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ORGANOCHLORINE PESTICIDES IN WATER, SEDIMENT AND FISH FROM THE NILE RIVER AND MANZALA LAKE IN EGYPT

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To assess the risks of organochlorine pesticides discharged into the hydrospheric environment of Egypt, river and lake water, drinking water, suspended solids, sediments and fish were collected during **1993-1994** from the Nile River and Manzala Lake in Egypt and were transported to Japan for chemical analysis. Among different organochlorine pesticides analyzed, p,p'-DDE was the most predominant in fish **(7.6** to **67** np/g wet wt.), sediments **(3.2** to **432** ng/g dry **wt.)** and suspended solids **(5.3** to **138** pg/L). However, in the dissolved phase of water samples HCH compounds predominated (a-HCH, **71** to **2,815** pg/L). Concentrations **of** organochlorine pesticides, except chlordane, were higher in Manzala Lake than in the River Nile. Concentrations of organochlorine pesticides in fish corresponded with those in sediments from each location. Comparison of organochlorine concentrations in Nile River water with those reported in earlier studies suggested a decrease in concentrations during the last decade. However, concentrations of p, p' -DDE has increased in fish. It appears that the release of this metabolite from contaminated sediment is the major **source** of *p,p'* -DDE in fish during recent years.

Keywords: Organochlorine pesticides; DDT; HCH; HCB; Egypt; Nile River

INTRODUCTION

Among several man-made agro-chemicals produced and used during the last four decades, organochlorine pesticides have received utmost attention due to their potential for bioaccumulation, persistence and toxicity in humans and wildlife. **In**

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Egypt, large amounts of organochlorine pesticides were used for agricultural purposes during the last three decades. Egypt is the largest pesticide market in Arabian countries and the fourth largest importer of pesticides among developing countries $[1]$. Because of the limited cultivable land available for agriculture, more than 30,000 metric tonnees of formulated pesticides are imported and used intensively to enhance production in the Nile River Delta **[21.** The Ministry of Agriculture of Egypt has reported that the total active ingredients of major organochlorine pesticides used during a 30 year period were: Toxaphene, 54,000 metric tones (1955-1961); Endrin, 10,500 mt (1961-1981); DDT, 13,500 mt (1952- 1971); and Lindane (y-HCH), 21,000 mt (1952-1978). Based on the reports of the harmful effects of these pesticides to wildlife and humans, they were banned or restricted from use during the last decade. However, due to their low biodegradability, organochlorine pesticides used during the last three decades still persist in the environment. Nevertheless, very little information is available for organochlorine pesticides discharged into the Egyptian environment during these periods, and no temporal trend studies have been conducted to evaluate the effectiveness of regulations on the use of these pesticides $[3, 4]$.

The present study examined pollution by organochlorine pesticides in the Nile River and Manzala Lake in Egypt. The Nile River supplies about 60 billion cubic meters of water annually, and one-third of its flow is used for agricultural irrigation. The Nile River water is also used for industrial activities, transportation, disposal of agricultural and industrial wastewater, which results in severe contamination by toxic pollutants. In addition, Manzala Lake, which receives drainages from Damietta branch of the Nile River, is a major source of fishery resources in Egypt, accounting for nearly 50% of the country's total fishery production during the early 1970s **[51** and **35%** during the 1980s **[61.** For the assessment of risks of organochlorine pesticides discharged into the Nile River and Manzala Lake, baseline concentration information is needed. Therefore, as a part of international collaborative research between the National Institute for Resources and Environment in Japan and the University of Mansoura in Egypt, this study examined concentrations of organochlorine pesticides in water, sediment and fish from those aquatic environments.

EXPERIMENTAL

Sample collection

The general location of Manzala Lake and the Nile River is shown in Figure 1. Almost all environmental samples were collected during January 1993. Water,

FIGURE 1 General location of Nile River **and** Manzala Lake in Egypt

suspended solid, sediment and fish samples were collected from several locations in Manzala Lagoon (M1 to **M5)** and the Nile River near Cairo **(C1** to **C3),** Egypt (Figures **2).** Drinking water was collected from a tap at the University of Mansoura in January **1994.**

About **20** L of water was collected using polyethylene containers. Water samples were transported **to** the laboratory and filtered using a glass fiber filter **(GEF;** diameter; **14** cm, pore size; **0.42** mm), which was cleaned by heating at **240°C** for **48** h prior to use. Filtered water samples were passed through a glass column **(2** cm i.d., **20** cm length) packed with **20** g of **XAD-2** resin (Amberlite). The flow rate was maintained at **20 mUmin. A** preliminary study showed that the recovery rates of all organochlorine pesticides analyzed in this study, using solid phase extraction with **XAD-2** resin, were **>91%. XAD-2** resin was cleaned with ethyl alcohol in a Soxhlet apparatus prior **to use.** Drinking water samples were collected twice using **XAD-2.** Surface sediment samples were collected using an

FIGURE 2 Sampling locations in Nile River near by Cairo and Manzala Lake

Eckman-Birdge sampler from all locations. Fish samples were purchased from fishermen at each location, except in Cairo where fishes were purchased at a local market. All samples of sediments, fishes, glass fiber filters and **XAD-2** columns were transported to Japan in dry ice,and analyzed.

Analytical Procedure

Dichlorodiphenyltrichloroethane (p,p'-DDT) and its metabolites **p,p'-DDD** and p, p' -DDE, hexachlorocyclohexanes (HCHs; α -, β -, γ - and δ - isomers), chlordane compounds (CHLs; cis-chlordane, trans-chlordane, cis-nonachlor, trans-nonachlor, heptachlor and oxychlordane) and hexachlorobenzene (HCB) were analyzed following the method described below.

Organochlorine pesticides adsorbed **on** XAD-2 resin were eluted with 300 **mL** of ethanol. The eluate was transferred to 100 **mL** of hexane in 500 **mL** of hexane-washed water in a separatory funnel. After partitioning, the hexane was concentrated and passed through 12 g of activated Florisil(l30 **"C** for 10 h) packed glass column for separation. The **fist** fraction eluted with 120 mL of hexane contained HCB and p, p' -DDE. The second fraction eluted with 150 mL of 20% dichloromethane in hexane contained HCHs, *p,p'* -DDD, p,p'-DDT and CHLs. Each fraction was concentrated using a K-D (Kuderna-Danish) concentrator and washed with concentrated sulfuric acid and hexane-washed water. Suspended solids collected on glass fiber filter were ground with anhydrous sodium sulfate and pulverized mixtures were Soxhlet extracted with 400 mL dichloromethane and hexane $(3:1)$. The extracts containing organochlorine residues were concentrated and treated with concentrated sulfuric acid. After washing with hexane-washed water, extracts were cleaned using Florisil column chromatography as described above. Because of the limited amount of samples, dry weight of suspended solids were not measured. Therefore, results for suspended solid concentrations are reported as pg/L.

Sediment samples were dried at room temperature, and then ground with anhydrous sodium sulfate and extracted in a Soxhlet extractor with 400 mL of dichloromethane : hexane $(3:1)$. The extracts containing organochlorine residues were concentrated and washed with concentrated sulfuric acid. The extracts were treated with activated copper to remove sulfur and cleaned by passing through a Florisil column as described above.

For fish samples, whole fish were minced and homogenized twice using a blender. Twenty g of homogenized fish was ground with anhydrous sodium sulfate and extracted in a Soxhlet extractor with **400** mL of diethylether and hexane $(3:1)$. The hexane extract containing organochlorine residues was concentrated and added to 20 g of Florisil packed in a glass column, and then hexane was removed by passing purified nitrogen. Organochlorine compounds adsorbed on Florisil were eluted using 150 mL of acetonitrile containing 20% water. The eluate was transferred to 100 mL of hexane and 500 mL of hexane-washed water in a separatory funnel. After partitioning, Florisil column separation was performed as described above.

Aliquots of final extracts were injected into a gas chromatograph (Hewlett Packard 5890 II) equipped with 63 Ni electron capture detector and capillary column (FS-WCOT, chemically bonded DB-17, 0.25 mm i.d., 30 m length). Column oven temperature was programmed from 160°C to 230°C at a rate of 1.5° C/min. Injector and detector temperatures were maintained at 250° C and 280"C, respectively. A few samples were also injected into a gas chromatograph equipped with OV-1701 (FS-WCOT, 0.25 mm i.d. **x** 25 m length) for confirmation of compounds detected using DB-17. Concentrations of organochlorine pesticides were quantified from individually resolved *peak* areas with corresponding standard peak areas. The recoveries of target pesticides **through** the entire **analyt**ical procedure were greater than 93%.

RESULTS *AND* **DISCUSSION**

Water

Concentrations of organochlorine pesticides measured in filtrated water samples (dissolved phase) are presented in Table I. HCHs were most predominant, followed by DDTs and CHLs. Among HCH isomers analyzed, β-HCH predominated in samples from the Nile River in Cairo. However, concentrations of HCH isomers in Manzala Lake were in the following order: α -HCH > γ -HCH > β -HCH > δ -HCH, which is similar to that in technical HCH preparations containing 55-70% α -, 10-18% γ -, 5-14% β - and 6-10% δ - isomer ^[7]. Among these, the β -isomer is the most persistent in the environment, while the α -isomer is more soluble in water. Prevalence of β -HCH isomer in water samples from the Nile River in Cairo suggests its leaching from soils contaminated by the past inputs. Use of HCH has been restricted in Egypt since the **1970s.** However, the pattern of HCH isomers in Manzala Lake resembling technical HCH preparations suggests recent usage. Al-Mansoura, a catchment area of Manzala Lake, represents one of the most productive agricultural areas in the Nile Delta. HCH may still be used in this region for agricultural purposes.

	Nile River			Manzala Lake				
	C1	C ₂	C ₃	M1	M ₂	M ₃	M4	M5
hexachlorobenzene	4.3	4.0	3.8	86	158	37	81	30
α-HCH	71	65	74	583	2,815	727	553	482
β -HCH	75	70	114	321	991	349	304	226
γ HCH	65	45	47	487	1,408	467	462	324
δ-HCH	5.8	4.3	5.0	74	167	87	72	61
p, p' -DDT	10	5.8	8.1	19	5.8	12	18	15
p, p' -DDE	22	21	21	218	122	83	207	27
p, p' -DDD	32	16	37	230	232	131	218	46
c-chlordane	4.9	3.9	4.7	5.3	33	13	5.1	1.8
t -chlordane	4.9	4.2	6.3	5.3	35	14	5.1	2.2
c-nonachlor	n.d.	n.d.	n.d.	4.1	2.9	4.4	3.9	n.d.
t-nonachlor	5.0	2.1	3.3	2.5	1.9	2.7	1.8	n.d.
heptachlor	4.5	3.8	1.9	n.d.	7.8	2.6	n.d.	n.d.
oxychlordane	2.4	1.5	1.2	14	3.6	4.5	11	3.1

TABLE I Organochlorine concentrations (pg *I* L) **in filtrated water samples collected from Nile River and Manzala Lake in** 1993

 $n.d. < 1$ pg $/L$

25 17 **44** 138 12 5.3 4.6 3.9 12 37 4.7 2.4 5.0 6.2 4.1 17 6.7 3.5 6.0 6.4 5.3 22 14 5.9 n.d. n.d. n.d. 2.6 n.d 2.5 2.8 2.6 1.9 6.2 1.3 2.3 1.6 3.6 1.7 3.0 2.6 1.9 0.89 2.1 2.5 2.5 1.3 1.5

TABLE II Organochlorine concentrations (pg $/ L$) in suspended solid samples collected from Nile River and Manzala Lake in 1993

> 31 11 7.8 8.0 1 .o 3.4 2.6 1.2

ox ychlordane n.d. < 0.1 pg $/L$

hexachlorobenzene

a-HCH β-HCH y-HCH 6-HCH p,p'-DDT p,p'-DDE p,p'-DDD c-chlordane i-chlordane c-nonachlor r-nonachlor heptachlor

Among all sampling locations in Manzala Lake, the highest concentrations of pesticides were detected at location M2 (see Figure 2) where two main drainages, Bahr El-bakar and Bahr Hados accounting for **75%** of the drainages in Manzala Lake, are located. Water concentrations of almost all pesticides decreased linearly from M2 to M5. The location M5, located near Gamil, is the main outlet of lake water to the Mediterranean Sea. Egyptian National Environmental Action Plan reported that Bahr El-bakar and Bahr Hados drainages were the major sources of heavy metals and nutrients in Manzala Lake $\left[8\right]$. High incidences of gill diseases and internal parasites in fish collected from this area have been attributed to pollution by contaminants $[9]$.

Concentrations of organochlorine pesticides in suspended solids and its contribution to total concentrations in water **are** shown in Tables **I1** and **111,** respectively. Proportions of **CHLs** in suspended solids were similar to that in dissolved phase. However, there were some differences in the proportions of organochlorine pesticides in dissolved and particulate phases, depending on sampling locations. Proportions of HCHs in particulate phases were 3 times higher in Manzala Lake than those from the Nile River in Cairo. On the other hand, **DDTs** and HCB

8.9 2.2 1.1 3.3 2.1 0.23 0.15 0.69 were predominant in dissolved fractions in Manzala Lake than those from the Nile River. This may suggest a lack of equilibrium in organochlorine pesticide concentrations in water. In addition, plankton productivity, organic carbon, resuspension of sediments and octanol-water partitioning coefficients can influence the partitioning of organochlorines between suspended and dissolved phases.

concentration in suspended solid *I* **(conc. in suspended solid** + **conc. in water)** x 100

Sediments

Residue levels of pesticides in sediments **from** Manzala Lake are shown in Table **IV.** Spatial distribution of concentrations of **HCB, HCHs,** DDTs and **CHLs** in sediments was similar to those found in filtrated water, which decreased with increase in distance from M2. In contrast to that in filtrated water, p, p' -DDE was the predominant contaminant in sediment. Despite the predominance of **a-HCH** in water samples, **P-HCH** was predominant in sediments from Manzala Lake, suggesting a greater potential of β -isomer than α -isomer to persistence.

		Nile River		Manzala Lake					
	C1	C ₃	M1	M2	M ₃	M4	M5		
hexachlorobenzene	4.5	3.8	0.35	0.60	0.10	0.37	0.17		
α -HCH	0.03	0.02	0.21	4.8	0.15	0.56	0.28		
β -HCH	1.4	0.95	2.4	8.5	4.0	3.5	3.0		
γ -HCH	0.02	n.d.	0.07	2.7	0.18	0.04	0.04		
δ-HCH	n.d.	n.d	n.d.	0.08	0.01	0.07	0.02		
p, p' -DDT	8.5	5.6	26	79	2.2	0.08	0.08		
p, p' -DDE	48	43	132	432	46	12	3.2		
p, p' -DDD	40	32	37	129	21	6.1	2.1		
c -chlordane	6.5	3.2	1.2	18	4.2	0.63	0.19		
t-chlordane	1.2	0.83	0.37	0.5	1.1	0.46	0.21		
c -nonachlor	0.53	0.42	n.d.	0.11	0.08	0.13	0.07		
t-nonachlor	2.56	1.65	0.31	9.4	2.0	0.43	0.09		
heptachlor	0.93	0.52	0.05	0.60	0.25	0.07	0.01		
oxychlordane	0.75	0.67	0.04	0.42	0.2	0.06	0.02		

TABLE IV Organochlorine concentrations (ng / **g** dry wt.) in sediment samples collected from Nile River and Manzala Lake in 1993

n.d. < 0.01 ng / **g**

An earlier study has reported the concentrations of organochlorine pesticides in Nile River water collected in 1982^[10]. Concentrations of pesticides reported in water were: Lindane **('y-HCH),** 0.1 ng/L; p,p'-DDT, 0.9 **ng/L;** p,p'-DDE, **4.4** ngL; p,p'-DDD, 2.7 ng/L. These values are remarkably higher than the samples collected in 1993 in this study, except Lindane. Concentrations of DDT compounds decreased by 100-200 fold while Lindane decreased by only 1.4-fold. Although, the differences in the sampling periods, in the particular rainfall $[11]$, and analytical methods may influence concentrations of organochlorines, these results suggest a decline in DDT inputs into the Nile River during **the** last decade, despite the continued, but limited use of DDT as **a** rodenticide and termiticide in recent years [12].

Fish

Organochlorine pesticides were found in almost all fish **from** the Nile River and Manzala Lake (Table **V).** Concentrations of p,p'-DDE was the highest in fish.

FIGURE 3 Isomer composition of HCHs in environmental samples from Egypt

Aly and Badawy [13] reported residue levels of Lindane (8.9 ng/g), p,p'-DDT (15 ng/g) , p, p' -DDE (7 ng/g) and p, p' -DDD (49 ng/g) in fish collected from the **Nile River in Cairo during 1981-1982. Compared with those of our results, concentrations of Lindane and p,p'-DDT declined during the last decade. However,** the reduction rate in fish was lesser than that estimated for water. Furthermore, residue levels of *p,p'* -DDE increased by 9-fold during the last decade. These results seem to suggest continuous exposure of fish **to** p,p'-DDE from contaminated sediment.

n.d. < 0.01 ng *I* g **i.f.:** not quantified because of interference of **unknown** *peak.*

Isomer profiles of HCHs and DDTs in fish, dissolved phase, suspended solids and sediment are presented in Figures 3 and **4.** The HCH isomer distribution patterns in the suspended particulate and dissolved fractions were similar, whereas the distribution of **DDT** compounds in the particulate fraction was similar to that in sediment. This reflects the flux of DDT compounds from sediments to suspended particles. However, a clear trend in the composition of **CHLs** was not discerned (Figure *5)*

FIGURE **4 Isomer composition of DDTs in environmental samples** from **Egypt**

Drinking water

Organochlorine concentrations were also measured in drinking water collected from **the University of Mansoura (Table VI). HCB,** *a-,* y-, **GHCH, p,p'-DDE and t-chlordane were detected in drinking water at concentrations similar to that in**

FIGURE *5* **Composition of CHLs** in **environmental samples from Egypt**

water from **Manzala Lake. Tap water collected in Alexandria, Egypt, contained** Lindane (0.29 ng/L), p, p' -DDT (0.47 ng/L) and heptachlor (0.12 ng/L) ^[14]. p, p' **-DDT and heptachlor were not detected in this study because** of **interferences** from **unknown peaks. However, detectable levels** of **organochlorine pesticides in**

Ħ

VZZ

 \overline{m}

YX

77.

 $\overline{\mathbb{Z}}$

TAC

100 $(%)$

VA.

drinking water is of concern from human exposure point. Considering an average daily intake of 2 L of water, exposure from water along would contribute to **1.5** to 1.9 ng HCHs/person/day. Intake from other food items including fish which was discussed above will be of further concern. In addition, several ECD-responsive compounds were also noticed in drinking water, for which identities were not examined, since our analytical procedure was optimized mainly for organochlorine pesticides.

n.d. *c* 1 **pg** *I* **L**

i.f. : not quantified because of interference of unknown *peak.*

CONCLUSIONS

Discharge of HCHs into the Nile River appears to have decreased after the ban on usage in 1978. However, agricultural regions near Al-Mansoura continues to be a source of HCHs in the Nile River Delta. In Manzala Lake, drainages from **Bahr** El-bakar and **Bahr** Hados are the main sources of organochlorine pesticides. Organochlorine pesticides in water has decreased during last decade. However, residue levels of p, p' -DDE in fish has increased, and contaminated sediments are the probable source of DDTs in fish. Although the dam constructed across the Nile River in Aswan in 1972 reduced the movement of sediments downstream, and therefore the transport of pollutants, higher accumulation in the Delta sediments and fish was still observed.

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