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Organochlorine pesticides in sediments from the atlantic coast of Morocco

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ABSTRACT: Organochlorine pesticides (DDT and its metabolites, HCH isomers and cyclodiènes compounds) were determined in sediment collected from five locations of the moroccan atlantic coast during the period of January – December 2010. Samples were analysed according to the UNEP/FAO protocol using non polar solvent extraction and a conventional GC-ECD apparatus. The highest levels of organochlorine pesticides were found in Sebou estuary (27.53 ng/g) and Moulay Bousselham lagoon (25.58 ng/g). DDT and its metabolites presented high concentrations at all locations, the concentration of DDTs ranged from 2.40 ng/g to 25.40 ng/g. HCH isomers were also present but at low levels, the concentration of γHCH ranged from 1.93 ng/g to 5.93 ng/g. Cyclodiènes compounds (Aldrin, Endrin, Dieldrin, Heptachlor and Heptachlor epoxide), banned since 1984 in Morocco, were only detected in a few sample locations at low concentrations, it implies that the exhibition to these products stopped. In five locations, the concentrations of parent compound (p,p' DDT) were always higher than other metabolites p,p'DDE and p,p' DDD in all sediment samples. The ratio p,p' DDT/DDE indicated slow degradation of DDT or recent input in this environment. The use of all OCP has been prohibited in Morocco since 1984, the reason for organochlorine pesticides contamination in this sites can be related to the still widely and illegal use of OCP in agriculture

KEYWORDS: Organochlorine pesticides, DDT, HCH, sediment, Atlantic coast, Morocco.

1 INTRODUCTION

Organochlorine pesticides (OCPs), such as DDTs (dichloro-diphenyl-trichloroethanes) and HCHs (hexachlorocyclohexane), represent an important group of persistent organic pollutants (POPs) that have caused worldwide concern as toxic environmental contaminants [1], [2], [3].

Because of their toxicity, persistence, accumulation in biota, and adverse impacts on wildlife, the majority of organochlorine pesticides were banned in the developing countries during 1970 [4]. In Morocco, these compounds were banned since 1984 except for lindane (γHCH) and DDTs that are tolerated in public health emergencies [5].

Organochlorine pesticides were widely used from 1960-1984 in Morocco both for agricultural and sanitary purposes. It estimated that about 10000 tons were used annually and DDT accounted for 50% of this group [6].

Many authors have been investigating the occurrence and distribution of OCPs in the marine sediment around the world. Some of the important publications in the field in the last decade are as follows : [1], [2], [4], [7], [8], [9], [10], [11], [12], [13],

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[14]. In Morocco, only one survey has been achieved on the OCPs in sediment and biota of the Moulay bousselham lagoon [15].

The purpose of the present study is to evaluate the levels of some organochlorine pesticides in sediments collected from five locations of the Moroccan Atlantic coast. The study includes the most important estuaries and coastal lagoons in Atlantic coast of Morocco: Moulay Bousselham lagoon (S_1), Sebou Estuary (S_2), Bou Regreg Estuary (S_3), Oum Rbia estuary (S_4) and Oualidia lagoon (S_5). (Fig. 1)

2 MATERIALS AND METHODS

2.1 STUDY SITES

We studied two stations in each location:

The Moulay Bousselham lagoon, commonly called merja zerga, is situated on the atlantic north coast of Morocco (34.83ºn, 6.27ºe, fig.1). the lagoon has an ellipsoidal shape in a north-south direction, is 9 km in length and 5 km wide in its widest portion; its area is 35 km². The lagoon system receives drainage mainly from the drader river to the east and the nador canal to the south. The drader river channel divides the lagoon into two sub-basins, merja kahla to the north with an average depth of 0.3-0.5 m and merja zerga to the south with 1-1.5 m depth. . The main activities are land cultivation and cattle raising, artisanal fishing and shellfishing.

The basin of the Sebou river (88°n and 35°n to 4°w and 7°w) covers an area of 40,000 km2. The river that empties into it passes through the gharb plain that has extensive agricultural and industrial activities. Kenitra harbor, about 10 km from the ocean, has commercial trafic, while Mehdiya harbor at only 1 km from the mouth is busy with fishing activities (fig. 1). The average Sebou river output reaches 200 $m^3 s^{-1}$. The annual input volume is about $5x10^9$ m3 of freshwater and may double in rainy seasons. According to Combe et al. [16], the tidal height varies from 0.9 to 3.10 m along the estuary, depending on location.

The catchment of the Bou Regreg river has an area of 9700 km2. The estuary (34°n– 6°50'w) located between the cities of Rabat and Sale (fig. 1), extends for some 23 km from the ocean to a dam (sidi Mohamed Ben Abdellah) built in 1974, and recently to a breakwater implanted at 18 km upper to the mouth. The absence of a real fluvial flow, following the disappearance of flooding events, clearly influences the estuary's functioning, where the hydrodynamics are governed by the tides (their height varies from 0.5 to 3.3 m). The estuary is dominated by seawater, with minimal influx of freshwater for most of the year. near the mouth, there are agricultural and industrial areas. Some urban sewage from Rabat and Sale is directly discharged into the estuary.

The basin of the Oum er Rbia river covers an area of 34,335 km2. The hydrological parameters of this river were studied by Snoussi [17]. Downstream, the annual average output reaches $117m^3s^{-1}$. The seasonal fluctuations reveal a maximum in spring occurring from the middle and grand atlas rivers. The summer outputs emanate mainly from the grand atlas sources. The estuary extends for some 15 km, from the ocean to a breakwater (33°21'n–8°21'w, fig. 1). The city of Azemmour, sited along the west bank, discharges urban waste water directly into the lower estuary (fig. 1). Upstream, the catchment is mostly influenced by agricultural activity and associated light industry. The removal of any important freshwater inputs and any flooding events make Oum er Rbia a system similar to the Bou Regreg estuary.

The Oualidia lagoon (32°40'4"n- 32°47'07"n and 8°52'30"w – 9°02'50"w) is located on the atlantic ocean (fig. 1). This lagoon is 7 km long, on average 0.5 km wide, and exchanges water with the ocean through a major inlet about 150 m wide and 2 m deep. During spring tides there is also a secondary, shallower inlet about 50 m wide. An internal delta with a surface area of about 0.2 km2 is normally found close to the inlet. The lagoon morphology is characterized by side channels, connected to a meandering main channel, in which the mean depth is 2 m and the maximum depth during flood tides does not exceed 5 m [18]. The major aquaculture activity in the lagoon is oyster culture. To date, five oyster farms occupy one-sixth of the lagoon surface. In addition, during summer Oualidia lagoon is the site of intense tourism activities. Discharging farm and domestic sewage water without treatment can cause increased contamination.

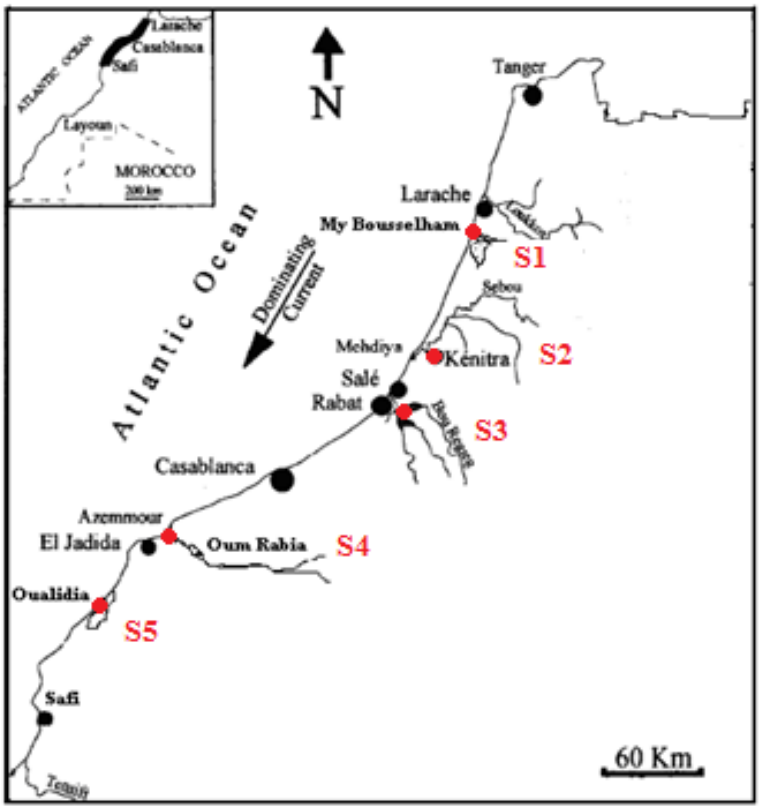

Fig. 1. Location of surveyed sites

2.2 SEDIMENT SAMPLING

A total of 40 sediment samples in five sites were collected during sampling campaigns on February, May, August and November 2010. Fig.1. shows the study area and the sampling locations.

The superficial sediments (top 0–3 cm) were collected with a stainless steel grab and then placed into polytetrafluoroethylene (PTFE) bags. All the samples were immediately transferred to the laboratory and kept at – 20°C in the refrigerator.

2.3 EXTRACTION AND ANALYSIS

The analytical procedures used for the extraction, purification and quantification of organochlorine pesticides in sediment were identical to those recommended by UNEP/FAO [19]. The samples were freeze-dried, homogenized and passed through a 63-µm sieve. Approximately 20g were spiked with ε hexachlorocyclohexane (εHCH) as an internal standard and extracted in a soxhlet extraction apparatus for 8 hours with 200 ml n- hexane. The extracts were concentrated to about 15 ml. These extracts were treated then by mercury to remove sulphur compounds. The separation was achieved by florisil column chromatography. The florisil was activated in the oven to 130 °C during 12 hours then it was partially deactivated with 0,5% distilled water (W/W). Approximately 2 cm of sodium sulphate were added evenly over the florisil to remove humidity.

The organochlorine pesticides were separated and quantified by gas chromatography using a VARIAN GC-3380 gas chromatograph equipped with ^{63}Ni electron capture detector (ECD). The separation was performed on a fused silica capillary column (CP-Sil 8 CB, 30 m x 0,25 mm id, and 0,25 µm film thickness) with nitrogen as carrier gas and make up gas. The operating conditions were as follows : Injector temperature 260 ° C; detector temperature 280 °C; oven temperature: initial 50 °C for 2 min, programmed to 160°C at 5°C/min, followed at 2°C/min to 260°C, final 260°C for 10 min.

The residue levels of OCPs were quantitatively determined by the internal standard method using peak area. For every set of five samples, a procedural blank and a spiked sample with standards were run to check for the interference and cross-

contamination The detection limits (LD) of OCPs were determined as the concentration of analyses in a sample that gives rise to peak a signal –to-noise ratio (S/N) of 3. The pesticides recoveries were determined relative to the ratio of direct injection of extract and the working standards prepared in hexane. The mean recovery of OCPs was estimated at mean concentration levels.

2.4 QUALITY CONTROL

A strict regime of quality control was employed before the onset of the sampling and analysis program. OCPs recoveries were undertaken to demonstrate the efficiency of the method.

Analytical standards of composite OCPs including DDTs (p,p'-DDT, p,p'DDD, p,p'DDE) and HCHs (αHCH, γHCH) as well as aldrin, endrin, dieldrin, heptachlor, heptachlor epoxide, HCB and α endosulfan were provided by IAEA/ILMR Marine Environmental Studies Laboratory in Monaco. The spiked recoveries of OCPs using 200 ng of composite standards were in the range of 87–103% and the relative standard deviation (RSD) values ranged from 2% to 14%. Table 1 listed the detection limits (DL), mean recovery and relative standard deviation (R.S.D.) of the methods.

Table 1. Mean recovery, relative deviation (R.S.D.) and detection limit (DL) of OCPs in sediment (at 10ng/g –dw)

OCPs	Sediment (n=5) Recovery (%)	R.S.D (%)	DL (ng/g- dw)
4,4'DDT	74	6	0.12
4,4'DDE	82	8	0.40
4,4'DDD	88	5	0.14
γ HCH	83	10	0.12
α HCH	78	5	0.14
Aldrine	75	6	0.12
Endrine	85	8	0.12
Dieldrine	76	6	0.12
Heptachlor	77	5	0.14
Heptachlor époxide	76	6	0.14
HCB	84	8	0.12
α Endosulfan	83	6	0.12

3 RESULTS AND DISCUSSIONS

3.1 CONTAMINATION STATUS OF OCPS

The analytical results for studied compounds in sediment from five locations are represented in table 2.

The total organochlorine pesticides (OCPs) concentration in sediments varied from 13.17 to 27.53 ng/g with an average 20.52 ng/g of wet weight. The concentrations of $\sum$OCPs were higher at Sebou Estuary (27.53 ng/g), Moulay Bousselham lagoon (25.58 ng/g) an Oualidia lagoon (22.86 ng/g). The minimum value was observed at Bou-regreg Estuary (13.17 ng/g).

Among compounds searched, the DDT and its metabolites (DDTs) and the lindane (γHCH) are the most fluently detected (83 -100%). The DDT group of compounds was detected in relatively high concentrations in all sediment samples, but the contribution of individual metabolites showed differences. The concentration of $\sum$DDTs reached maximum value at Sebou Estuary (17.73 ng/g) followed by Moulay Bousselham lagoon (17.03 ng/g) and Oualidia lagoon (14.46 ng/g). The minimum value of DDT total was recorded at Bou-regreg Estuary (7.10 ng/g) and Oum Rbia Estuary (5.73 ng/g).

Table 2. Concentration range and mean (ng/g wet wt.) of Organochlorine pesticides from the five sampling sites

Compounds		My Bousselham Lagoon n=8	Sebou Estuary n=8	Bou Regreg Estuary n=8	Oum Rbia Estuary n=8	Oualidia Lagoon n=8
4,4'DDT	x ±SD	8.24 ± 3.43	7.95 ± 3.45	3.71 ± 1.98	2.67± 0.90	8.33 ± 3.43
	Range	6.25 -10.60	5.60 – 14.40	1.20 – 6.80	1.82 – 4.10	4.88 – 13.10
4,4'DDE	x±SD	4.85 ± 0.56	5.13 ± 1.83	1.79 ± 0.61	1.91 ± 0.45	3.62 ± 2.29
	Range	3.58 - 5.70	2.40 – 7.60	1.20 – 2.50	1.40 – 2.60	1.04 – 5.21
4,4'DDD	x±SD	3.94 ± 1.50	4.23 ± 0.61	1.60 ± 0.90	1.15 ± 0.48	2.51 ± 1.28
	Range	1.58 - 6	3.4 – 5.20	<0.14 – 2.47	0.80 – 1.96	0.9 – 4.69
∑ DDTs	x±SD	17.03 ± 4.49	17,31 ± 5.89	7.10 ± 3.49	5.73 ± 1.83	14.46 ± 7
	Range	11.41 – 20.50	13.20 – 25.4	2.40 – 11.87	4.22 – 7.29	7.49 - 23
γ HCH	x±SD	3.64 ± 2.26	5.83 ± 1.20	1.93 ± 1.31	2.96 ± 2.25	3.46 ± 0.88
	Range	<0.12 – 6.20	3.78 –6.80	<0.12– 4.10	0.88 – 6.30	2.40 – 4.40
α HCH	x±SD	1.65 ± 1.55	1.01 ± 1.31	0.96 ± 1.15	<0.14	1.70 ± 1.00
	Range	<0.14 – 4.10	<0.14 – 3.30	<0.14 – 2.50		<0.14 – 4
∑ HCHs	x±SD	5.29 ± 3.81	6.84 ± 2.51	2.89 ± 2.46	2.96 ± 2.25	5.16 ± 1.88
	Range	1.40 – 6.20	3.78 – 9.10	<0.14– 6.20	0.88 – 6.30	2.54 – 6.20
Aldrine	x±SD	0.60 ± 0.68	0.80 ± 0.99	1.06 ± 0.71	1.10 ± 0.21	0.50 ± 0.52
	Range	<0.12– 1.5	<0.12– 2.45	<0.12 – 1.25	<0.12– 1.30	<0.12– 1.25
Endrine	x±SD	0.84 ± 0.90	0.35 ± 0.48	0.52 ± 0.40	0.65 ± 0.51	0.46 ± 0.40
	Range	<0.12– 2.45	<0.12– 0.95	<0.12– 0.85	<0.12– 1.15	<0.12– 1.96
Dieldrine	x±SD	0.46 ± 0.52	0.28 ± 0.40	0.45 ± 0.74	0.80 ± 0.49	0.45 ± 0.78
	Range	<0.12 – 1.2	<0.12– 0.85	<0.12– 1.75	<0.12– 1.25	<0.12– 1.50
Heptachlor	x±SD	0.40 ± 0.44	0.28 ± 0.40	0.34 ± 0.54	0.69 ± 0.39	0.38 ± 0.30
	Range	<0.14–0.85	<0.14 - 0.90	<0.14– 1.20	<0.14 – 0.95	<0.14– 0.80
Heptachlor époxide	x±SD	0.13 ± 0.31	0.26 ± 0.37	<0.14	0.32 ± 0.43	0.28 ± 0.49
	Range	<0.14 – 0.78	<0.14 – 0.85		<0.14– 0.85	<0.14– 0.85
HCB	x±SD	0.70 ± 0.79	1.06 ± 0.85	0.79 ± 0.69	0.48 ± 0.65	0.57 ± 0.58
	Range	<0.12– 1.75	<0.12– 1.85	<0.12– 1.75	<0.12– 1.25	<0.12– 1.25
α Endosulfan	x±SD	0.12 ± 0.30	0.30 ± 0.42	<0.12	0.70 ± 0.40	0.55 ± 0.57
	Range	<0.12–0.75	<0.12 - 0.95		<0.12– 0.95	<0.12– 1.25
∑OCPs		25.58	27.53	13.17	13.46	22.86

x = mean, SD = Standard deviation
∑ DDTs = sum of DDT, DDE and DDD
∑ HCHs = sum of γ HCH and α HCH
∑ OCPs = sum of total organochlorine pesticides

The concentrations of total HCHs (sum of α and γHCH) ranged from 2.89 ng/g to 6.84 ng/g. The levels are comparable to those found in sediment of Moulay Bousselham lagoon reported by Mehdaoui et al. [15]. γHCH (lindane) was the major HCH compounds being over 50% at most stations. Concentrations of HCHs in all sediment samples from five locations of Moroccan atlantic coast were much lower than those of DDTs (table2).

The isomeric of HCH was not uniform. The Concentration of γHCH ranged from 1.93 ng/g to 5.93 ng/g, were always higher than αHCH (<0.14 –1.70 ng/g). The mean percentage composition of α and γHCH to ∑HCHs for sediments analysed in our study are 0 - 33% and 66 - 100 % respectively.

Compounds cyclodiènes (Aldrin, Endrin, Dieldrin, Heptachlor and Heptachlor epoxide), banned since 1984 in Morocco, were only detected in a few sample locations at low concentrations, it implies that the exhibition to these products stopped. Aldrin was detected in 50 % of all sediments collected, with higher frequency in samples from S_1 at low concentration range (<0.12-2.45 ng/g). Endrin was only detected in a few sample locations at low concentrations between 0.35 ng/g and 0.84 ng/g. Concentrations of dieldrin were lower, ranging from 0.28 ng/g to 0.80 ng/g. Heptachlor was detected at up 1.20 ng/g and heptachlor epoxide, a degradation product of heptachlor, at a peak concentration of 0.85 ng/g.

3.2 SPATIAL AND TEMPORAL DISTRIBUTION OF OCPS IN SEDIMENT

The distributions of ∑DDT and ∑HCH in sediments in four seasons along the five sampling sites were shown in Figs. 2 and 3 respectively. Fig.2 shows that the concentrations of ∑DDT in sediments for samples S1 and S2 collected from Moulay Bousselham lagoon and Sbou Estuary displayed different high distribution.

The level of contamination of sediment by POC especially DDTs presented a temporal variation (Fig. 2), for all stations the highest concentrations are obtained in may (spring) and the lowest in the november (autumn). Whereas, the level of lindane showed no temporal and spatial variation (Fig. 3).

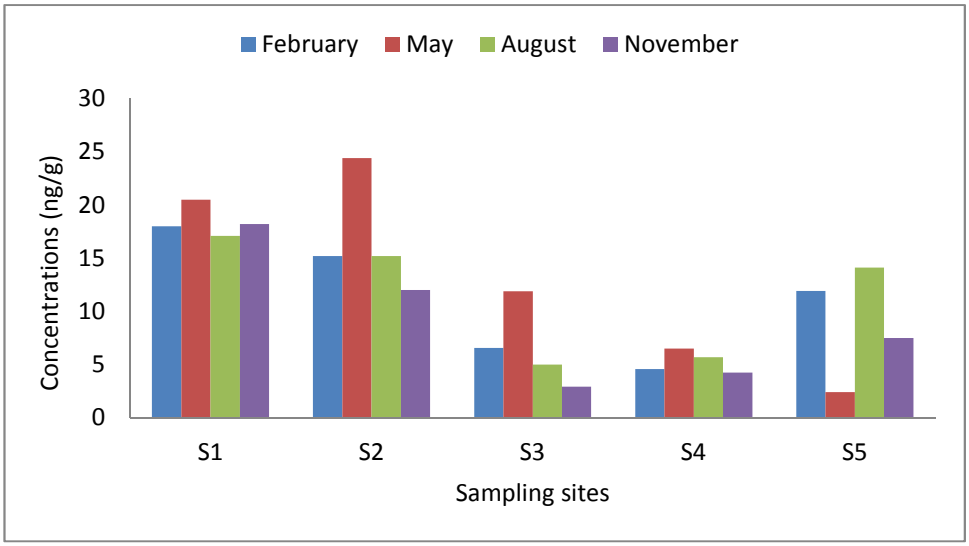

Fig. 2. Distribution of DDTs in sediments from five sampling sites

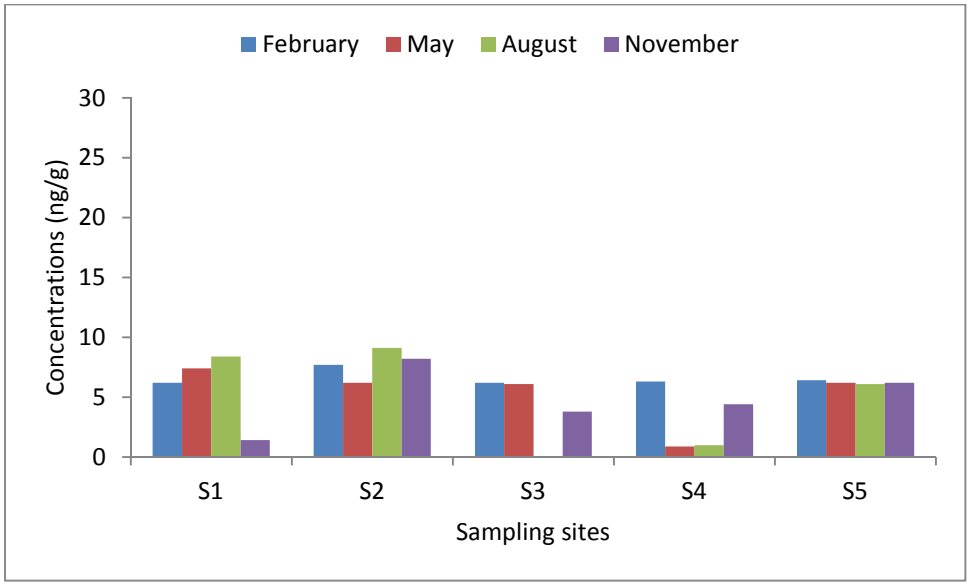

Fig. 3. Distribution of HCHs in sediments from five sampling sites

The analysis of variance carried out on the levels of pesticides organochlorines (POC) and DDTs in sediment revealed through the Fisher test significant differences ($p < 0.05$) between stations and seasons (Table 3). Furthermore, comparison of the levels of the variances on Lindane showed no significant difference between the sediment in different stations.

Table 3. ANOVA for pesticides total organochlorines (POC), DDTs and lindane in sediment (F test significant if p <0.05).

	POC	DDTs	Lindane
locations	0.0084	0.0069	0.3042

3.3 COMPOSITION ANALYSES IN SEDIMENTS

The relative concentrations of the parent compounds and their metabolites are very useful in providing information on source and history of input to environment and possible degradation pathway involved. In this investigation, the concentrations of parent compound (p,p' DDT) were always higher than other metabolites p,p'DDE and p,p' DDD in all sediment samples. The occurrence of DDT isomers is predominant in the following order : p,p' DDT> p,p'DDE > p,p' DDD. The study showed p,p'DDT contributed to 48 %, 46 %, 52 %, 47 % and 57 % of the total DDT at the sites in S_1, S_2, S_3, S_4 and S_5 Respectively. The predominance of DDTs in the sediment was reported by Mehdaoui et al. [15] from Moulay Bousselham lagoon.

According to Strandberg at al. [20], the ratio of p,p'-DDT /p,p'DDE provides a useful index to know whether the DDTs at given site is fresh or aged. Further, a value <0.33 generally indicates an aged input. In the present study, the value of >0.33 was found in five locations, indicating slow degradation of DDT or recent input of DDT in this environment.

Composition differences of HCH isomers in the environment could indicate different contamination sources [21]. Regarding the composition of HCH isomers measured in this study, a high percentage of γHCH was recorded in five sampling sites. The average composition of HCH isomers measured in this sediment samples are α-HCH: 25 %, γ-HCH : 75 % (Fig). It can be concluded that lindane may be used recently in this environment

3.4 COMPARISON OF OCPS LEVELS IN DIFFERENT REGIONS

The comparison of organochlorine pesticide concentrations from various regions was shown in Table 4. Level of OCPs in surface sediment in this study were generally low in comparison with other Atlantic coastal areas (lagoon of Abidjan and road of Brest), Mediterranean coastal areas (Alexandria Harbour, Lake Manzala) and coastal areas of Asian including Arabian Sea, juilong river estuary, Hanoi region and Pearl river estuary. The residues of OCP in all sites of Moroccan Atlantic coast were similar to those found in Victoria harbour (Hong Kong), Salton Sea and Sparta, IL (USA), but much higher than those reported for Northeastern coast (India) and Rio Palizada (Mexico).

Table 4. Comparison from other marine environments in the world of total Organochlorine pesticides OCPs (ng/g, dry wet)

Locations	OCPs	References
Abidjan Lagoon, Ivory coast	2.52-48	[22]
Road of Brest, France	4 - 49	[23]
Alexandria Harbour, Egypt	5.13-1214	[24]
Victoria harbour, Hong Kong	3.5-32	[25]
Arabian sea, India	4.25-68.40	[26]
Juilong river Estuary, China	15-115	[27]
Lake Manzala, Egypt	9-114	[28]
Rio Palizada, Mexico	0.88- 18	[29]
Northeastern coast, India	0.65-2.48	[1]
Salton sea, California, USA	19-34	[4]
Hanoi region, Vietnam	8-86	[13]
Pearl river estuary, China	10.4-70.6	[30]
Sparta, Il, USA	8-35	[31]
Atlantic Coast, Morocco	13.17-28	Present study

4 CONCLUSION

The analyse of sediment samples from five sites of Moroccan Atlantic coast showed a very lower contamination by organochlorine pesticides although the use of these compounds is only banned since 1984 in Morocco. The high percentage

composition of DDT observed in this study clearly illustrates that DDT usage has not been eradicated yet in country, and there might be new input of DDT to the coastal marine environment. Morocco continued to use extremely DDTs and lindane (γ HCH) in control of some pest disease.

Consequently, the reason for organochlorine pesticides contamination in this sites can be related to the still widely and illegal use of OCP in agriculture. Although use of all OCP has been prohibited in Morocco by a decision made by Moroccan government in the 1984, we observed the unfortunately illegal use of some OCP such as DDT still continues in Morocco

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