

Application of the QuEChERS Method for the Analysis of Contamination by Pesticide Residues in the Sediments of Three Moroccan Lagoons

Rachida Fegrouche^{1*}, Hicham Dahak¹, Taha El Kamli², Mohammed Oussekeur³, Saida Zaza³, Abdelkrim Ben Salem⁴, Badreddine Benyacoub⁵

¹ Laboratory of Biodiversity, Ecology, and Genome (BioEcoGen), Faculty of Sciences, B.P. 1014, Plant and Microbial Biotechnologies, Biodiversity and Environment (BioBio) Research Center, Mohammed V University in Rabat, Morocco

² Hassan II Agronomic and Veterinary Institute in Rabat, Doping Control Laboratory, Morocco

³ The National Office of Electricity and Potable Water. Water sector, Morocco

⁴ Botany and Valorization of Plant and Fungal Resources Team (BOVAREF), Faculty of Sciences, B.P. 1014, Plant and Microbial Biotechnologies, Biodiversity and Environment (BioBio) Research Center, Mohammed V University in Rabat, Morocco

⁵ National Institute of Statistics and Applied Economics, Rabat, Morocco

* Corresponding author's e-mail: r.fegrouche@um5r.ac.ma

ABSTRACT

Agricultural, industrial, and domestic activities are major contributors to the contamination of natural environments. The aim of this study is to assess the level of sediment contamination by organic pollutants in three Moroccan lagoons: Moulay Bouselham, Oualidia, and Khnifiss. samples were analyzed using liquid chromatography (LC) coupled with mass spectrometry (MS) in Multiple Reaction Monitoring (MRM) mode to detect organophosphorus, carbamate, urea and its derivatives, and other chemical groups. Gas chromatography (GC) coupled with mass spectrometry (MS) was also used to analyze organochlorines. The samples were subjected to dispersive solid-phase extraction (dSPE) using QuEChERS before analysis. Fifteen active substances were detected, including organochlorines, organophosphates, carbamates, ureas, pyrethroids, and others. Three active substances, known for their high toxicity in aquatic environments (carbendazim, malathion, and chlorpyrifos), were identified. The heptachlor molecule (organochlorine family), although banned in Morocco, was still detected in the sediments of the lagoons of Oualidia and Khenifiss. Given the potential harm that these pesticides can cause to living organisms, it is crucial to introduce new crop protection techniques to address this issue.

Keywords: Sediments, organic contamination, pesticides, lagoons Morocco, QuEChERS extraction, LC-MS/MS, GC-MS/MS.

INTRODUCTION

Wetlands are important ecosystems that provide a range of ecological and socio-economic benefits for human well-being (Ibara et al., 2015). They are used for various activities, such as agriculture, fishing, fish farming, hunting, and tourism. The Ramsar Convention of 1971 recognized the importance of wetlands by defining conservation objectives and creating a list of the main

sites worldwide (Matthews, 1993). Wetlands are highly diverse in terms of size, biodiversity, and number of endemic species (Williams et al., 2004; Biggs et al., 2005; Oertli et al., 2008). Morocco has the richest wetlands in the southern Mediterranean, with a marine coastline of approximately 3500 km and two large mountain ranges that have generated a very dense hydrographic network (Elkhiati et al., 2013). The country has over 200.000 hectares of lake and lagoon systems, as

well as thousands of kilometers of river network and coastal areas. These environments have high biodiversity and landscape value, which is important for socio-economic and tourist development. However, these environments are often subject to intense socio-economic activities (urbanization, agriculture, wastewater discharge, coastal works, etc.) that lead to the loss and transformation of wetland habitats into anthropized habitats, modification of the hydrological functioning of the site, and degradation of water and sediment quality (pollution by fertilizers and pesticides), which hinders the growth and development of aquatic organisms (El Hammoumi and Himmi, 2010). The intensification of agriculture has led to a greater consumption of pesticides (Racke et al., 1997), which can lead to a wide dispersion of these pesticides in the environment (Mansingh et al., 2000; Azevedo et al., 2004). Pesticides are adsorbed onto organic matter incorporated into sediments (Nhan et al., 2001; Sun et al., 2010), and their persistence depends on their physicochemical properties. Pesticides are generally classified into insecticides, fungicides, herbicides, rodenticides, and nematocides based on their target species (Sharma et al., 2019), but can also be divided based on their molecular structure, including organophosphates, organochlorines, carbamates, and pyrethroids (Kamel and Hoppin, 2004).

Numerous studies worldwide have investigated the presence and distribution of pesticides in marine sediments, as reported by some researchers (Ahmad et al., 1996; Racke et al., 1997; Baumard et al., 1998; Mansingh et al., 2000; Nhan et al., 2001; Pandit et al., 2001; Djomo et al., 2004; Guzzella et al., 2005; Rajendran et al., 2005; Wurl and Obbard, 2005; Sun et al., 2010). In Morocco, two studies have examined the prevalence of organochlorine pesticides in sediments along the Atlantic coast, as reported by two authors Mehdaoui and Benbakhta (Mehdaoui et al., 2001; Benbakhta et al., 2014). The objective of this study is to evaluate the extent of pesticide contamination in the sediments of three stations: Moulay Bousselham lagoon (S1), Oualidia lagoon (S2), and Khnifiss lagoon (S3). Figure 1 illustrates the geographical locations of the stations (S1, S2, and S3) and corresponding sampling sites.

MATERIALS AND METHODS

Study area

This study was conducted in three separate lagoons in Morocco: Moulay Bousselham lagoon (S1), Oualidia lagoon (S2), and Khnifiss lagoon (S3) (Figure 1).

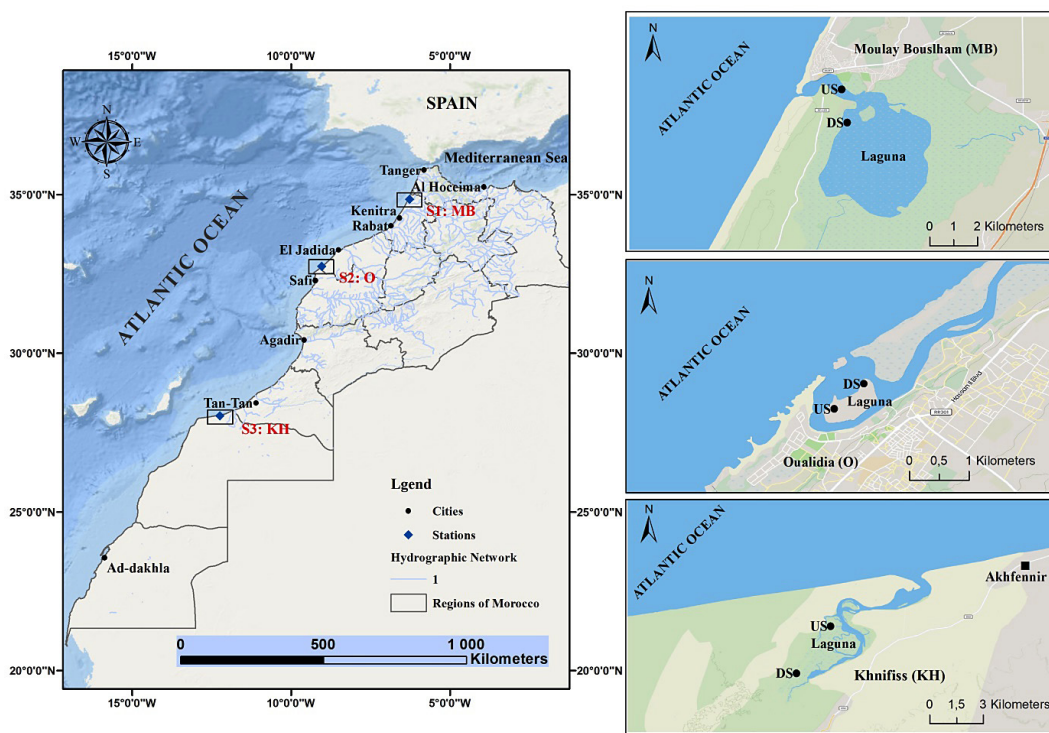


Figure 1. Map showing geographical location of stations (S1, S2 and S3) and sampling sites

S1: Moulay Bouselham lagoon (34°51' N, 06°16' W) is located on the Moroccan Atlantic coast, commonly known as Merja Zerga. It has an elliptical shape stretching from north to south, covering an area of 35 km². The lagoon is divided into two Merjas of unequal dimensions, with Merja Khala to the north being very shallow and triangular in shape, and Merja Zerga to the south, having an area of 27 km² and a maximum depth ranging from 1 to 5.5 meters. The lagoon system is mainly drained by the Drader River to the east and the Nador Canal to the south. Since 1980, this lagoon has been designated as a Ramsar site, a convention for internationally important wetlands. The surrounding area is mainly used for agriculture and sheep and cattle farming, with strawberry production being the most widespread crop. Artisanal fishing and shellfish harvesting are also common activities in this lagoon.

S2: Oualidia lagoon (32°40'4" N – 32°47'07" N and 8°52'30" W – 9°02'50" W) is situated along the Atlantic Ocean and extends for 7 km in length and approximately 0.5 km in width, covering an area of 3 km². It has a general tetrad shape and two unequal passes downstream, a permanent main pass 150 meters wide and averaging about 3 meters deep, and a secondary pass about 50 meters wide and averaging 1 meter deep, active in periods of high tides, providing communication between the lagoon and the ocean (Hilmi et al., 2017). Oyster farming is a recognized aquaculture activity in this lagoon. During the summer, Oualidia lagoon is a site of intense tourist activity. Untreated agricultural and domestic wastewater discharges can lead to increased contamination.

S3: Khnifiss lagoon, commonly known as Khnifiss or Naïla Bay, is the largest Moroccan Atlantic lagoon (20,000 ha), situated in southwestern Morocco (27.80–28.05° N, 12.21–12.39° W), 120 km southwest of Tantan and 70 km northeast of Tarfaya, and is the only Saharan lagoon in North Africa (Idardare et al., 2013). It is an internationally important wetland listed on the Ramsar Convention since 1980, and is a Biological and Ecological Interest Site (SIBE) classified as a national park in 2006. This lagoon is home to 30 species of macroalgae and a diversity of marine invertebrates. It is an important feeding, spawning, and nursery site for fish and a stopover site for a large number of

migratory birds from the Western Palearctic, sometimes hosting more than 20,000 waterbirds during the wintering period. The main recognized activities in this site are tourism, fishing, and aquaculture.

Sediment sampling

In 2020, a total of 18 sediment samples were collected from the three selected stations along the Atlantic coast (Figure 1). At each station, two sites (upstream (US) and downstream (DS)) were chosen, and three replicates were taken for each sampling site. Sediment samples were collected at low tide using a plastic shovel and homogenized before being placed in polyethylene stomacher bags (Casado-Martinez et al., 2019). The samples were transported to the laboratory, where they were frozen at -20°C and subsequently lyophilized in an oven at 40°C to remove water content. After lyophilization, the samples were sieved ($\phi < 0.63 \mu\text{m}$) and stored in the dark at -20°C until analysis (Afnor, 2009; Casado-Martinez et al., 2019).

Extraction

Pesticide standards with a purity of 96.7% were obtained from Thermofisher Scientific (USA). Stock and working solutions were prepared using HPLC grade acetonitrile obtained from VWR (USA). The SPE extraction and purification kits included PSA, obtained from VWR (USA). The QuEChERS method (Bronzi et al., 2011) was slightly modified to extract the soil samples. Three grams of homogenized and dried sediment sample were placed in a 50 ml tube and mixed with 7 ml of water and 10 ml of acetonitrile. The mixture was vortexed for 5 min, and then an extraction salt consisting of 4g of MgSO₄, 1g of NaCl, 0.5g of sodium citrate dibasic sesquihydrate, and 1g of sodium citrate tribasic dehydrate was added. The mixture was shaken for 1 min and centrifuged for 5 min at a speed of 3000 rpm. Next, 1 ml of the acetonitrile was transferred to a new tube containing the purification salt consisting of 300 mg of MgSO₄ and 50 mg of PSA. Subsequently, 1 ml of the supernatant was immediately transferred to a GC vial and acidified with 10 μL of 5% formic acid in acetonitrile. Finally, 1.0 μL of the extract was injected into the LC-MS/MS and GC/MS/MS system (Quarles et al., 2014).

Analysis techniques

For the analysis of pesticides in this study, two separation methods have been commonly used in literature: liquid chromatography (LC) for the analysis of groups such as organophosphates, carbamates, urea and its derivatives, and others, and gas chromatography (GC) for the analysis of the organochlorine group (Thurman et al., 2005).

UHPLC-MS/MS analysis

All UPLC separations were performed using an Acquity UPLC system (Waters, USA) equipped with a reversed-phase Acquity UPLC BEH C18 column (100×2.1 mm) (Waters, USA) and a mobile phase consisting of acetonitrile and water at a flow rate of 0.2 mL/min. The injected extract volume was 10 µL. A gradient was used, starting at 10% at $t = 0$ and reaching 50% at $t = 2$ min, 80% at $t = 12$ min, and 100% at $t = 13$ min, held until $t = 16$ min, and then returning to 10% at $t = 17$ min. The initial conditions were maintained for 8 min (until $t = 25$ min) for column re-equilibration.

For pesticide detection, a TQD Xevo mass spectrometer (Waters, USA) was used in positive electrospray ionization (ESI+) mode. The MS/MS transitions and analyte-dependent parameters such as collision energy and cone voltage were selected and adjusted by direct infusion of an individual pesticide standard solution into the mobile phase flow (methanol-water; 50:50, v/v, 0.3 mL/min) for all analytes.

The following parameters were used in all experiments: capillary voltage of 3.5 kV, extractor voltage of 4 V, source temperature at 120 °C, desolvation temperature at 250 °C, cone gas flow

rate of 100 L/h, and desolvation gas flow rate of 700 L/h (both gases being nitrogen). Argon was used as the collision gas at a pressure of 3.3×10^{-3} mbar. The optimized MS/MS transitions, as well as specific cone voltages and collision energies, are summarized in Table 1. The chromatographic elution zones were divided into appropriate time segments, and the corresponding MS/MS transitions were monitored using multiple reactions monitoring (MRM) mode (Kovalczuk et al., 2006).

UHPGC-MS/MS analysis

For the analysis of organochlorine pesticides, a Thermo gas chromatograph, model Trace 1310, was employed, equipped with a TG-5SILMS capillary column of 30 m length, 0.25 mm internal diameter, and 0.25 µm particle diameter. Helium was used as the carrier gas with a flow rate set at 1 mL/min. An aliquot of 1 µL of the sample was injected into the gas chromatograph. A temperature gradient was used to separate the targeted molecules, with an initial temperature of 80 °C, followed by a first ramp of 30 °C/min up to 150 °C, a second ramp of 5 °C/min up to 200 °C, and a third ramp of 20 °C/min up to 280 °C with a 5 min hold.

To detect organochlorine pesticides, a Thermo triple quad mass spectrometer, model TSQ Evo 8000, was used in SRM mode. The MS/MS transitions for the pesticides were developed by injecting and optimizing parent ions, daughter ions, and collision energies, which are summarized in Table 2.

In all experiments, the following parameters were used: a source and transfer line temperature of 250 °C, an electron energy of 70 eV, an emission current of 50 µA, a frequency of the three

Table 1. Pesticide identification ions and their respective retention times

Analyte	Tr (min)	MRM Transitions				S-lens (v)	
		Precursor ion	Product ion 1	CE 1	Product ion 2		CE 2
Carbendazim	4.9	192.09	132.08	29	160.08	17.0	81
Dictyophos	5.9	283.01	108.60	33	126.58	17.0	73
Tebuthiuron	8.8	228.95	115.59	26	171.63	17.0	72
Carbaryl	9.0	201.95	126.97	29	144.96	6.00	40
DEET	10.1	191.95	90.66	28	118.63	15.0	92
Malathion	12.3	331.01	98.57	2	126.86	12.0	60
Tebuconazole	14.2	308.01	69.66	29	124.56	35.0	97
TPP	14.4	327.09	77.02	37	152.07	33.0	98
Zoxamide	14.4	335.92	158.91	36	186.91	19.0	89
Chlorpyrifos	16.4	349.70	96.81	29	197.76	20.0	81
Bifenthrin	18.2	440.04	165.21	39	180.42	11.0	66

Table 2. Pesticide identification ions and their respective retention times

MRM Transitions						
Analyte	Tr (min)	No.	Precursor ion	Product ion	CE	Lens (v)
Héptachlore	8.87	MRM 1	271.8	236.9	15	15
		MRM 2	273.8	238.8	15	15

quadrupoles of 1091.5, 1861.5, and 1086.4, a detector gain of 1.0×10^5 , and the use of argon as the collision gas.

RESULTS AND DISCUSSION

Descriptive and multivariate statistical analyses were conducted using trial versions of IBM SPSS Statistics and R software. Table 1 presents the different chemical families (CF) and active substances (AS) of pesticides. Fifteen active substances were identified, of which 12 out of 15 belong to 5 chemical families. The families of the remaining three active substances were unidentified and classified under the “others” category. Table 3 summarizes the five chemical families clusters to be explored in this study.

The statistical software IBM SPSS Statistics (IBM Corp, 2023) was used to carry out the statistical calculations. Table 4 shows the average concentration of chemical families evaluated for

the three stations. Moreover, min value, max value, mean value (average concentration), standard deviation and p-value of a 95% confidence Fisher’s-test, have been calculated for different chemical families and tabulated in Table 4. As can be seen from Table. 4, for organochlorines, Fisher’s

Table 3. Chemical groups and active substances

Chemical groups	Active substances
Organophosphates	Dicrotophos DEET (N,N-diethyl-m-toluamide) Malathion Chlorpyrifos
Carbamates	Carbendazim Carbaryl Dazomet Mancozeb
Ureas and derivatives	Tebuthiuron Linuron
Organochlorines	Heptachlore
Pyrethroides	Bifenthrin
Others	Tebuconazol TPP (triphenyl phosphate) Zoxamide

Table 4. Mean concentration (Mean ± SD) ($\mu\text{g}/\text{kg}$) chemical groups of pesticides detected in sediment by station

Stations		Organophosphates	Organochlorines	Carbamates	Ureas	Pyrethroides	Others
		Mean ± SD ($\mu\text{g}/\text{kg}$) (min, max)	Mean ± SD ($\mu\text{g}/\text{kg}$) (min, max)	Mean ± SD ($\mu\text{g}/\text{kg}$) (min, max)	Mean ± SD ($\mu\text{g}/\text{kg}$) (min, max)	Mean ± SD ($\mu\text{g}/\text{kg}$) (min, max)	Mean ± SD ($\mu\text{g}/\text{kg}$) (min, max)
S1	US	494.67 ± 236.50 (326; 765)	ND -	433.67 ± 222.76 (287; 690)	248.67 ± 101.01 (147; 349)	85.33 ± 60.58 (45; 155)	408.00 ± 141.27 (245; 495)
	DS	390.33 ± 150.59 (217; 489)	ND -	369.67 ± 89.11 (267; 427)	204.33 ± 8.02 (196; 212)	138.33 ± 127.12 (0; 250)	187.33 ± 193.79 (0; 387)
S2	US	340.67 ± 74.14 (282; 424)	4.63 ± 0.04 (4,6; 4,67)	529.33 ± 189.44 (415; 748)	213.67 ± 204.26 (0; 407)	159,67 ± 139,64 (0; 259)	450.00 ± 023.85 (280; 676)
	DS	444.67 ± 48.78 (389; 480)	2.83 ± 0.07 (2,77; 2,91)	372.67 ± 185.51 (161; 507)	429,33 ± 122,45 (327; 565)	171.67 ± 151.37 (0; 286)	263.00 ± 426.42 (0; 755)
S3	US	419.00 ± 276.02 (167; 714)	2.07 ± 0.06 (2; 2,11)	473.67 ± 47.44 (420; 510)	133.33 ± 58.06 (68; 179)	85.67 ± 148.38 (0; 257)	468.33 ± 135.95 (320; 587)
	DS	544.33 ± 364.56 (125; 786)	12.90 ± 0.02 (12.89; 12.92)	208.67 ± 46.54 (161; 254)	174.00 ± 112.90 (67; 292)	60.33 ± 53.11 (0; 100)	292.67 ± 166.63 (130; 463)
Σ	US	418.11 ± 197.11	2.23 ± 2.01	478.89 ± 153.85	198.56 ± 128.24	110.22 ± 112.57	442.11 ± 143.93
	DS	459.78 ± 209.92	5.25 ± 5.87	317.00 ± 133.17	269.22 ± 146.78	123.44 ± 113.67	247.67 ± 252.98
	Σ	438.94 ± 198.69	3.74 ± 4.53	397.94 ± 162.55	233.89 ± 138.56	116.83 ± 109.95	344.89 ± 223.32
FI	Sts	P <0.79	P <0.001*	P <0.46	P <0.05*	P <0.43	P <0.82
	US/DS	P <0.69	P <0.001*	P <0.04*	P <0.23	P <0.82	P <0.10

Note: * – significant difference = 5%; SD – standard deviation; FI – fisher; min – minimum; max – maximum; US – upstream; DS – downstream; ND – not determined; Σ – total; Others – (Tebuconazole ; Zoxamide and TPP); S1 – Lagoon of Moulay Bousslham; S2 – Lagoon of Oualidia; S3 – Khnifiss Lagoon.

test revealed a significant difference between the three stations ($p < 0.001$) and between upstream and downstream of the three stations ($p < 0.001$). The means values indicate a relatively high concentration downstream of station 3 ($12.90 \pm 0.02 \mu\text{g/kg}$) (see Table 4). For carbamates, Fisher's test showed a significant difference between upstream and downstream of the three stations ($p < 0.04$). The maximum value was recorded at station 2 ($529.33 \pm 189.44 \mu\text{g/kg}$), as shown in Table 4. Concerning ureas, Fisher's test indicated a significant variation between the three stations ($p < 0.05$). The maximum concentration of urea was observed downstream of station 2 (see Table 4). Table 4 reports that there is no significant statistical difference between stations or between sampling points for the three families of pesticides (organophosphates, pyrethroids, others) using Fisher's test.

In this work, the Principal Component Analysis (PCA) was applied using the software R version 4.4.2 (R Core Team, 2022). The PCA technique is employed to analyze the chemical families of pesticides measured from the three stations of different lagoon sediments. This technique was performed on the dataset describing the concentrations of all chemical families over three stations (3 stations \times 15 chemical families), in order to characterize their correlations. Principal component analysis was performed on a data matrix formed by the results obtained over the study period. For this purpose, four principal components; plus one that was dismissed, were retained for the presentation of the data. Figures 2 and 3 show the results of the PCA performed on the used dataset.

Taking into account these results, the chemical families of organophosphates, organochlorines,

and carbamates are grouped on axis 1, while urea and others are on axis 2. The two axes (principal component) are retained, together explaining 79% of the total variance (inertia of axis 1:47.8% and inertia of axis 2:31.2%) as shown in Figure 2.

Stations and chemical families projection on the first factorial plane (presented by two principal components) as illustrated in Figure 3 can highlight four groups:

- Group 1: located on the positive side of axis 1, defined by high values of carbamates in the Oualidia lagoon (S2) upstream.
- Group 2: opposed to group 1 along axis 1, consisting of samples downstream of the Khnifiss lagoon (S3) characterized by high values of organophosphates and organochlorines.
- Group 3: based on the positive side of axis 2, composed of samples downstream of Oualidia (S2) with high values of urea, as well as the corresponding samples of the Moulay Bouselham lagoon (S1) downstream with moderate values.
- Group 4: opposed to group 3 along axis 2, characterized by high values of the "others" family (tebuconazole, zoxamide, and TPP) upstream of the Khnifiss station (S3).

The Moulay Bouselham lagoon (S1), whether upstream or downstream, is characterized by low concentrations of all chemical families. It can be noted that in both Stations, S1 (downstream) and S2 (downstream), similar concentrations of pyrethroids can be seen.

The aim of the study in the three lagoons along the Atlantic coast was to assess the level of sediment contamination by pesticides. Lagoons

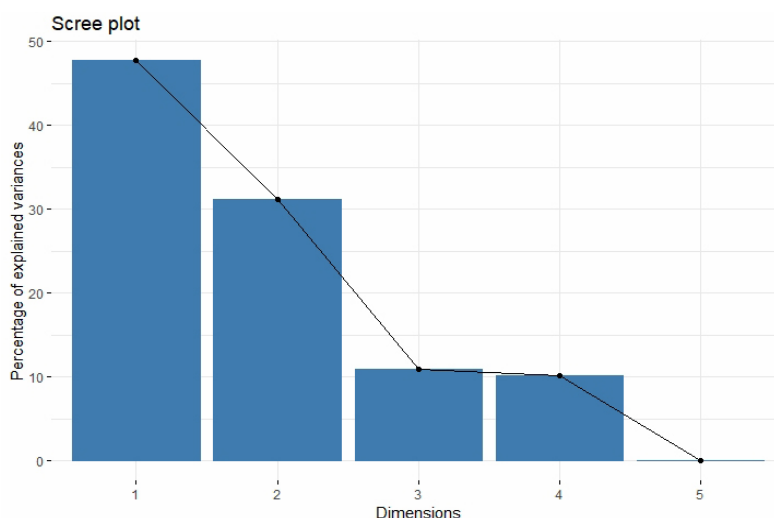


Figure 2. Absorption of PCA dimensions

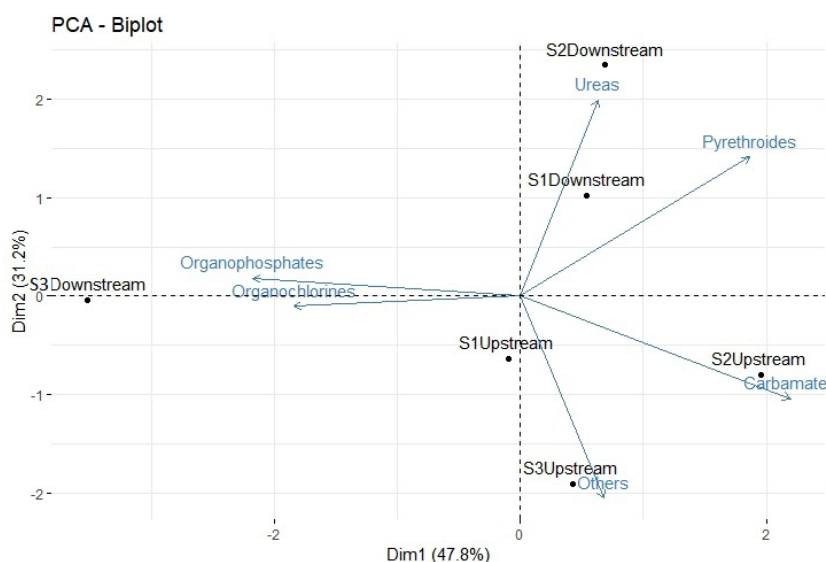


Figure 3. PCA of pesticide chemical families in sediments from the three stations

are susceptible to various types of pollution, yet only a few studies Benbakhta et al. (2014) and Lakhalki et al. (2017) have been conducted in this area. Fifteen active substances from five chemical families (Organophosphates, Carbamates, Ureas and their derivatives, Organochlorines, Pyrethroids, and others) were identified. In contrast, a previous study by Koffi Simplicite et al. (2018) conducted in the Ibrier lagoon in the Ivory Coast only identified two chemical families, triazines and substituted ureas.

Carbendazim and Mancozeb (carbamate family) were found in both Moulay Bouselham and Oualidia stations, which can be explained by the excessive use of these substances for crop protection against fungi. However, (Onssa, 2022), has banned both active substances after publication of the list of prohibited plant protection products. Mancozeb is known to have toxic effects on aquatic organisms and can lead to long-term adverse effects on the environment. Some pesticide families, such as organophosphates and carbamates, have a mode of action that can disrupt the normal functioning of the nervous system of living organisms (Sapozhnikova et al., 2004; Colović et al., 2013; Dara and Drabovich, 2023) induced by various inhibitors, leads to acetylcholine accumulation, hyperstimulation of nicotinic and muscarinic receptors, and disrupted neurotransmission. Hence, acetylcholinesterase inhibitors, interacting with the enzyme as their primary target, are applied as relevant drugs and toxins. This review presents an overview of toxicology and pharmacology of reversible and irreversible

acetylcholinesterase inactivating compounds. In the case of reversible inhibitors being commonly applied in neurodegenerative disorders treatment, special attention is paid to currently approved drugs (donepezil, rivastigmine and galantamine).

The analysis of herbicide results revealed the presence of two molecules (Tebuthiuron and Linuron) from the urea family, with higher average concentrations in the Moulay Bouselham and Oualidia lagoons, respectively (116.66 $\mu\text{g}/\text{kg}$; 153.5 $\mu\text{g}/\text{kg}$) and (109.83 $\mu\text{g}/\text{kg}$; 168 $\mu\text{g}/\text{kg}$). Exposure to excessive amounts of herbicides can lead to endocrine disruption, causing various animal health problems, including reproductive issues (Gupta, 2018).

The analysis of organochlorine molecule results revealed the presence of a cyclodiene group compound (Heptachlor), which has been banned in Morocco since 1984, along with other organochlorine molecules, due to its high toxicity, persistence in the environment (Yun et al., 2014) and potential threat to biodiversity and human health (Spalding et al., 2003; Rajendran et al., 2005; Teklu et al., 2016; Rohani, 2023) hexachlorocyclohexane (HCH. Heptachlor was found in the Khnifiss and Oualidia lagoons, with low concentrations of 7.48 $\mu\text{g}/\text{kg}$ and 3.73 $\mu\text{g}/\text{kg}$, respectively. However, it was not detected in the Moulay Bouselham lagoon.

A previous study by Benbakhta et al. (2014) conducted in the same station in 2014 revealed traces of heptachlor with a content of 0.4 $\mu\text{g}/\text{kg}$, while in the Oualidia lagoon, the concentration of heptachlor was 3.73 $\mu\text{g}/\text{kg}$, which far exceeded

the values obtained by authors, Benbakhta et al. (2014) and Lakhalki et al. (2017), with concentrations of 0.38 $\mu\text{g}/\text{kg}$ and 0.67 $\mu\text{g}/\text{kg}$, respectively. The source of contamination may be agricultural or due to industrial and sewage discharges, as their use is highly variable for weed control in agricultural fields and shrubbery on forage crop sites (pastures and ranges) (Artiola et al., 2019).

The active substances identified in the study (Malathion, Chlorpyrifos, Dicrotophos) belong to the Organophosphate family, (Carbaryl, Dazomet) to the Carbamate family, and (Bifenthrin) to the Pyrethroid family. These pesticides are commonly used as insecticides to protect crops against harmful insects, but they are also among the most prevalent plant protection products in terms of pollution, as they are increasingly found in the environment. The results confirm their presence in all stations, with values ranging from 68 $\mu\text{g}/\text{kg}$ to 203.5 $\mu\text{g}/\text{kg}$.

It is worth noting that some of these pesticides have been banned in many countries, including the European Union, such as Malathion, which has been banned since 2007, and Chlorpyrifos in December 2019. In Morocco, Chlorpyrifos, Dicrotophos, and Carbaryl are not on the list of approved active substances according to ONSA's website (Onssa, 2022). Another insecticide, DEET (N,N-diethyl-3-methylbenzamide), was identified in the Khnifiss region, with a concentration of 113.16 $\mu\text{g}/\text{kg}$. It is used for protection against annoying and biting mosquitoes.

CONCLUSIONS

The analysis of sediment samples from Moroccan Atlantic coast lagoons indicates the presence of pesticides, raising significant environmental concerns. Although some active substances, such as Chlorpyrifos, Dicrotophos (Organophosphates), and Heptachlor (Organochlorines), have been banned, their persistent presence in the sediments of Khnifiss and Oualidia lagoons highlights the long-term impact of pesticides on the environment. However, the Moulay Bousselham lagoon appears to have been less affected by contamination.

Morocco has implemented new and highly effective regulations to combat the use of harmful pesticides in agriculture. Nevertheless, there are still challenges to be addressed to ensure responsible use of these products. Additional actions

should be considered to limit pesticide pollution, protect biodiversity, promote environmental sustainability, and preserve human health. Close collaboration between governments, farmers, scientists, and consumers is necessary to create a more sustainable and healthier food system for all.

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