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Note

Contamination of some aquatic species with the organochlorine pesticide chlordecone in Martinique

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Abstract – Martinique is a French overseas department whose economy relies heavily on agriculture. Organochlorine pesticides, mainly chlordecone, were used for banana cultivation to eradicate banana weevil over a period of 40 years. Chlordecone is chemically stable,and has a strong affinity for fatty tissues. It is therefore able to bioaccumulate in animals and thereby represent a threat to ecosystems and man. Soils from banana plantations in Martinique are heavily contaminated with chlordecone. Possible transfer of these molecules from agricultural watersheds to the aquatic environment and the organisms that live in it is feared. The hypothesis that ecosystems of Martinique might be highly contaminated with this organochlorine pesticide was investigated. Chlordecone levels were measured in various freshwater and marine species. Data show a heavy contamination of many carnivorous and detritivorous species (fish and prawns). Concentrations measured in wild or farmed tilapia are among the highest ever reported in the literature. Some coastal species (fish and lobster) were also found to be contaminated, although to a lesser extent. Given the biogeochemical behavior of chlordecone, the most likely route of contamination is food. Detected concentrations in marine organisms are below the tolerated limits established by authorities, however, the impact of other sources of exposure, namely, contaminated water and root vegetables, remains to be investigated.

Key words: Pesticides / Contamination / Chlordecone / Aquatic organisms / Food web / Caribbean Islands / W Atlantic

1 Introduction

Intensive banana farming practiced in tropical climates such as that of Martinique, leads to an increased vulnerability of crops to parasites and to the use of considerable amounts of pesticides. The use of organochlorines to eradicate insects such as the banana weevil first started in the nineteenfifties. Organochlorines are chemically stable lipophilic molecules which persist in the environment and are known as ecotoxic molecules. Molecules of this family have been prohibited in mainland France because of their ability to bioaccumulate. Authorization to sell chlordecone was withdrawn in 1990. As no other approved insecticide against banana weevil was available at the time, an exemption was granted that allowed the use of chlordecone (Kepone®) in the French West Indies until September 1993.

Chlordecone is still be detected in the different ecosystems of Martinique 10 years later (DSDS 2001). Investigations on chlordecone in the environment of Martinique first started in June 1999. Chlordecone was first detected in streams and rivers that received water from agricultural watersheds located in the northern part of the island. During this study, the local Health Authority of Martinique identified chlordecone in root vegetables, such as Chinese cabbages, sweet potatoes and Carribean cabbages, grown in contaminated soils.

Organochlorines are hydrophobic and adsorb to the organic matter in the soil. Due to erosion of soil particles, desorption phenomena, slow solubilisation and infiltration processes, these compounds reach the groundwater table as well as many small streams that flow directly into the sea, which is never very far on the island of Martinique. The ensuing contamination of the aquatic environment threatens the organisms that live in it. Part of the fishery products in Martinique may therefore be contaminated.

This study focuses on contamination of freshwater and marine organisms which may contribute to increasing human population exposure to organochlorines in Martinique, and as such it is aimed at assessing the level of contamination of these organisms with chlordecone.

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Table 1. Characteristics of sampled species and sampling techniques.

Scientific names	Common names	Characteristics	Sampling techniques
Acanthurus bahianus	Surgeon fish	Coral reef fish, herbivorous	Fish pots
Sparisoma sp.	Parrot fish	Coral reef fish, herbivorous	Fish pots, fishing nets
Mugil sp.	Mullet	Fish that lives on sandy bottoms, feeds on small marine organisms, detritivorous, omnivorous	Bottom gillnets
Decapterus sp.	Mackerel scad	Pelagic fish, carnivorous	Fishing nets
Engraulidae, Dussumieridae	Anchovies Blue sprat	Coastal fish. Juveniles of many species	Beach seines
Anchoa lyolepis	Shortfinger anchovy	Coastal fish, plankton feeder	Beach seines
Oreochromis sp.	Wild red tilapia	Brackish water fish, omnivorous	Line fishing
Sciaenops ocellatus	Red drum	Marine fish, carnivorous	Farm-raised in floating cages
Oreochromis sp.	Farmed red tilapia	Freshwater fish	Farm-raised in ponds
Macrobrachium rosenbergii	Freshwater prawn	Freshwater shellfish	Farm-raised in ponds
Panulirus argus	Spiny lobster	Marine shellfish, necrophagous and detritivorous	Coastal cages Trammel nets
Lucina pectinata	Tropical clam	Bivalve mollusc	Collected in shallow waters
Crassostrea rhizophorae	Mangrove oyster	Bivalve mollusc	Collected on mangrove roots
Cardiidae	Cockles	Bivalve mollusc	Collected in shallow waters

2 Materials and methods

Sampling

Samplings were performed in fishing areas from which most of the local fish and shellfish consumed in Martinique comes from, as well as in inland streams and rivers which may have been exposed to organochlorines. Species were selected based on their feeding behaviors. Selected carnivorous fish species and detritivorous shellfish species were known to contribute to the biomagnification of chlordecone up the food chain. Other strict herbivorous species were also selected. The surgeon fish and the parrot fish were selected because they are ubiquitous, abundant, and able to ingest fine sediment. We selected filter-feeding bivalves in which contaminated particulate matter might have accumulated. Nine fish species, two shellfish species, and three bivalve species were collected in rivers, in the sea, and in fish farms in January and February 2002 (Table 1). A total of 260 animals were collected in the 27 sampling sites (Fig. 1). Numbers in bold correspond to the number of chlordecone-positive samples. Collected animals were kept frozen (−80 °C) and shipped to mainland France on dry ice for analysis. Samples from animals of the same species collected at once at the same site were pooled. A total of 99 50-g samples of flesh were thus obtained, each sample being a pool of 10 individuals.

Chlordecone levels were measured in the dorsal muscle in fish, in the abdominal muscle in shellfish, and in total flesh in molluscs.

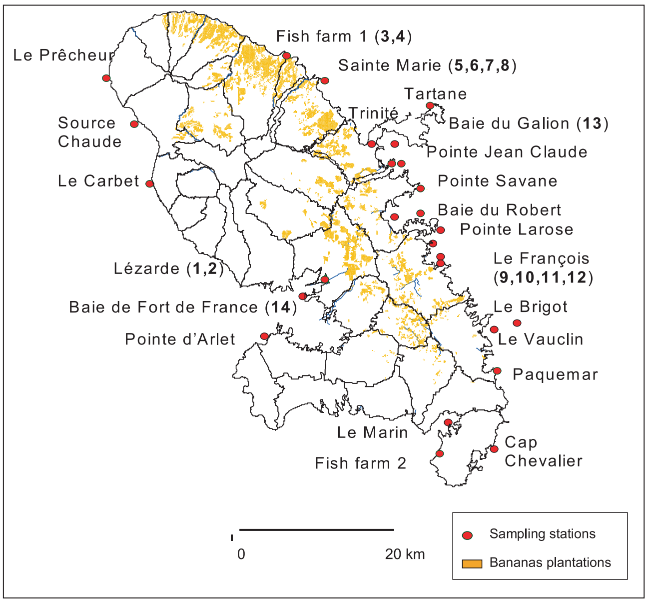

Fig. 1. Sampling stations (numbers in brackets correspond to samples where chlordecone levels were positive) in Martinique, West Indies, Caribbean Islands.

Chemical analysis

Sample processing (extraction and purification) and measurement of chlordecone pesticide concentrations were carried out at the Laboratoire Départemental d'Analyses de la Drome (Valence).

Cryogenic centrifugation extraction were NF EN 1528-compliant (European Reference Norme for the analysis of organochlorine compounds). Solid phase extractions were carried out according to the AFSSA-LERHQA-CENPOP/01 method developed by l'Agence Française de Sécurité Sanitaire des Aliments (Laboratory for Studies & Research on Hygiene

Table 2. Chlordecone levels in a number of aquatic animal species. Results are given in μg kg^{-1} wet weight on a pool of 10 individuals.

N°	Sampling site	Species	Chlordecone (µg kg^{-1})	Comments
1	Lézarde	Wild red tilapia	386	This river runs through banana plantations.
2			196	
3	Fish farm 1	Farmed red tilapia	132	Fish farm located on polluted grounds in the Northeast of the island.
4		Freshwater prawn	23	
5	Sainte-Marie	Mackerel scad	3	These fish migrate upstream to the mouth of the river in the Northeast of the island.
6			4	
7		Shortfinger anchovy	7	
8			4	
9	Baie du François	Spiny lobster	20	Enclosed areas (bays, inlets).
10			31	
11			13	
12		Surgeon fish	4.1	
13	Baie du Galion	Spiny lobster	10	
14	Baie de Fort-de-France	Surgeon fish	1.2	

	Freshwater		Brackish water		Seawater

& Quality of Foods). This method follows a four-step procedure:

1. Fats are extracted from samples with hexane.
2. Pesticides are extracted from fats by acetonitrile-hexane partitioning.
3. Purification, i.e., elimination of interfering substances, is achieved using C18 and Florisil solid phase extraction cartridges.
4. Levels of organochlorines are measured by gas-chromatography (GC) with electron capture detector (ECD). GC conditions were as follows: DB5 column, 60 m lenght, 0.25 mm diameter. The GC oven was held at 60 °C for 4 min, then ramped to 200 °C at 20 °C min^{-1}, held at 200 °C for 20 min, then ramped to 320 °C at 5 °C min^{-1}and finally held at 320 °C for 10 min. The GC inlet was operated in splitless mode at 250 °C, with a 1 μl injection volume. The detector was held at 300 °C. Confirmation was undertaken using a DB1701 column, 60 m lenght, 0.25 mm diameter. Here the GC oven was held at 60 °C for 2 min, then ramped to 200 °C at 20 °C min^{-1}, held at 200 °C for 20 min, then ramped to 280 °C at 5 °C min^{-1} and finally held at 280 °C for 16 min. Inlet and detector conditions were as above. Detection limit was 1 μg kg^{-1} (wet weight).

Quality assurance

The laboratory involved in the chlordecone analysis is ISO 17025-compliant (ISO 17025 is the equivalent of ISO 9000 for calibration and testing laboratories) and have been accredited by the French Accreditation Committee (COFRAC). Quality assurance testing included the analysis of blanks, assessment of recoveries and reproducibilities and accuracy of the data relative to standard reference materials. The laboratory is also approved for official control by the French Ministry of the sustainable Environment (MEDD): approval No. 8, organic contaminant for brackish and salt waters including organochlorine compounds. The performances for recovery of chlordecone were >80%.

3 Results

Results are expressed as micrograms of chlordecone per kilogram of flesh (wet weight). Fourteen of the 99 samples analyzed were found to contain chlordecone. Positive results are presented in Table 2.

The highest chlordecone concentrations were detected in wild red tilapia samples collected in the Lézarde River, which runs through banana plantations before flowing into the Bay of Fort-de-France. Chlordecone concentrations of 386 and 196 μg kg^{-1} of flesh were detected in 2 sample composites of wild red tilapia collected at the Lézarde River sampling site. Both farmed-raised tilapia and prawn from freshwater ponds located in the Northeast part of the island were also found to be contaminated with chlordecone. Chlordecone levels were 132 μg kg^{-1}and 23 μg kg^{-1} in tilapia and prawn, respectively.

Certain beach-seined marine fish and shellfish species were also contaminated with chlordecone: levels ranged from 3 to 7 μg kg^{-1} in 4 of 8 anchovy and mackerel scad samples collected at the Sainte Marie sampling site, in the Northeastern section of the island. Chlordecone concentrations measured in 4 spiny lobster and 2 surgeon fish samples from various sampling sites were the following: 10, 13, 20, and 31 μg kg^{-1} in spiny lobster samples, 4.1 and 1.2 μg kg^{-1} in surgeon fish samples.

Table 3. Concentrations in organochlorine pesticides in the aquatic fauna of various regions worldwide (μg kg^{-1} wet weight).

Location, date	Species	Predominant organochlorines detected and mean concentration range			Reference
Martinique 2002	wild tilapia farmed tilapia	chlordecone: **196-386** chlordecone: **132**	β HCH: **Ø**		This publication
Virginia, USA 1977	freshwater fish	chlordecone: **2500**			Nichols (1990) in ATSDR (1995)
	phytoplankton	chlordecone: **1300**			
	zooplankton	chlordecone: **4800**			
	migrating fish	chlordecone: **400**			
	benthic molluscs	chlordecone: **1500**			
Hawaii, 2001	various fish species	dieldrin: **50-1700**	*cis*-chlordane: **17-460**	p, p'-DDE: **4-180**	Brasher and Wolff (2003)
Georgia, USA, 1997	Silver sea trout *(Cynoscion nothus)*	γHCH : **0.8**	*cis*-chlordane: **7.08**	p, p'-DDE: **1.03**	Loganathan et al. (2001)
Jamaica, 1983	Shrimps	dieldrin: **0.427-5.59**	γHCH : **2.90**	DDE: **0.344-14.57**	Mansingh et al. (2000)
Lake Ontario, USA, 1992	Coho salmon *(Oncorhynchus kisutch)*	mirex: **240**			Makarevicz et al. (2003)
Jamaica, 1992	Aquatic fauna	dieldrin: **Ø**	α-endosulfan: **68.5-110.6**	β-endosulfan: **4.31-53.7**	Robinson and Mansingh (1999)
Mexico, 1996	Shrimp	δ HCH: **48.8-127**	lindane: **Ø-132**	DDE: **19-29**	Osuna-Flores and Riva (2001)
Italy, 1997	Mackerel *(Scomber scombrus)*	dieldrin: **0.63-1.17**	γ HCH: **0.20-0.83**	p, p'-DDE: **15.32-25.18**	Stefanelli et al. (2002)
India, 1999	various fish species	γ HCH: **1-3**	total HCH: **1-6**	DDT: **13-55**	Sarkar et al. (2003)
Egypt, 2001	Mullet *(Mugil* spp.)	dieldrine: **5.3-15.1**	lindane: **5.9-14.3**	p, p'-DDE: **9.3-33.7**	El Nemr and Abd-Allah (2003)
Brazil, 1999	various fish species	p, p'-DDE: **Ø-19.7**			Da Silva et al. (2003)
Taiwan, 1999	various fish species	p,p'-DDE: **95.38-99.11**	p, p'-DDD: **80.01-488.33**		Yuan et al. (2004)

Ø: below detection limit

HCH: hexachlorocyclohexane

DDE and DDD: DDT metabolites

As opposed to animals that were collected in enclosed or coastal areas, or near estuaries, fish collected offshore (mackerel, parrot fish, and farmed red drum) had no detectable chlordecone (data < detection limit).

4 Discussion

Analyses revealed the presence of chlordecone in a number of aquatic animal species. Half of the 14 species studied (5 of 9 fish species, 2 of 2 shellfish species) were contaminated. Chlordecone was also detected in coastal and coral reef fish species, such as the surgeon-fish, and in spiny lobster. No chlordecone was, however, found in filter-feeding bivalves or fish collected offshore, such as the red drum.

This study shows that the highest contamination levels are found in wild and farm-raised tilapia (carnivores) and crustaceans (detritivores). Great variations are observed among the different species. The highest levels are detected in wild tilapia (386 and 196 μg kg^{-1}), farm-raised tilapia (132 μg kg^{-1}) and, although to a lesser extent, freshwater prawn (23 μg kg^{-1}) and spiny lobster (31, 20, and 13 μg kg^{-1}). Contamination by pesticides was recently observed for other organochlorines detected in fish species collected in Egypt. Ten organochlorines (HCB, lindane, heptachlor, heptachlor epoxide, dieldrin, p,p'-DDT, p,p'-DDE, p,p'-DDD, toxaphene, chlordane) were detected in the following fish species (from the most to the least contaminated): the mullet (*Mugil cephalus*), sea bream (*Sparus auratus*), bogue (*Boops boops*), and sand sole (*Pegusa lascaris*) (El Nemr and Abd-Allah 2004). In the Adriatic Sea, the highest concentration in mackerel (*Scomber scombrus*) was 25.18 μg DDE kg^{-1} wet weight (Stefanelli et al. 2004). In a study conducted in Tanzania, reported organochlorine levels detected in a tilapia species (*Tilapia jipe*) were 20.4 μg of total DDT (p,p'-DDT, p,p'-DDE, and p,p'-DDD) per kg fresh weight

(Mwevura et al. 2002). Variation in the levels of organochlorine pesticide residues found in aquatic species is multifactorial. The nature and solubility of these molecules, the degree and duration of exposure, the type of diet, as well as the animal's lipid load are influential factors. The type of diet and lipid load play an especially important role in the accumulation of organochlorine pesticides. Due to its poor solubility in water and strong affinity for organic matter (log K_{ow} = 4.5), chlordecone is preferentially found in sediment rather than in the water column, and as such, mostly accumulates in detritivorous and carnivorous species. Studies conducted in marine fish species have reported BCF-values of 16 000 in the fathead minnow (WHO-IPCS 1984). Zhou et al. (1999) studied contamination of tilapia with chlorinated compounds in their laboratory. They showed that ingestion of sediment and/or contact with chlorinated particles were the predominant pathways of sediment-sorbed accumulation in tilapia.

Our results suggest that contamination is generally less important in herbivorous fish species. Chlordecone levels detected in the surgeon fish, which is a strict herbivore, were very low (1.2–4.1 μg kg^{-1}). A study of contamination of the common carp (*Cyprinus carpio),* also an herbivor, with lindane, alpha-chlordane, gamma-chlordane, and 4,4'-DDE, in its natural habitat was conducted in Kansas by Eaton and Lydy. These authors reported that the mean total organochlorine concentration of all fish was 9.32 ± 3.07 μg kg^{-1} (Eaton and Lydy 2000).

In Martinique, contamination of animals in farm ponds may be due to the presence of contaminated clay soils and suspended particulate matter present in water from an upstream source, or else from the contaminated microflora and microfauna present in fish feeds. Analyses done in 2001 by the DSDS on soil samples from banana plantations that had been treated for long periods of time, revealed the presence of chlordecone in all of them. Levels reaching 5000 μg kg^{-1} of chlordecone were measured in soils of the Gradis farm, located near the River Rouge, in August 1999, and levels up to 13 000 μg kg^{-1} dry weight were recorded in hydromorphic soils, in a parcel of land along the north Atlantic coast (Bellec and Godard 2002). These contaminated soils constitute decades worth of chlordecone "supplies". River water analyses carried out by the DSDS (DSDS 2001) between June 1999 and June 2001 revealed an average chlordecone concentration of 0.3 μg L^{-1} in the Capot River. In some samples, values even reached 1 μg L^{-1}. A recent study of contamination of water in estuaries of Martinique showed that chlordecone was mainly found in suspended particulate matter and sediment. It was found in sediment samples of the Lorrain (1.44 μg kg^{-1}), François (4.38 μg kg^{-1}), and Lézarde (9.31 μg kg^{-1}) Rivers, as well as in suspended particulate matter samples collected in the plume of the François (4.57 μg kg^{-1}), Galion (3.52 μg kg^{-1}), Lézarde (9.45 μg kg^{-1}), and Cacao (5.22 μg kg^{-1}) Rivers (Bocquené and Franco 2005).

It is interesting to note that farmed red drum reared in sea cages and fed with imported synthetic feeds were not contaminated. This observation supports the strong probability that a trophic transfer of organochlorine contaminants occurs in the environment.

Organochlorines have also been massively used for long periods of time in many other regions around the world. Many studies now report on the ensuing contamination of aquatic systems. Some data regarding concentrations of various organochlorines in aquatic species are presented in Table 3.

The presence of organochlorine insecticides in the aquatic fauna is ubiquitous and can even be found in isolated regions. For example, Sarkar et al. (2003) reported the presence of γ HCH and DDT in two freshwater fish species, the putitor mahseer (*Tor putitora)* and the snow trout (*Schizothorax richardsonii*), in the Himalaya.

The highest levels of chlordecone in the aquatic fauna were detected in the State of Virginia, USA, near a chlordecone production plant (Nichols 1990 in Agency for Toxic Substances and Disease Registry: ATSDR 1995). In 1977, two years after the plant had closed, chlordecone concentration was monitored in various species living in the James River. Chlordecone levels reached 2500 μg kg^{-1}in freshwater fish, and the highest levels, i.e., 4800 μg kg^{-1}, were recorded in zooplankton. In this particular case, contamination of the James River and neighbouring streams was not due to diffuse pollution related to an extensive use of this molecule in neighbouring cultures but rather to industrial negligence. Chlordecone levels measured in Martinique are among the highest worldwide after those recorded in Virginia. They are much higher than the following average concentrations of total DDT (DDT and metabolites, p,p'- DDE and p,p'- DDD) measured along the European coasts: 2.42, 8.8, and 11.32 μg kg^{-1} wet weight in the common sole *(Solea solea*), mackerel (*Scomber scombrus*), and estuarian flounder (*Platichtys flesus*), respectively, along the Atlantic coast (Cossa et al. 1990), 25.00 μg kg^{-1} in mackerel from the Adriatic Sea (Stefanelli et al. 2004), and 38.4 ± 17 g kg^{-1} wet weight in flounder from Garston in the inner Mersey Estuary in UK (Leah et al. 1997).

Given the biogeochemical behaviour of these contaminants, the disappearance of these molecules from the environment may take centuries (Makarewicz 2003).

Consumption of fish in Martinique, which reaches 16 000 tons a year, is one of the highest in the world in proportion to the number of inhabitants. Local production (fishing and aquaculture), however, accounts for approximately 6000 tons (INSEE 2002). Fresh fish, which mainly comes from traditional fishing, is distributed in a number of harbours.

Only part of the population of Martinique consumes fish distributed by coastal fishing villages. Therefore significant exposure to the pesticides that have been used in banana plantations through consumption of aquatic organisms only concerns that part of the population. Most of the fish distributed by these coastal fishing communities is, however, caught offshore and unlikely to be contaminated. Exposure to chlordecone is therefore rather associated with consumption of organisms from enclosed bays, rivers, and farm ponds.

A correlation between lipid load and concentration in organochlorines is usually observed in fish and molluscs (Mehdaoui et al. 2000; Das et al. 2002). Further investigation in species with a higher lipid load, such as Clupeidae (herring, sprat, sardine) or tunas (although these are rather pelagic species that live away from contaminated coastal areas) is thus warranted. The toxicity reference value, the maximum concentration of organochlorine compound residues tolerated in food, determined by the Institut Français National de Veille Sanitaire (INVS) is equivalent to that established by American Authorities, i.e., 0.5 μg kg^{-1} d^{-1}, or 30 μg d^{-1} for a 60 kg adult (Bonvalot and Dor 2004). In order to reach the acceptable daily intake (ADI), an individual must consume 77 g of wild tilapia, 227 g of farmed tilapia, 1 kg of spiny lobster, or 1.3 kg of prawn from contaminated areas every day.

Chlordecone concentrations of 0.3 mg kg^{-1} of edible fish tissue and 0.4 mg kg^{-1} of crab flesh were determined by the US Food and Drug Administration (FDA) as the intervention limit, which gives some leeway before consumption of these aquatic products is prohibited.

Organochlorines also bioaccumulate in humans. Organochlorine pesticides were detected in fatty tissue samples of Martinique residents (Martin 1973). Mirex, a compound related to chlordecone, also used as an insecticide, was found in the breast milk of Canadian women whose diet essentially consisted of fish from Lake Ontario (Makarewicz et al. 2002). Organochlorine pesticides were also detected in the serum of Japanese individuals. Average concentration of beta-hexachlorocyclohexane (βHCH), hexachlorobenzene (HCB), and total DDT (p,p'-DDE and p,p'-DDT) were respectively, 0.50 ng ml^{-1}, 0.20 ng ml^{-1}, and 5 ng ml^{-1} (Hanaoka 2002).

It is important to keep in mind that aquatic organisms are not the only source of intake for organochlorines in Martinique but one of the major routes of exposure. The above ADI values should therefore also take into account intake levels associated with the consumption of water and other contaminated foods, such as root vegetables grown on lands contaminated by banana plantation run-offs.

5 Conclusion

Although the use of chlordecone has been prohibited for over 10 years, high levels of chlordecone are still detectable in a number of aquatic species of Martinique (up to 386 μg kg^{-1} chlordecone in locally consumed tilapia). Measured values are impressively high compared to those reported for other regions around the world. Detected concentrations in marine organisms are below the tolerated limits established by authorities; however, the impact of other sources of exposure, namely, contaminated water and root vegetables, remains to be investigated.

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