, 3-(2,2-dichloro vinyl)-2,2-dimethyl cyclopropane carboxylic acid (DCVA) and 3-phenoxybenzoic acid). Cypermethrin and its metabolites are very toxic to aquatic species. In humans, it can cause a skin allergy [8].

The absence of acetamiprid in the sediments of the Alibori River during both seasons could be explained by its low bioaccumulation potential and its short lifespan.

Indeed, according to [11], acetamiprid and its families can be introduced into surface waters by runoff after rainy events. However, it is very soluble in water ($4.25*10^5$) and has a low octanol-water partition coefficient (Kow) (6.27) according to [12]. This confirms the assertion of [1] [2], which states that the present ETM in river sediments would be due to the degradation of pesticides. When acetamiprid degrades, it turns into associated metabolites which are Methylamine, N2-cyano-N1-methylacetamidine N-cyanoacetamidine, N-methyl (6-chloro-3-pyridyl) methylamine. The appearance of other peaks close to the acetamiprid peak on the chromatogram could be that of these metabolites.

Lambda-cyhalothrin is a synthetic pyrethroid just like cypermethrin. Lambda-cyhalothrin is poorly soluble in water [13], it is immobile, this is due to its strong adsorption on sediments. The results obtained highlight its absence in the sediments of the Alibori River during both seasons despite the frequency of its use in the control of cotton pests. Indeed, the chromatograms obtained present peaks at retention times lower than that of lambdacyhalothrin; this explains its absence. Previous studies have shown that lambda-cyhalothrin dissipates very quickly in water due to its adsorption on aquatic organisms and particles [14] [15]. The peaks which appear on the chromatogram could be that of its metabolites such as (1RS)-cis-3-(ZE-2-chloro-3,3,3-trifluoroprop-1-enyl) 2,2-dimethylcyclopropanecarboxylic acid, (1RS)-cis-3-(Z-2-chloro3,3,3,-trifluoroprop-1-enyl)2,2-dimethylcyclopropanecarboxylate of (RS)-"cyano-3-(4-hydroxyphenoxy)benzyl". These metabolites are less toxic than the molecule itself [9]. Gas chromatography coupled with an electron sensor detector has been used by several authors for the determination of Pyrethroids in various water, sediment, meat, fish, etc. matrices, because of its advantages [13] [16]-[20]. Furthermore, [21] showed in his 2017 work that CG-MS is only suitable for synthetic pyrethroids. The pyrethroids contained in insecticides are synthetic. The absence of lambda-cyhalothrin in the sediments of the Alibori River during the two seasons contrary to what should be according to its characteristics could also be explained by the analysis method used (HPLC).

5. Conclusion

The objective of this part of the work is to identify pesticides in river waters and sediments. The results show the presence of cypermethrin and the absence of acetamiprid and lambda-cyhalothrin in the river sediments. The absence of these molecules in sediments could be explained by their presence in water or by their degradation. This observation confirms the assertions of our previous work which linked the presence of the ETM, nitrogen, and phosphate derivatives to degradation of pesticides. The presence of cypermethrin in these sediments allows us to conclude that the use of pesticides in cotton production has negative impacts on water resources, mainly surface water. To achieve the set objective, it would therefore be appropriate to continue the analysis of these molecules in the water and sediments of the river by adopting several analysis methods (coupled HPLC, coupled GC, etc.).

Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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