Pollution by organochlorine pesticides in Navachiste-Macapule, Sinaloa, Mexico

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Abstract The lagoon system of Navachiste-Macapule is located in northern Mexico, in the state of Sinaloa, with an area of 24,000 ha. The main economic activity in the area is agriculture, and the lagoon lies next to the irrigation district ID-063 which covers 116,615 ha. The purpose of this study is to evaluate the levels of pollution generated by organochlorine pesticides (OC) in the surface sediments of the lagoon and in the agricultural drains of the ID-063 that are supposedly transported into the system as a result of agricultural activities and runoff from adjacent land. For this, between 2006 and 2007, 45 surface sediment samples were collected (warm dry, rainy and cold dry) from 15 sampling sites, during the three climatic seasons. Of these, eight were located inside the lagoon in marine conditions (salinity >31 PSU) and seven in the agricultural drains of the ID-063 in freshwater conditions (salinity <5 PSU). The average concentration of the OC in the sediments was 44.75 ng g^{-1} , among which the group of the alicyclic compounds presented the greatest concentrations. The aver-

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age value of the total organic carbon (TOC) in the sediments of the system was 0.90%. The sediments collected inside the lagoon had an average OC concentration of 18.97 ng g^{-1} , and the predominant type of sediment was fine to very fine sand. The average OC concentration in the sediments collected in the agricultural drains was 75.69 ng g^{-1} , where fine sediments (silt) were predominant. The presence of methoxychlor, endrin and heptachlor suggested that these compounds were continuously used in the system, even though their use is forbidden in Mexico.

Keywords Organochlorine pesticides **·** Coastal lagoon **·** Total organic carbon **·** Sediment **·** Mexico

Introduction

Tropical coastal lagoons are ecologically relevant because they provide feeding grounds, as well as reproductive and protection areas for many species, some of which are endangered. They are also important from the economic point of view, mainly for fisheries and aquaculture (Flores-Verdugo [1989\)](#page-9-0). However, increases in human settlements and agricultural wastes such as pesticides and the direct discharge of sewage into the lagoons have generated severe pollution problems that jeopardise the ecology and biodiversity of these areas (Botello et al[.](#page-9-0) [2000\)](#page-9-0).

The coast of the state of Sinaloa has 16 coastal lagoons where commercial fisheries and aquaculture farms represent important economic activities. Most of these lagoons are surrounded by irrigation districts in which highly technified agriculture uses large amounts of agrochemicals, including pesticides (González-Farias [2003;](#page-9-0) González-Farias et al. [2006](#page-9-0)).

Pesticides occupy a unique position among the chemicals that man encounters daily, as they are deliberately added to the environment with the purpose of killing or injuring some life form (Osuna-Flores and Riva [2002](#page-10-0)). In Mexico, organochlorine pesticides (OC) were used mainly to control soil and crop pests in agricultural fields, as well as the vectors that transmit diseases such as dengue and malaria. At present, Mexico has forbidden the use of OC, except for endosulphan, that is used in agriculture, DDT and lindane ($γ$ -HCH) that are restricted to health campaigns (CICOPLAFES[T](#page-9-0) [2004\)](#page-9-0). However, great amounts of OC have been used and have dispersed in previous years. The physicochemical properties of the OC, including low water solubility, moderate vapour pressure, high octanol-water partition coefficient, persistence in the environment and liposolubility make them suitable for longrange transportation (Albert and Loer[a](#page-9-0) [2005\)](#page-9-0). In aquatic environments, OC are removed from the water column and adsorbed onto particulate matter in response to their high affinity for organic matter, after which the particles tend to settle and accumulate in the sediment. These pollutants may also accumulate in sediment dwelling organisms and be transferred to higher trophic levels through the food chain (Lee et al[.](#page-10-0) [2001;](#page-10-0) Leyva-Cardoso et al[.](#page-10-0) [2003](#page-10-0); Singh et al[.](#page-10-0) [2005\)](#page-10-0).

The main purpose of this study was to determine the current state of OC pollution in this tropical coastal lagoon that lies adjacent to a highly technified irrigation district in northwestern Mexico. It is important to mention that these pollutants have not been previously reported for sediments from Navachiste, Macapule, Sinaloa.

Study area

The Navachiste-Macapule lagoon is a complex coastal system with an approximate area of 24,000 ha. It is located in the northern area of the Mexican state of Sinaloa, in the municipality of Guasave, at 25.4◦–25.7◦ N and 108.85◦–108.55◦ W (Fig. [1\)](#page-2-0). According to Köppen's modified classification, the climate in this region is type $BW(h'),$ semi-arid with Summer and Winter rains (INEGI [2000;](#page-10-0) Hernández-Cornejo et al. [2005](#page-9-0)).

Locally, this lagoon has a great ecological and economic importance, as it supports a variety of oyster, clam, mullet, mojarra, puffer, snapper, jewfish and snook fisheries, as well as intensive shrimp aquaculture activities (Orduña-Rojas and Longoria-Espinoza [2006\)](#page-10-0).

The main sources of pollution for the Navachiste-Macapule lagoon are the agricultural drains from Irrigation District #063 (ID-063) which transport fertilisers and pesticides from the fields, as well as the drains from the shrimps farms located along the margins of the lagoon (Hernández-Cornejo et al. [2005](#page-9-0)). The ID-063 is one of the most important agricultural regions in Mexico with ∼116,615 ha of agricultural land, of which 107,036 ha are highly technified, artificially irrigated, and contains great amounts of agrochemicals that are applied each agricultural cycle (CNA [2007](#page-9-0); González-Farias [2003](#page-9-0); González-Farias et al. [2006\)](#page-9-0).

Materials and methods

Sample collection

A total of 45 sediment samples were collected from 15 sampling sites (Fig. [1\)](#page-2-0), together with salinity and temperature data. The Navachiste-Macapule system was geographically divided into the lagoon (stations 1 to 8) and the agricultural drains (stations 9 to 15), with eight stations inside the lagoon under marine conditions (salinity >31) PSU) and seven stations in the agricultural drains of the ID-063 with freshwater conditions (salinity <5 PSU). Salinity was recorded in situ with a 1% accuracy refractometer (American Optical) using the Practical Salinity Scale (PSU) and the temperature was measured directly with a multiparametric equipment (HORIBA).

Sediment samples were collected with a stainless steel 5 kg capacity van Veen grab. Aliquots

Fig. 1 Study area and sampling points

of approximately 200 g wet weight were obtained from the center of the sample with a solvent cleaned spatula, and placed in clean glass jars. After collection, all samples were transported on ice and kept deep frozen for later chemical analysis.

Sampling was carried out during the three climatic seasons: warm dry (June 2006), rainy (August 2006) and cold dry (March 2007).

Chemical analyses

The analytical procedures followed for the collection, extraction, purification and quantification of the OC in the sediments were those recommended by the UNEP/IAEA [\(1982](#page-10-0)). For each sample, 100 g of sediment were dried at 35◦C for 72 h and sieved (250 μ m). Aliquots of 3 g dry weight (dw) were introduced into previously acetone-extracted cellulose thimbles (Whatman), and were Soxhlet-extracted with 250 ml of hexane (pesticide grade) for 8 h. A spiked blank was runned each five samples. The extract was concentrated in a rotovapor (30◦C, 40 psi), and purified by adsorption chromatography in a glass column (3 cm id \times 30 cm length) that was packed with fiberglass, 13 g of Florisil (deactivated with

1.25% tri-distilled water) and a 0.5-cm layer of anhydrous sodium sulphate on top. The column was eluted with 60 ml of hexane (fraction 1), followed by 50 ml of hexane/ethyl ether (9:1) and then 20 ml of hexane:ethyl ether (8:2; fraction 2). All solvents were pesticide grade. The elution volumes were concentrated to 0.5 ml, and 1 μl aliquots were injected in a gas chromatograph (GC; Hewlett Packard 5890 series II). The GC was equipped with an electron capture detector $(ECD⁶³Ni)$ and a VF5 pesticides capillary column of fused silica, with a 5% methyl-phenylsilicon phase (30 m, 0.25 mm id and 0.25 μ m layer thickness). Helium was used as the carrier gas, and argon with 5% methane was the auxiliary gas. The conditions of the GC were: injector temperature 250◦C, detector temperature 300◦C, initial temperature 110◦C, initial time 1 min, after which the temperature was raised to 190◦C at a rate of 20◦C min[−]¹ and maintained for 1 min (ramp 1). The temperature was then raised to 243◦C at a rate of 2◦C min[−]¹ and kept at 243◦C for 1 min (ramp 2). The temperature was again raised to 300◦C at a rate of 30◦C min[−]¹ and kept at 300◦C. The final time was 36.40 min, and the mode of injection was split-splitless. The internal standard 2,4,5,6-tetrachloro-m-xylene was added prior to the GC/ECD analysis. The final concentrations were determined using an external standard of 18 organochlorine compounds: α-HCH, β-HCH, γ-HCH, δ-HCH, 4,4 -DDT, 4,4 -DDE, 4,4 -DDD, methoxychlor, aldrin, dieldrin, endrin, endrin aldehyde, endrin ketone, heptachlor, heptachlor epoxide, α-endosulphan, β-endosulphan and endosulphan sulphate (SUPELCO Inc.). The detection limit values for each compound were: 0.26, 0.33, 0.26, 0.33, 0.51, 0.39, 0.24, 0.42, 0.46, 0.25, 0.11, 0.25, 0.22, 0.35, 0.35, 0.16, 0.24 and 0.67 ng g⁻¹, respectively.

Granulometric analyses and organic matter determination

The grain size analysis of the sediment samples was performed using the combined dry sieve and pipette method (Fol[k](#page-9-0) [1974\)](#page-9-0), and the percentage of organic matter was determined by titration using ferrous sulphate (Gaudette et al[.](#page-9-0) [1974\)](#page-9-0).

Results

The mean values of salinity and temperature recorded during the three seasons in the lagoon system are presented in Table 1.

The predominant sediments were fine sands in the lagoon (stations 1 to 8), and silt in the agricultural drains (stations 9 to 15; Fig. 2).

The total organic carbon (% TOC) values for the sediment samples are shown in Fig. 3. The mean percentage of TOC in the lagoon sediments (stations 1 to 8) was $0.92 \pm 0.46\%$ TOC dw, while that in the agricultural drains (stations 9 to 15) was $0.87 \pm 0.28\%$ TOC dw.

Table 1 Salinity and temperature mean values

Season	Stations	Salinity	Temperature
		(PSU)	$(^{\circ}C)$
Warm dry	Lagoon	38	30.6
Warm dry	Agricultural drains		25.8
Rainy	Lagoon	37.6	32.5
Rainy	Agricultural drains	2.1	31.4
Cold dry	Lagoon	35.5	20
Cold dry	Agricultural drains	1.7	18.2

(–) value was not recorded

Fig. 2 Types of sediment and stations in Navachiste-Macapule lagoon (stations inside)

OC in sediments

Warm dry season (June 2006)

The total average value of the OC in the sediments during the warm dry season (June 2006) in the lagoon system was 51.01 ng g^{-1} dw. The average value of the OC in stations 1 to 8 was 19.67 ng g^{-1} dw, whereas in stations 9 to 15 in the agricultural drains, it was 86.83 ng g[−]¹ dw. The group of alicyclics reported the greatest average concentration of 26.17 ng g^{-1} dw, with the greatest value of 49.23 ng g^{-1} dw in the agricultural drains (stations 9 to 15), and the lowest value of 5.99 ng g^{-1} dw in the lagoon (stations 1 to 8). δ-HCH was the compound with the highest concentration (70.23 ng g^{-1} dw), recorded in the agricultural drains (stations 9 to 15). The general behaviour of the chemical families in the system was as follows: alicyclics (26.17 ng g^{-1} dw) > cyclodienes (13.74 ng g^{-1} dw) > aromatics (12.87 ng g^{-1} dw). α -HCH and methoxychlor were

Fig. 3 Percentage of TOC in sediments

Table 2 Concentrations of OC ($n g g^{-1}$ dw) recorded in sediment samples collected in the Navachiste-Macapule system

the compounds with the greatest detection frequency in the system during this season (Table [2\)](#page-4-0).

Rainy season (August 2006)

The greatest concentrations of OC in the sediment samples were recorded during the rainy season (August 2006). The total average value for the OC in the lagoon system sediments was 64.97 ng g^{-1} dw. For stations 1 to 8, the average value of the OC was 24.87 ng g^{-1} dw, whereas in the agricultural drains (stations 9 to 15) the average value was 110.79 ng g^{-1} dw. The group of alicyclics presented the greatest average in the system (41.76 ng g^{-1} dw), with the maximum value of 80.62 ng g^{-1} dw in the agricultural drains (stations 9 to 15), and the minimum of 7.77 ng g⁻¹ dw in the lagoon (stations 1 to 8). The general behaviour of the chemical families in the system was as follows: alicyclics (41.76 ng g^{-1} dw) > aromatics (14.46 ng g^{-1} dw) > cyclodienes (9.70 ng g^{-1} dw). β-HCH was the compound with the highest concentration of 553.25 ng g^{-1} dw, followed by methoxychlor with 31.87 ng g^{-1} (Table [2\)](#page-4-0). α-HCH, methoxychlor and endrin ketone were the compounds with the highest detection frequencies in the system (Table [2\)](#page-4-0).

Cold dry season (March 2007)

The total average value of the sedimentary OC recorded during the cold dry season (March 2007) in the lagoon system was 16.39 ng g^{-1} dw. The average OC value in stations 1 to 8 was 12.37 ng g^{-1} dw, and that in the stations 9 to 15 was 21.75 ng g^{-1} dw. The group of alicyclics had an average concentration of 6.36 ng g^{-1} dw. The higher value was recorded in the lagoon (stations 1 to 8) with 6.64 ng g^{-1} dw, and was followed by a value of 5.99 ng g^{-1} dw in the agricultural drains (stations 9 to 15). The general behaviour of the chemical families in the system was as follows: alicyclics (6.36 ng g^{-1} dw) > aromatics $(5.84 \text{ ng g}^{-1} \text{ dw})$ > cyclodienes $(4.17 \text{ ng g}^{-1} \text{ dw})$. Methoxychlor was the compound with the highest value (23.61 ng g⁻¹ dw), and α- HCH, β-HCH, γ-HCH and δ-HCH were the compounds with the highest detection frequencies (Table [2\)](#page-4-0).

The average values of the OC (ng g^{-1} dw) in the sediments, recorded from June 2006 to March 2007, were: 44.75 ng g^{-1} dw in the Navachiste-Macapule system, 18.97 ng g^{-1} dw in the lagoon (stations 1 to 8), and 75.69 ng g^{-1} dw in the agricultural drains (stations 9 to 15). The highest concentrations of OC were recorded in the agricultural drains during the rainy season. Considering the three chemical OC families, the alicyclics presented the highest average in the system with 25.77 ng g⁻¹ dw, an average of 7.10 ng g⁻¹ dw in the lagoon (stations 1 to 8) and an average of 47.25 ng g^{-1} dw in the agricultural drains (stations 9 to 15). These were followed by the aromatic and cyclodiene compounds.

Prediction of toxicological and biological effects

In order to predict the potential toxicological and biological effects of the OC pollution in Navachiste-Macapule sediments, our data were compared with the NOA[A](#page-10-0) [\(2004](#page-10-0)) sediment quality guidelines (SQGs) and those of the Canadian Council of Ministers of the Environment (CCM[E](#page-9-0) [2002\)](#page-9-0). These guidelines specify the "Effects Range Low" (ERL), the "Threshold Effects Level" (TEL), the "Effects Range Median" (ERM) and the "Probable Effects Level" (PEL). The ERL and TEL represent the chemical concentration below which an adverse effect is rarely observed. The ERM and PEL represent the concentration above which an adverse effect frequently occurs. The γ -HCH in the Navachiste-Macapule system exceeded the TEL and PEL. Respect to dieldrin, the average concentration at each site lagoon stations were well above the ERL. Endrin average concentrations at each site in the agricultural drains exceeded the TEL. The total DDT average concentrations in the Navachiste-Macapule stations were above the ERL.

Discussion

The highest salinity and temperature values were recorded in the lagoon (stations 1 to 8) during the warm dry season (June) and the rainy season (August), when high temperatures favored evaporation and increased salinity. It is important to mention that evaporation is greater than rainfall in the Gulf of California (Delgadillo-Hinojosa et al. [2001\)](#page-9-0). The salinity in the agricultural drains (stations 9 to 15) was \lt 5 PSU, as the freshwater carries agricultural wastes from the ID-063.

The presence of fine and very fine sand in the lagoon (stations 1 to 8) responds to the high energy of the tidal currents (Escobedo-Urias et al. [2006\)](#page-9-0). The silt and clay sediments predominant in the agricultural drains (stations 9 to 15) are the result of erosion in the ∼116,615 ha of the ID-063.

The distribution of TOC in the sediments of the Navachiste-Macapule system is influenced primarily by the importation (from allochthonous sources), exportation (from autochthonous sources), dilution, flocculation, sedimentation and degradation (González-Farias et al. [2006\)](#page-9-0) of the sediments. The highest mean percentage of TOC in a sediment sample was 1.67% TOC dw (Fig. [3\)](#page-3-0), that was recorded in station 7 inside the lagoon and is a reflection of the organic matter that enters the lagoon via agricultural drains and settles aided by the lagoon morphology. The lower TOC recorded in the sediments of the Navachiste-Macapule system is related to the high energy of the tidal currents (Escobedo-Urias et al. [2006](#page-9-0)) that prevents organic matter from settling and enhances dilution with salt water, as well as exportation to the adjacent coastal area (González-

Table 3 OC

during the period 2006–2007

Farias et al. [2006\)](#page-9-0). Another important factor in the lower organic matter content in the Navachiste-Macapule sediments is the change in land use or agricultural management in the adjacent ID-063. Changes may affect the degree of soil disturbance, as well as the nature and amount of organic inputs, which may result in a decrease in TOC in the Navachiste-Macapule system (Haynes et al[.](#page-9-0) [2003;](#page-9-0) McCarthy et al[.](#page-10-0) [2008;](#page-10-0) Wang et al[.](#page-10-0) [2008\)](#page-10-0). It is a well established fact that converting natural forests and grasslands into agricultural fields generally leads to a decline in TOC and that different tillage systems and crop rotations may also decrease it (Apezteguía et al. [2009\)](#page-9-0).

Comparing the maximum concentrations of organic carbon recorded in this study (1.67%) with the available information for other similar environments in Sinaloa, Páez-Osuna et al. [\(1992](#page-10-0)) reported 0.8% for the Altata lagoon and Ruíz-Fernández et al. [\(2007\)](#page-10-0) recorded 3% for the Culiacán River estuary and 0.5% for the Altata-Ensenada del Pabellón lagoon.

The summary of the concentrations of each compound in the system during the period 2006– 2007 is presented in Table 3. The HCH isomers were the compounds with greater concentrations (β-HCH, δ-HCH, α-HCH and γ-HCH) suggesting a historical pollution generated by the use of technical HCH mixture. β-HCH is also the most

concentrations (ng g^{-1}) in the lagoon system of Navachiste-Macapule $Commoned$ Minimum Maximum Σ Detection frequency (%) α-HCH 0.32 41.26 210.23 93.18 β -HCH 0.34 533.25 611.20 86.36 γ -HCH 0.35 7.65 48.95 84.09 δ-HCH 0.30 70.23 237.78 81.82 DDT 0.59 1.78 7.39 15.91 DDE 0.40 2.80 10.68 27.27 DDD 0.26 0.59 2.46 11.36 Methoxychlor 0.49 42.08 444.02 81.82 Aldrin 0.58 0.58 0.58 2.27 Dieldrin 0.25 2.50 15.75 45.45 Endrin 0.35 26.99 152.91 75.00 Endrin aldehyde 0.26 1.38 8.30 27.27 Endrin ketone 0.28 9.81 75.69 84.09 Heptachlor 0.37 17.92 56.69 34.09 Heptachlor epoxide 0.39 0.61 1.91 9.09 α-Endosulphan 0.17 6.17 21.14 47.73 β-Endosulphan 0.24 2.53 8.92 27.27 Endosulphan sulphate 0.71 9.41 54.56 47.73

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persistent and stable isomer in face of microbial decomposition in soils and sediments.

With regard to DDT, the DDT–DDE ratio suggests a historical pollution, through its long degradation time.

Methoxychlor was found in high concentrations in the sediments, suggesting its constant use in the system.

Endrin had the highest concentrations, followed by endrin ketone, dieldrin, endrin aldehyde and finally aldrin, indicating a recent application.

With respect to the concentrations of heptachlor, this compound had the highest concentration compared with its epoxide, suggesting a continued use in the system.

Endosulphan sulphate recorded the highest concentration, followed by α-endosulphan and finally β-endosulphan. These values suggest a predominance of degraded forms, resulting from the oxidation of α-endosulphan.

Methoxychlor, endrin and heptachlor occurrence suggest a presently continuous usage in spite of their prohibition by the government laws.

The highest levels of OC were recorded in the agricultural drains that transport agricultural wastes from the ID-063. This suggests that the distribution of the OC in the system is controlled by the proximity and importance of the pollution source.

The highest concentrations of each residue reported in this study were compared with the maximum concentrations in the sediments of other coastal lagoons of Sinaloa and Sonora (Table [4\)](#page-7-0). In general, our concentrations are similar to those previously reported, except for endrin, endrin aldehyde, α-HCH and β-HCH (Carvalho et al. [1996,](#page-9-0) [2002](#page-9-0); Galindo et al. [1999a,](#page-9-0) b; González-Farias et al. [2002,](#page-9-0) [2006](#page-9-0); Osuna-Flores and Riva [2002;](#page-10-0) Rosales et al. [1985\)](#page-10-0).

Pollution in the sediments of the Navachiste-Macapule system exceeded the maximum values suggested by NOAA and CCME to avoid adverse effects on aquatic life. It is important to mention that Páez-Osuna et al. [\(2002](#page-10-0)) analysed pollutants in mangrove oysters of the Pacific coast of Mexico, and recorded the highest organochlorine concentrations in the Navachiste lagoon.

A linear correlation analysis was used to examine relationships between OC and total organic carbon and sediment size fractions (sand, silt, clay, silt-clay). No significant relationship ($p < 0.05$) was found. However, many authors have reported a scarce correlation between TOC and concentrations of organic pollutants (González-Farias et al[.](#page-9-0) [2006;](#page-9-0) Secco et al[.](#page-10-0) [2005](#page-10-0)). Edgar et al[.](#page-9-0) [\(2003](#page-9-0)) recorded no significant correlation between the concentration of total organic pollutants and the percentage of TOC, and suggested that the nature of the organic carbon influences its affinity to the sediment, as well as its mineralogical composition and sediment size fraction, rather than the absolute amount of TOC present.

Conclusions

This study indicated the presence of OC in the Navachiste-Macapule system, Sinaloa, Mexico. Pesticides of the HCH group, as well as methoxychlor, endrin and the endosulphan group were frequently present in the system.

Methoxychlor, endrin and heptachlor concentrations suggest that some OC are still being used illegally.

The highest concentrations of pesticides in the system appeared during the rainy season due to runoff from agricultural fields. It is suggested that the OC enter the coastal ecosystem via the agricultural drains, then, it is proposed that OC occurrence is controlled by the proximity to those drains.

Considering the dynamics of the system and the low TOC in the sediments, it is concluded that OC may well be exported to the open sea. Consequently, a constant monitoring of the area is recommended.

Finally, several stations in the Navachiste-Macapule system exceeded the level above which aquatic life is affected. It is, therefore, recommended that management plans be designed and implemented in the area, including educational programmes and specific regulations on pesticide handling.

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