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Levels, Sources, Toxicity and Ecological Risk Assessment of OCPs and PCBs in Lake Edku Waters, Egypt

Mohamed A. Okbah[1], Mohammed Nassar [2], Mahmoud S. Ibrahim[2], Maie I. El-Gammal[2]

[1]Marine Chemistry Lab, Marine Environment Division, National Institute of Oceanography and Fisheries, NIOF, Cairo, Egypt
[2]Environmental Sciences Department, Faculty of Science, Damietta University, Egypt
[*]**Corresponding Author: m_okbah@yahoo.com**

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ABSTRACT

Organochlorine pesticides (OCPs) and polychlorinated biphenyls (PCBs) have the ability to bioaccumulate in food chains and bodies. For Lake Edku, hexachlorocyclohexane (HCH) was isometrically arranged as β > γ > α during both seasons and seasonally arranged as spring> summer. The β-HCH was the most dominant isomer that can be attributed to the biodegradation of α-HCH and γ-HCH to β-HCH. Total Cyclodienes were arranged as Aldrin> Dieldrin> Endrin, and seasonally as spring> summer. The average contributions of DDT congeners mixture detected in Edku Lake waters were in the order of o,p′-DDD> o,p′-DDT> o,p′-DDE> p,p′-DDE>p,p′-DDT > p,p′-DDD during spring and o,p′-DDT> o,p′-DDE> o,p′-DDD> p,p′-DDE and p,p′-DDT > p,p′-DDD during summer; total DDTs recorded higher values during the summer compared to spring. Seasonal distribution is referred to the fact that PCB 28> 52>138> 101> 118> 153> 180 during both seasons. α-HCH/γ-HCH indicated the dominance of lindane. β-/(α+γ)-HCH ratio suggested the recent HCH input for station VI and past input for most stations. DDE/DDD ratios indicate anaerobic and aerobic degradation. (DDE + DDD)/∑DDTs ratios suggesting past input for DDT for most stations during both seasons. o,p′-DDT/p,p′-DDT ratio indicates that stations (I and VIII), as well as station VIII during spring and summer, respectively, recorded technical grade DDT, while the rest of the stations recorded dicofol-type DDT. While, the risk quotient recorded very high risk, except for α-HCH which recorded a moderated risk at some stations. Upon comparing the results of the current study with water quality standards, it was noted that some OCPs and PCBs recorded values higher than the criteria continuous concentration (CCC), while others were higher than the criteria maximum concentration (CMC) during spring and summer.

INTRODUCTION

Lake Edku is the third largest northern Egyptian coastal lake in the Nile Delta in the Beheira Governorate and one of the most threatened aquatic environments in Egypt due to anthropogenic activities and pollution (**Emam *et al.*, 2021)**. Lake Edku suffers from numerous pollutant sources, represented in point and non-point sources due to anthropogenic activities such as agricultural and municipal wastewater, which may represent a significant threat to the aquatic environment (**Radwan *et al.*, 2019**).

Organochlorines are also called chlorinated hydrocarbons, chlorinated insecticides, chlorinated synthetics and chlorinated organics **(Ramamoorthy & Ramamoorthy, 1997)**. On the other hand, hexchlorocyclohexane (HCH) contains five isomers: alpha (α), beta (β), gamma (γ), delta (Δ) and epsilon (E). HCH has a strong flavor and odor, which can be transported and accumulated in crops and animal products and subsequently humans. HCH is still used and manufactured in many developing countries due to its low cost **(Karabelas *et al.*, 2009)**. Organochlorine pesticides (α-HCH, β-HCH, γ-HCH, aldrin, dieldrin, endrin, o,p′-DDE, p,p′-DDE, o,p′-DDD, p,p′-DDD, o,p′-DDT, and p,p′-DDT), organochlorine pesticides (OCPs), particularly DDT and HCH, are of major international concern due to their persistent properties and wide reach, especially in developing countries. Agricultural runoff, domestic and industrial wastewater, and atmospheric deposition is the most source of Organochlorine pesticides in the water environment. The danger of organochlorine pesticides lies in their high potential for bioaccumulation, which poses a real threat to humans and the environment **(Zhu *et al.*, 2005; Syed *et al.*, 2013)**. OCPs can bioaccumulate (enter of OCPs into the tissues of organisms) in organisms due to their lipophilic nature **(Sparling, 2016)**, causing dizziness, headache, vomiting, convulsions, tremors, muscle weakness, salivation, and confusion symptoms may occur in the short term, while the long-term exposure can cause deleterious effects on kidneys, liver, thyroid gland and nervous system. In addition, it can cause cancer in humans and can impact the reproductive organs of humans by inhibiting T stimulated by Leydig cells, and hence the effect would penetrate to the human placenta. Furthermore, experiments have proven its effect on the testicles and sperm formation through impacting the testicular steroidogenesis **(Chatterjee & Agarwal, 1988; Wango *et al.*, 1997)**. OCPs and PCBs have four critical characteristics: persistence, high toxicity, migration and bioaccumulation. Remarkably, persistence has the ability to resist photolysis processes, biodegradation and chemical decomposition. Whereas, bioaccumulation is characterized by low water solubility, and high lipid solubility, making them trend to bioaccumulation in food chains and bodies. For migration, it can travel long distances by several methods such as air (wind) and water (water currents) without degradation. On the other hand, high toxicity can cause endocrine disruptors, inhibit the immune system, fatty liver and reduce virus resistance **(Liu, 2021)**.

MATERIALS AND METHODS

Study area and sampling

Lake Edku is the third most important lake in northern Egypt, which is located in the Beheira Governorate, 30km from Alexandria Governorate, especially from Abu Qir coast. There are four drains (Idku, Al-Busaili, Al-Khairi and Al-Baraziq) representing the most important sources of the lake's supply of sewage and agricultural wastewater. Moreover, the lake is connected to the Mediterranean Sea through the Ma'adiyah inlet. Eight main points representing sampling stations were identified. These stations cover the whole lake, as shown in Fig. (1). Stations I, II, and III were selected to cover the southern and middle part of the lake; stations IV, V, VI and VII were chosen to cover the northern part of the lake, and station VIII covers the point where the sea is connected to the lake. Via using an 5- liter amber glass bottle, the surface water samples were collected. Before sampling, nitric acid (10%) was used to wash the bottle, followed by diluted and deionized water; bottles were finally rinsed with water samples before collection to

prevent cross-contamination during the process **(Salaudeen *et al.*, 2018; Ambade *et al.*, 2021)**. Samples were preserved and kept in the icebox and transferred to the laboratory; water samples were stored in the refrigerator at – 3°C for further analysis of OCPs, and PCBs. During the present study, organochlorine pesticides (OCPs) hexachlorocyclohexane or benzene hexachloride (α-HCH, β-HCH, γ-HCH), Cyclodienes organochlorine (aldrin, dieldrin, and endrin), dichlorodiphenyltrichloroethane (o,p-DDE, p,p′-DDE, o,p-DDD, p,p′-DDD, o,p-DDT, and p,p′-DDT) and polychlorinated biphenyls PCBs (PCB 28, 52, 101, 118, 138,153, and 180) were determined for analysis.

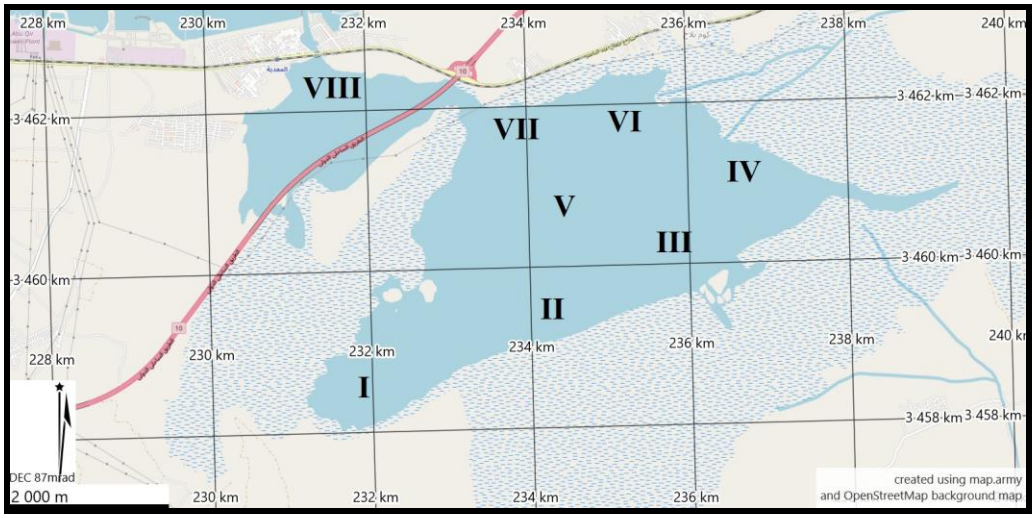


Fig. 1. Map of sampling stations at Lake Edku, Egypt

Extraction of dissolved OCPs and PCBs in water samples

Water samples were extracted through liquid-liquid extraction, followed by gas chromatographic analysis according to **USEPA (1980)** and **Gómez-Gutiérrez *et al.* (2007)**. Samples were filtered through GF/F 45mm- filter paper. One liter of each filtered water sample was mixed with 200mL of dichloromethane (DCM) and shacked vigorously for 5min in a 5L- conical flask, and then separated in a separating funnel. The lower layer of the organic solvent containing pollutants was collected on anhydrous Na_2SO_4 while adding pentachlorobenzene as an internal standard for organic chlorinate. The extract was concentrated by rotary evaporation to 2ml, and then to 1ml under a gentle stream of pure N_2, and then it was transferred to 10ml- glass tubes while adding few amounts of n-hexane.

OCPs and PCBs Instrumental analysis

For the quantification of OCPs and PCBs in water samples, Hewlett Packard was used having 5890 series II GC gas chromatograph equipped and connected with an electron capture detector (ECD), and fused-silica capillary (30× 0.32 mm× 0.52 μm) column was used. GC was programmed as follows: (1) initial temperature from 70 to 280°C with a rate of 5°C min^{-1} and then kept at 280°C for 20min. (2) Temperatures detector and injector were maintained at 300 and 270°C, respectively. (3) Carrier gas (Helium) of 1.5ml min^{-1}, with nitrogen gas at a following rate of 60ml min^{-1}, was used as make-up (**El Nemr *et al.*, 2012)**.

RESULTS AND DISCUSSION

Hexachlorocyclohexane (HCHs) or benzenehexachloride (BHC)

Hexachlorocyclohexane (HCH) is a widespread contaminant that entered different environments during manufacture, and it is used as a pesticide and waste incineration **(Van Birgelen, 1998)**. During the present study, as illustrated in Tables (1, 2) and Fig. (2), the total hexachlorocyclohexane (HCH) varied between a minimum value (1.07-2.96) µg/L at station (VIII-I) and a maximum value (6.53-5.11) µg/L at station (IV), with an average recorded (3.77±199 & 4.30±0.93µg/ L) during spring and summer, respectively. α-HCH varied between the lowest value (0.07- 0.09) µg/L at station III and the highest value (1.02- 1.22) µg/L at stations II & I, with an average (0.36± 0.3- 0.1± 0.38µg/ L); β-HCH ranged between a minimum value (0.10- 0.53) µg/L at stations VIII& V and a maximum value (5.33- 3.34) µg/L at station IV, with an average recorded value (2.04±1.62- 1.86±0.87µg/ L). Whereas, γ -HCH fluctuated between a minimum (0. 69-0.75) µg/L at stations IV & I and a maximum (3.15- 2.54) µg/L at stations II & VIII, with an average recorded value (1.37± 0.86 & 1.73± 0.60 µg/L) during spring and summer, respectively, which is considered the most dominated HCHs with insecticidal properties (lindane); however, HCH does not occur as a natural substance. Eminently, γ-HCH can be introduced to the water body via "down-the-drain" resulting from wash-off bodies that use products containing γ-HCH, which is used for lice and scabies treatments; or via surface runoff (dissolved or absorbed) or by deposition from air **(Battaglin & Fairchild, 2002)**. HCHs were isometrically arranged as β > γ > α during both seasons, while they were regionally assessed as III> II> V> VIII> VII> I> IV> VI during spring, and VIII> IV> VII> II> VI> III> V> I seasonally for spring> summer. HCHs are considered one of the less persistent organochlorine pesticides (POPs). HCH is a moderately stable compound that only decomposes under alkaline to yield trichlorobenzene (**Said *et al.*,2015)**.

Chlorinated cyclodienes (Aldrin, Dieldrin and Endrin)

Total cyclodienes (TC) containing Aldrin, Dieldrin and Endrin form a strong soil insecticide due to their relative stability thus being used as termites' control **(Burkhard *et al.*, 2023)**. Toxicity of cyclodienes increases with increasing ambient temperature. Cyclodienes can cause nervous disorders followed by tremors and convulsions **(Costa, 2015)**. As illustrated in Tables (1, 2), Aldrin recorded values ranging from 0.46µg/ L at station VIII to 5.12µg/ L at station V, with an average of 2.38 ±1.76µg/ L during spring, while values from 0.32 at station VIII to 7.36µg/ L at station IV were recorded with an average of 3.18±2.73µg/ L during summer. Aldrin is less resistant to oxidation compared to aromatic compounds. In the ecosystem, Aldrin converts to Dieldrin in the environment (**Said *et al.*, 2015)**. Dieldrin recorded values ranging from 0.46µg/ L at station IV to 4.26µg/ L at station III, with an average of 2.34±1.20µg/ L during spring, and from 0.35µg/ L at station VIII to 7.18µg/ L at station III, with an average of 2.61±2.27µg/ L during summer. On the other hand during spring, Endrin fluctuated between 0.79µg/ L at station VI and 7.12µg/ L for station II, with an average of 3.31±2.08µg/ L. While, during summer, it fluctuated between 0.86µg/ L at station VIII and 4.34µg/ L at station II, with an average of 2.48 ±1.22µg/ L. Total cyclodienes recorded a minimum value of 4.90µg/ L at station VIII and a maximum value of 14.64µg/ L, with an average of 8.04±2.99µg/ L during spring. During summer, the recorded values fluctuated between 1.53 and 14.13

µg/L with an average of 8.28±5.03µg/ L, as shown in Fig. (2). Sites close to agricultural drains and fish farms (II, III, V & VI) recorded the highest concentrations, which indicates that they are the main source of these compounds. Total cyclodienes were arranged as Aldrin> Dieldrin> Endrin, seasonally as spring> summer, and regionally as II> III> V> I> IV> VII> VI> VIII during spring, and III> V> IV> I> II> VII> VI> VIII during summer.

Diphenyl aliphatic (DDE, DDD, and DDT):

Total diphenyl aliphatic (∑DDTs) concentration ranged from 1.75-1.14µg/ L at station VIII and 10.50- 17.43µg/L at station I, with an average of 7.39±3.13µg/ L & 9.63 ±5.59 µg/L) during spring and summer, respectively, as shown in Table (1, 2) and Fig. (2). o,p′-DDE and p,p′-DDE recorded minimum values (0.32- 0.09) µg/L at stations V & VIII and maximum values (3.44- 2.99) µg/L at stations (I & III), with an average of 1.84 ±1.24 µg/L & 1.64 ±1.09 µg/L, respectively, during summer. While, during spring, it ranged between a minimum value (0.13- 0.24)µg/L at stations V & VI and a maximum value (4.22- 3.36)µg/L at stations IV & II, with an average value (1.30 ±1.47-1.23 ±1.05)µg/L. o,p′-DDD and p,p′-DDD concentrations recorded minimum values (055–0.70) µg/L at stations I & VI and maximum values (4.26–0.35) µg/L at stations III & II, with an average of 1.97±1.22 & 0.17±0.10µg/ L, respectively, during spring. On the other hand, during summer it ranged from 0.27- 0.03µg/ L at the same station (VIII) to 4.03-3.02µg/ L at station I, with average values of 1.84 ±1.40 & 1.57 ±1.03µg/ L, respectively. o,p′-DDT and p,p′-DDT ranged between a minimum value (0.05-0.09) µg/L at stations VIII & II and a maximum value (5.12-7.05) µg/L at stations VI & I, with an average (1.64 ±1.89-1.08 ±2.42) during spring. During summer, it ranged between 0.09 & 0.34 µg/L at station VIII and a maximum value (3.30-2.21) µg/L at stations I & VII, with an average of 1.52 ±1.06 & 1.24 ±0.62µg/ L, respectively, as shown in Table (1, 2). p,p'-DDT is considered the main component of insecticides containing DDT which it contains (65–80%) as an active ingredient **(Battaglin & Fairchild, 2002)**. The high concentrations of total DDTs might result from the discharge of untreated effluents from the main drains that flow into the lake, or that release from the agricultural areas located upstream in the drains (Bousaly, Edku, Berseek and El-Khairy) could be transported and deposited. The average contributions of DDT congeners mixture detected in the water samples were in the order of: o,p′-DDD> o,p′-DDT> o,p′-DDE> p,p′-DDE>p,p′-DDT > p,p′-DDD during spring and o,p′-DDT> o,p′-DDE> o,p′-DDD> p,p′-DDE and p,p′-DDT > p,p′-DDD during summer (Tables 1, 2). DDE and DDD resulting from DDT break down, DDE, DDD and DDT do not dissolve easily in water and take a long time (2:15 year) to degradation by Microorganisms **(ATSDR, 2022)**. swallowing large amounts of DDT can lead to headaches, nausea and seizures and can cause Type II diabetes mellitus; scientific experiments revealed that exposure of animals to large quantities of DDT may lead to nervous system, liver and reproductive system disorders **(Peter & Cherian, 2000; Bernardes *et al.*, 2015)**. The increase in DDT concentration, especially p,p-DDT is due to being the most prevalent. Solar radiation and metabolism of aquatic organisms are considered the most important reasons for DDT degradation **(Sudharshan *et al.*, 2012)**.

Polychlorinated biphenyls (PCBs)

Regional and seasonal distribution of PCBs (28, 52, 101, 118, 138, 153 and 180) in Lake Edku water samples is illustrated in Table (1, 2) and Fig. (2). PCB congener 28 was

predominant with concentration values ranging between 0.70 at station VIII and 12.35μg/ L at station II, with an average of 4.35±4.13μg/ L−1during spring; and from 0.09 μg/L at station VIII and 7.01 μg/L at station III, with an average of 3.33±2.38 μg/L−1 during summer. Other PCBs ranged from 0.06 at station VII to 11.2 at station II, with an average of 2.75 ±3.60μg/ L, and from 0.51 at station V to 3.65 at station IV, with an average of 2.07 ±1.15μg/ L, and from 0.95 at station II to 2.33 at station VI, with an average of 1.48 ±0.52μg/ L. While, it fluctuated from 0.89 at station VII to 5.15 at station V, with an average of 2.46 ±1.56μg/ L), and from 0.09 at station I to 2.01 at station VII, with an average of 0.70 ±0.67μg/ L. On the other hand, values varied from 0.01 at station V to 1.25 at station II, with an average of 0.52 ±0.47 for PCBs (52, 101, 118, 138, 153, and 180), respectively, during spring. Additionally, during summer, the range was from 0.23 at station VIII to 6.23 at station III, with an average of 2.63 ±2.00μg/ L, and from 0.32 at station VIII to 4.02 at station I, with an average of 2.35 ±1.31μg/ L, while values ranged from 0.11 at station VIII to 6.79 at station III, with an average of 2.32 ±2.32μg/ L, and differed from 0.09 at station VI to 6.12 at station V, with an average of 2.60 ±2.44μg/ L. Other values were recorded (from 0.11 at station VIII and 3.22 at station II with an average 1.62 ±1.13 μg/L), (from 0.02 at station VIII and 2.14 at station III with an average 1.37 ±0.69 μg/L) for PCBs (52, 101, 118, 138, 153 and 180), respectively. Seasonal distribution is referred to the fact that, PCB 28> 52>138> 101> 118> 153> 180 during both seasons, regional distribution referred to that the station II> III> IV> VII> VI> I> V> VIII during spring, and station III> II> I> V> IV> VIII> VI> VIII, respectively, during summer. The presence of high concentrations in their station (III) in both seasons is an indication that the source of the PCBs in the lake originates from agricultural drainage or human activities. The presence of some high concentrations in some different areas is an indication of the transport of PCBs (**Weiss *et al.*, 2000)**. The outer areas of the lake are gradual to the center of the lake, then the lowest levels are in the area where the lake is connected to the sea, which may be a result of the volume of water flowing in that area. PCBs are considered stable and semi volatile compounds, thus they can move long distances **(Mansour, 2009)**. The PCBs concentration near drains effluents is higher than the areas near the Bogaz (inlet), whose characteristics are considered the main reason for PCBs distribution, especially for high-molecular-weight PCB **(Motelay-Massei *et al.*, 2004)**. The finding of the current study agrees with that of **Khadhar *et al.*, (2018)** who elucidated that, the downstream stations contain high levels of higher molecular PCB, predominantly indicating anthropogenic sources.

Table 1. Levels (μg/L) of OPCs and PCBs in water samples collected from Lake Edku during spring 2022

	I	II	III	IV	V	VI	VII	VIII	Min.	Max.	Aver.	SD.
α-HCH	0.42	1.02	0.07	0.51	0.12	0.25	0.33	0.15	0.07	1.02	0.36	0.31
β-HCH	1.56	2.33	2.48	5.33	0.25	1.88	2.39	0.10	0.10	5.33	2.04	1.62
γ-HCH	2.16	3.15	1.25	0.69	1.26	0.90	0.76	0.83	0.69	3.15	1.37	0.86
HCHs	4.13	6.51	3.80	6.53	1.63	3.03	3.47	1.07	1.07	6.53	3.77	1.99
Aldrin	0.96	4.26	1.55	2.24	5.12	3.70	0.78	0.46	0.46	5.12	2.38	1.76
Dieldrin	2.41	3.26	4.26	0.46	2.35	1.35	3.03	1.63	0.46	4.26	2.34	1.20
Endrin	4.56	7.12	3.13	4.53	0.98	0.79	2.56	2.81	0.79	7.12	3.31	2.08
TC	7.94	14.64	8.93	7.22	8.46	5.83	6.37	4.90	4.90	14.64	8.04	2.99
o,p-DDE	0.54	2.35	2.16	4.22	0.13	0.24	0.36	0.37	0.13	4.22	1.30	1.47
p,p-DDE	1.01	3.36	2.06	1.35	0.55	0.24	1.03	0.26	0.24	3.36	1.23	1.05
o,p-DDD	0.55	2.36	4.26	2.65	1.35	2.48	1.37	0.75	0.55	4.26	1.97	1.22
p,p-DDD	0.23	0.35	0.14	0.26	0.09	0.07	0.12	0.13	0.07	0.35	0.17	0.10
o,p-DDT	1.13	1.12	0.15	0.33	1.26	5.12	4.01	0.05	0.05	5.12	1.64	1.89
p,p-DDT	7.05	0.09	0.25	0.36	0.15	0.31	0.22	0.18	0.09	7.05	1.08	2.42
DDTs	10.50	9.64	9.01	9.17	3.52	8.44	7.10	1.75	1.75	10.50	7.39	3.13
PCB 28	0.70	12.35	8.26	5.41	1.26	0.98	3.25	2.55	0.70	12.35	4.35	4.13
PCB 52	2.36	11.32	2.04	3.11	0.69	1.36	0.06	1.06	0.06	11.32	2.75	3.60
PCB 101	2.06	1.26	2.54	3.65	0.51	3.25	2.54	0.72	0.51	3.65	2.07	1.15
PCB 118	1.56	0.95	2.14	1.35	1.02	2.33	1.45	1.03	0.95	2.33	1.48	0.52
PCB 138	1.06	2.37	3.02	2.01	5.15	1.06	0.89	4.13	0.89	5.15	2.46	1.56
PCB 153	1.02	0.15	0.32	0.14	0.75	1.13	2.01	0.09	0.09	2.01	0.70	0.67
PCB 180	0.22	1.25	1.09	0.85	0.01	0.15	0.36	0.22	0.01	1.25	0.52	0.47
TP	22.57	30.79	21.74	22.91	13.60	17.31	16.95	7.72	7.72	30.79	19.20	6.95
PCBs	8.97	29.65	19.42	16.53	9.38	10.26	10.56	9.79	8.97	29.65	14.32	7.27

Table 2. Levels (μg/L) of OPCs and PCBs in water samples collected from Lake Edku during summer 2022

	I	II	III	IV	V	VI	VII	VIII	Min.	Max.	Aver.	SD.
α-HCH	1.22	1.03	0.09	0.66	0.65	1.02	0.71	0.31	0.09	1.22	0.71	0.38
β-HCH	0.98	1.88	1.95	3.34	0.53	1.55	2.38	2.25	0.53	3.34	1.86	0.87
γ-HCH	0.75	2.02	1.36	1.10	2.04	2.07	1.93	2.54	0.75	2.54	1.73	0.60
HCHs	2.96	4.93	3.40	5.10	3.21	4.64	5.02	5.11	2.96	5.11	4.30	0.93
Aldrin	4.42	0.99	3.97	7.36	6.38	1.03	1.02	0.32	0.32	7.36	3.18	2.73
Dieldrin	3.66	2.67	7.18	1.85	3.73	0.89	0.58	0.35	0.35	7.18	2.61	2.27
Endrin	1.99	4.34	2.99	3.63	2.99	1.03	2.03	0.86	0.86	4.34	2.48	1.22
TC	10.07	8.01	14.13	12.83	13.10	2.94	3.63	1.53	1.53	14.13	8.28	5.03
o,p-DDE	3.06	1.99	3.02	3.44	1.43	0.34	1.08	0.32	0.32	3.44	1.84	1.24
p,p-DDE	2.96	2.01	2.99	2.35	0.76	0.92	0.99	0.09	0.09	2.99	1.64	1.09
o,p-DDD	4.03	3.05	3.04	1.99	0.98	0.52	0.81	0.27	0.27	4.03	1.84	1.40
p,p-DDD	3.02	1.11	2.34	2.21	2.22	1.02	0.57	0.03	0.03	3.02	1.57	1.03
o,p-DDT	3.30	1.35	2.59	1.30	2.02	0.62	0.88	0.09	0.09	3.30	1.52	1.06
p,p-DDT	1.07	0.99	1.33	2.07	1.05	0.88	2.21	0.34	0.34	2.21	1.24	0.62
DDTs	17.43	10.50	15.31	13.36	8.46	4.30	6.55	1.14	1.14	17.43	9.63	5.59
PCB 28	5.53	4.99	7.01	3.65	2.33	1.04	2.02	0.09	0.09	7.01	3.33	2.38
PCB 52	3.33	4.21	6.23	2.94	1.32	0.55	2.21	0.23	0.23	6.23	2.63	2.00
PCB 101	4.02	3.55	3.55	2.32	2.10	0.94	1.98	0.32	0.32	4.02	2.35	1.31
PCB 118	3.92	3.00	6.79	0.34	3.02	0.87	0.54	0.11	0.11	6.79	2.32	2.32
PCB 138	3.06	3.10	6.00	0.65	6.12	0.09	1.65	0.12	0.09	6.12	2.60	2.44
PCB 153	2.02	3.22	1.01	0.93	3.22	0.91	1.55	0.11	0.11	3.22	1.62	1.13
PCB 180	1.92	2.02	2.14	1.12	1.32	1.04	1.38	0.02	0.02	2.14	1.37	0.69
PCBs	23.80	24.09	32.73	11.95	19.43	5.44	11.32	1.00	1.00	32.73	16.22	10.64
TP	30.45	23.44	32.84	31.29	24.77	11.88	15.20	7.78	7.78	32.84	22.21	9.53

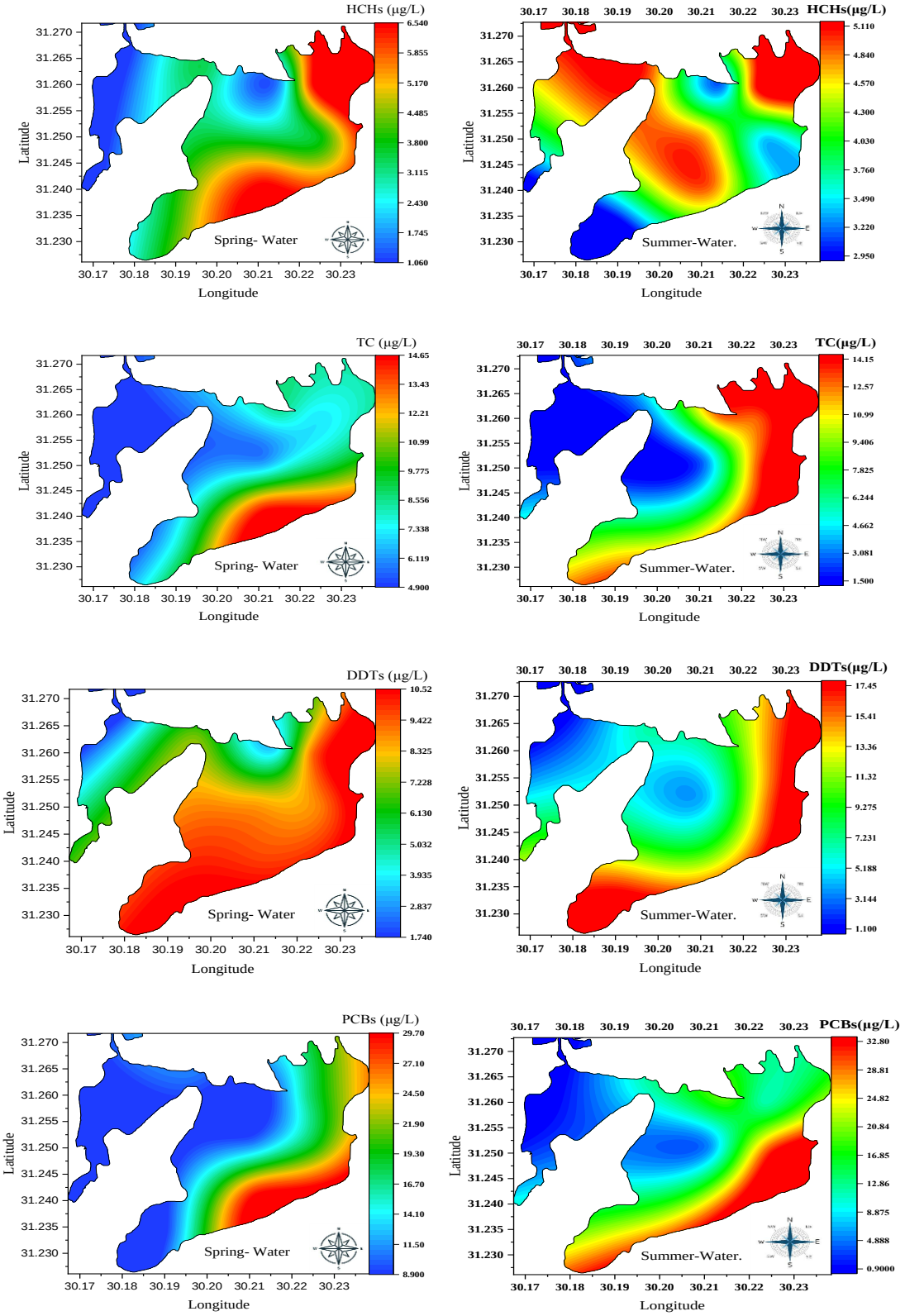


Fig. 2. Horizontal distribution contours stimulating OCPs and PCBs in Lake Edku during spring and summer 2022

Sources of OCPs and PCBs in surface water

α-HCH/γ-HCH and β-/(α+γ)-HCH ratios were used to determine the sources of OCPs, Moreover, β-/(α+γ)-HCH ratio is another method used to determine the usage history of technical HCH and lindane. The ratio of DDD/DDE was used to detect the degradation pathways of DDTs; DDT degrades to DDD, and DDE under aerobic and anaerobic conditions. Whereas, o,p′-DDT/p,p′-DDT ratio was used to determine the sources of DDTs (DDE+DDD)/ ∑DDTs in order to determine possible historical and recent pollution sources of DDTs, as illustrated in Table (3).

Fig. (3) shows that, the ratio of α-HCH/γ-HCH in all studied stations was below 4, with β-HCH and γ-HCH as the predominant isomer; β-HCH is considered the most dominant isomer persistent found in different environments; α-HCH and γ-HCH can be biodegraded to β-HCH in the environment **(Willett *et al*., 1998; Wu *et al.*, 2013)**. The result suggested the use of pure lindane for agricultural purposes **(Botwe *et al.*, 2017; Hao *et al*., 2019; Malhat *et al.*, 2021)**, where agricultural drainage water is considered one of the most important sources of water for Lake Edku. The ratio of β-/(α+γ)-HCH varied between 1.88 and 0.18 during spring, and from 1.91 to 0.20 during summer; stations VI & VIII as well as I & II recorded values below 0.5, suggesting that the recent HCH input **(Zhou *et al.*, 2014)** occurred in the study area, as illustrated in Fig. (3). It may be assumed that HCHs contamination is mainly originated from past usage. During dechlorination and dehydrochlorination processes, DDT can be biodegraded into DDE under aerobic conditions and DDD under anaerobic conditions in the environment; DDE/DDD ratio if greater than 1, then it would indicate the occurrence of aerobic degradation of parent DDT; if it is lower than 1, it indicates that the losses of DDT were predominantly by anaerobic degradation (**Zhou *et al.*, (2014)** (Table 3). As illustrated in Fig (4), the DDE/DDD ratios ranged between 0.08 and 10.61during spring and from 0.67 to 1.72 during summer; the results of ratio values greater than 1 at stations II, III, IV and VIII during spring and stations I, II, III and IV during summer are referred to anaerobic degradation; on other hand stations I, V, VI and VII and stations V, VI, VII and VIII during spring and summer, respectively, showed aerobic degradation. Observation data referred to the aerobic and anaerobic degradation processes as the main pathway for DDT degradation in the study area during the present study.

DDE + DDD)/∑DDTs ratio is used to assess the possible modern/recent input past input pollution sources of DDT. In the present study, as illustrated in Fig (4), (DDE + DDD)/∑DDTs ratios ranged between 0.22 at station I and 0.98 at station II during spring, while during summer, a range between 0.52 at station VII and 0.77 at station II was recorded. Observed values in all stations during spring and summer > 0.5, except stations I, VI and VII during spring that recorded a value > 0.5, suggesting past input for DDT.

The o,p′-DDT/p,p′-DDT ratio is used to determine the source of origin of DDT, which may be technical grade DDT or from dicofol-type DDT. Dicofol is an organochlorine pesticide that is synthesized from technical DDT and is commonly used as a miticide pesticide, especially against spider mites **(Schwarzbach, 1991)**. During the present study, as shown in Fig. (4), the o,p′-DDT/p,p′-DDT ratio fluctuated between 0.15 at station I and 18.47 at station VII during spring, and between 0.25 at station VIII and 3.08 at station I during summer; stations I & VIII in addition to station VIII during spring and summer, respectively, recorded technical grade DDT, while the remaining stations recorded dicofol-type DDT.

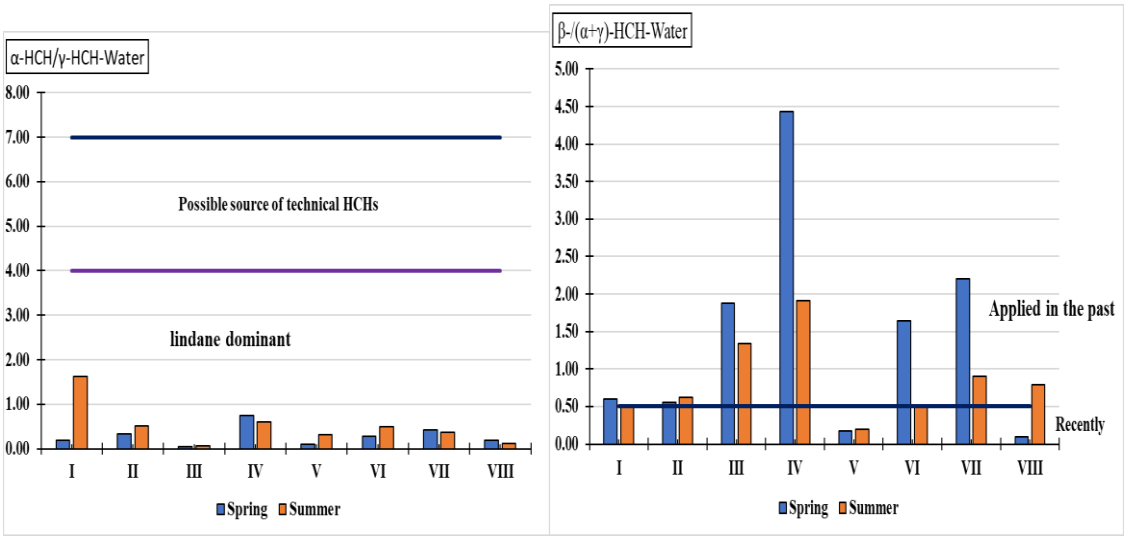
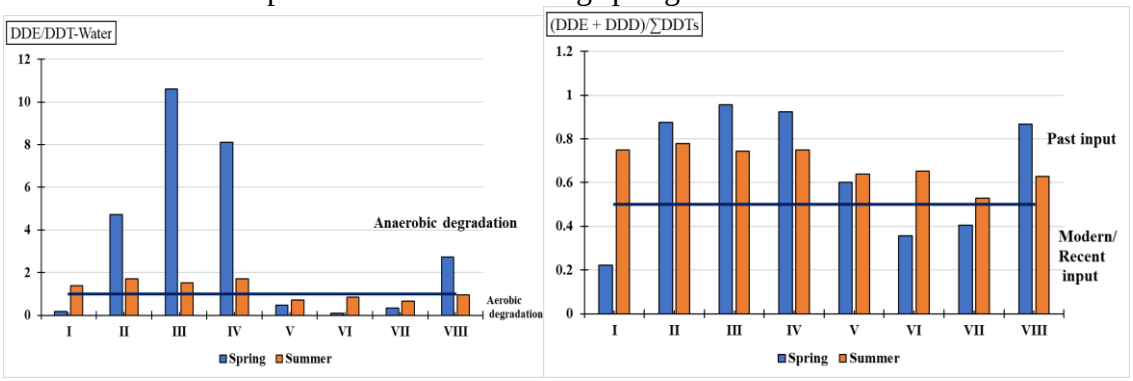


Fig. 3. Hexachlorocyclohexane ratio (α-HCH/γ-HCH, and β-/(α+γ)-HCH) for water samples of Lake Edku during spring and summer 2022



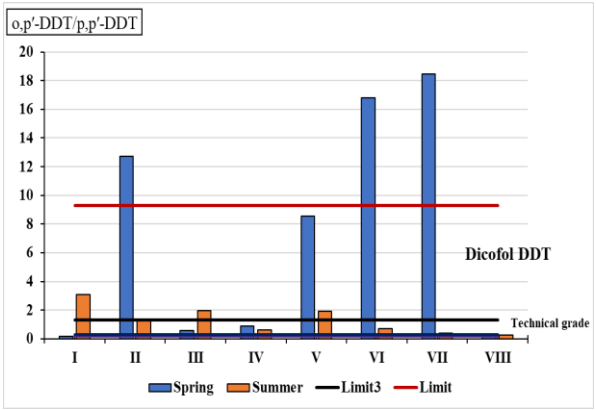


Fig. 4. (DDE + DDD)/∑DDTs, o,p′-DDT/p,p′-DDT and DDE/DDT ratio for water samples of Lake Edku during spring and summer 2022

Toxicity and ecological risk assessment

Ecological risk assessment was applied to detect the toxicity induced by organochlorine pesticides (OCPs) based on two methods (1) RQ, as illustrated in bellow equation:

$$RQ=EEC\times PNEC$$

Where, RQ: risk quotient; EEC concentration of each OCP (mg/L) from the present study, and PNEC: the predicted no-effect concentration for a particular OCPs (mg/L) according to **Khadhar *et al.* (2018)**.

Predicted No effect concentration (PNEC) values according to **Di Lorenzo *et al.* (2018)** were assessed as follows: o,p DDD (0.0071), o,p DDE (0.0015), p,p DDE (0.0007), o,p DDT (0.0013), p,p DDT (0.0098), α-HCH (0.1571), β-HCH (0.1571), γ -HCH (0.0163), Aldrin (0.0312), Dieldrin (0.0054). Risk quotients ranking as $RQ < 0.01$ (very low risk), $0.01 \leq RQ < 0.1$ (low risk), $0.1 \leq RQ < 1$ (moderate risk), $1 \leq RQ < 10$ (high risk), $RQ \geq 10$ (very high risk) **(Peng *et al.*, 2014; Yu *et al.*, 2014)**.

Second methods by comparing the results with EPA water quality criteria; Criteria Maximum Concentration (CMC) and Criterion Continuous Concentration (CCC) for (γ-HCH , Aldrin, Dieldrin, Endrin, p,p'-DDT, PCBs) as (0.95, 3, 0.24, 0.086, 1.1, and 0.014 μg/L) and (0.16, 1.3, 0.56, 0.036, 0.001, and 0.03 μg/L) respectively; WHO for (γ-HCH, Dieldrin, and p,p'-DDT) as (2, 0.03, and 2 μg/L) based on **(EPA, 2000)**.

As shown in Fig. (5), the RQ of the studied organochlorine pesticides (OCPs) most of which are more than 10, demonstrating a very high risk, except for α-HCH that recorded a moderated risk at stations III, V and VIII during spring, and station III during summer. Whereas, a very high risk was detected at stations I, II, IV, VI and VII during spring and at stations I, II, IV, V, VI, VII and VII during summer. On the other hand, β-HCH recorded a moderated risk at station VIII during spring and recorded a high risk at stations I and VIII during spring and stations I, V and VI during summer. Upon comparing the current results with water quality standards, it was noted that, γ-HCH during spring and summer recorded values higher than criteria continuous concentration (CCC); while during spring, stations I, II, III and V recorded values higher than the criteria maximum concentration (CMC), and other stations recorded a low value. It is worthy to mention that, during summer all stations recorded values higher than the criteria maximum concentration, (CMC) excepted for station I. Aldrin in stations II, III, IV and V recorded values higher than criteria continuous concentration (CCC) during spring and summer; stations I, III, IV and V recorded values higher than the criteria maximum concentration (CMC) during spring and summer, respectively. Endrin during spring and summer in all stations recorded values higher than the criteria maximum concentration (CMC) and criteria continuous concentration (CCC). In the same case for PCBs, all stations recorded values higher than criteria maximum concentration (CMC) and criteria continuous concentration (CCC). For Dieldrin, almost all stations recorded values higher than criteria maximum concentration (CMC) and criteria continuous concentration (CCC), except for station IV during spring and station VIII during summer. While for p,p'-DDT, all stations recorded values higher than CMC; station (I) during summer and stations IV and VII during summer recorded values higher than CCC, as illustrated in Fig. (5).

Table 2. Sources of OCPs and PCBs in surface water

Compound	Ratio	Indicate	Reference
α-HCH/γ-HCH	04:07	possible source of technical HCHs	**(Iwata *et al.*, 1993, 1995; Botwe *et al.*, 2017; Hao *et al.*, 2019; Malhat *et al.*, 2021)**
	Higher than 7	Atmospheric source	
	lower than 4	lindane dominant	
β-/(α+γ)-HCH	lower than 0.5	HCH and/or lindane are used recently	**(Liu *et al.*, 2012; Sari *et al.*, 2020)**
	Higher than 0.5	Applied in the past	
DDD/DDE	less than one	aerobic degradation	**Hidayati *et al.*, (2021)**
	higher than one	Anaerobic degradation	
(DDE+DDD)/ ∑DDTs	less than 0.5	Modern/recent input	**(Neves *et al.*, 2018; Tran *et al.*, 2019)**
	higher than 0.5	Past input	
o,p′-DDT/p,p′-DDT	0.2 to 0.3	Technical grade	**(Qiu *et al.*, 2005; Aamir *et al.*, 2017)**

Statistical analysis

1. Principal component analysis (PCA)

Principal component analysis (PCA) for OCPs and PCBs pollutants under examination were determined using Origin Lab 2019 program; two principal components were extracted and illustrated as follows:

- **First component (PCI).** This factor represented 41.55% during spring and 57.77% during summer of the total variance, as illustrated in Fig. (6). During spring, all stations recorded strong positive loading plots reaching 0.34, except for o,p-DDT, p,p-DDT, PCB 118, PCB 138 and PCB 153, which recorded a negative loading; during summer they reached 0.29, except β-HCH and γ-HCH, which recorded a negative loading. The first component may be suggested due to anthropogenic activities and sources of pollution from POPs including the disposal of industrial chemicals and agrochemicals **(Thakur & Pathania, 2020)**.
- **Second component (PCI).** The second factor that can suggest that these compounds could be transferred to the lake from the drains, and fish farms that are accumulated and deposited of it in the lake. The second factor recorded 19.52% and the loading plot reached 22.12 during spring; 13.94% and the loading plot reached 0.56 during summer, respectively; during spring the following pollutants β-HCH, o,p-DDE, p,p-DDE, o,p-DDD, o,p-DDT, p,p-DDT, PCB 28, PCB 101, PCB 118, PCB 153; and β-HCH, Aldrin, Endrin, o,p-DDE, p,p-DDE, o,p-DDD, p,p-DDD, p,p-DDT, PCB 28, and PCB 52 during summer; these compounds can represent the second factor, as illustrated in Fig. 6).

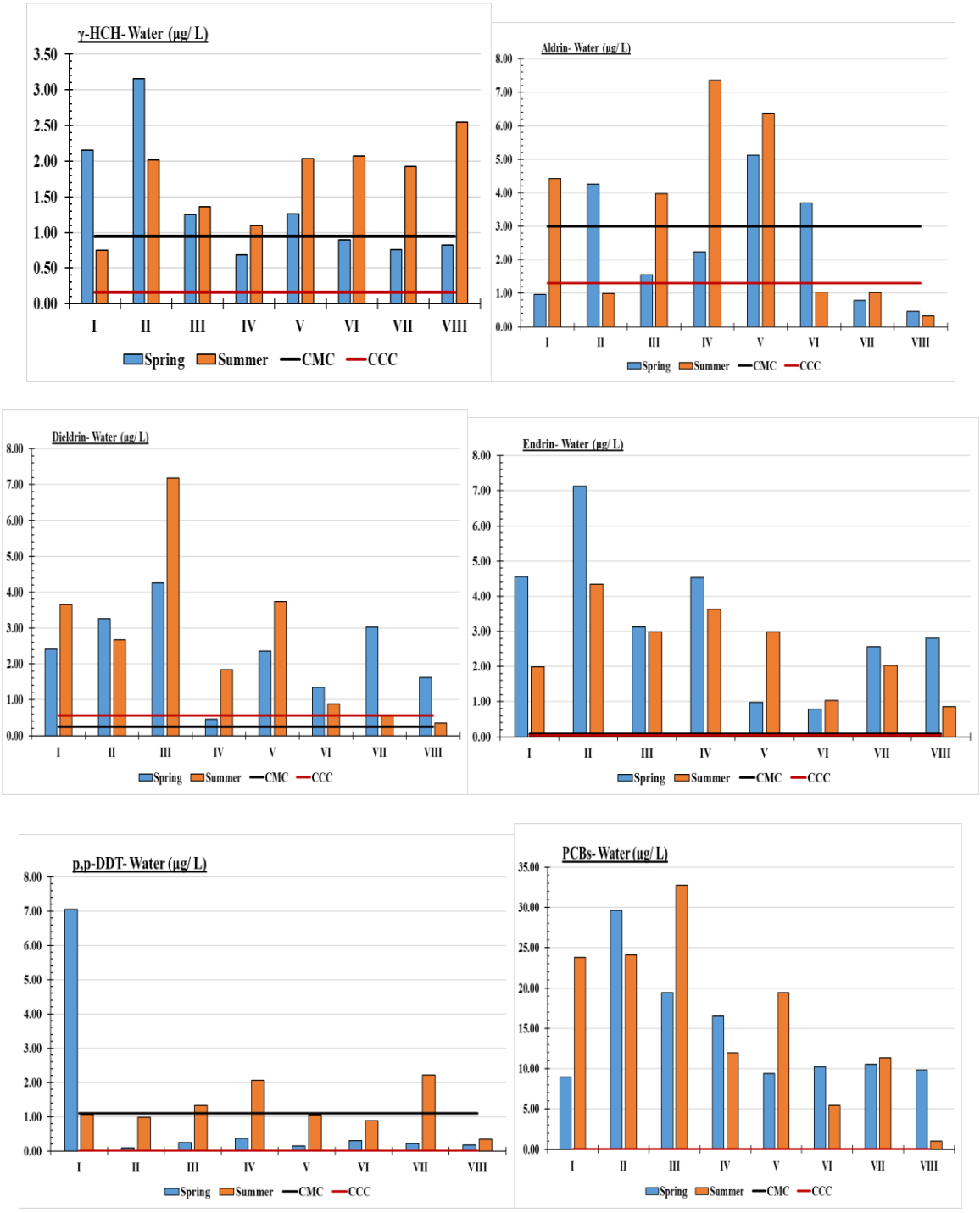


Fig. 5. Comparing the results with EPA water quality criteria for OCPs and PCBs toxic pollutants

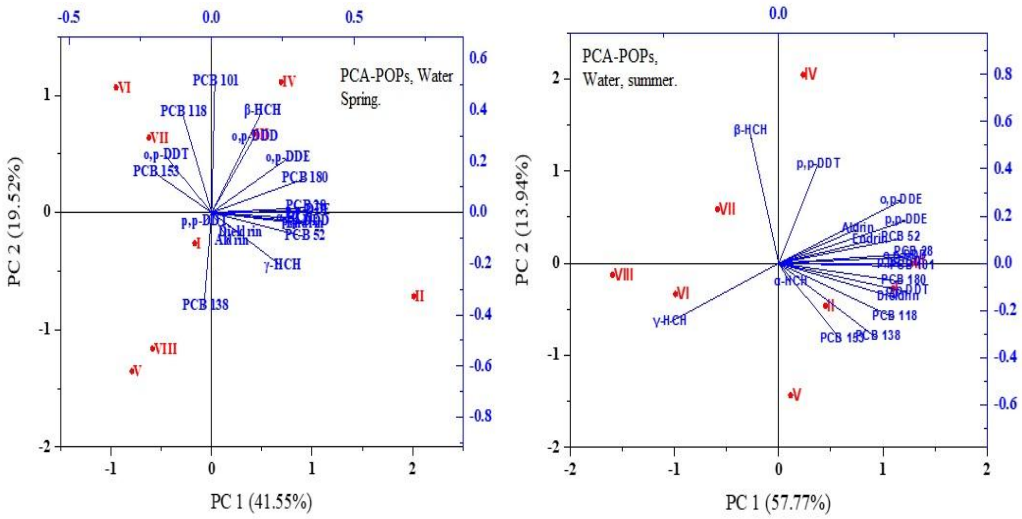


Fig. 6. Loading matrix of the first two principal components of POPs (OCPs and PCBs) in water samples from Lake Edku during spring and summer 2022

2. Hierarchical cluster analysis (HCA)

- **Single linkages.** The results of hierarchical cluster analysis (HCA) during spring and summer are illustrated in Fig. (7). Three principal clusters were detected during spring recording seven stages as follows: stage (1) with a distance of 5.18 between stations 6 and VII, stage (2) with a distance of 5.58 between stations 5 and VIII, stage (3) with a distance of 6.57 between stations 5 and VI, stage (4) with a distance of 6.72 between stations 3 and IV, stage (5) with a distance of8.23 between stations 3 and V, stage (6) with a distance of8.63 between stations 1 and III, and stage (7) with a distance of11.91 between stations 1 and II. During summer, HCA was noted as follows: stage (1) with a distance of 2.83 between stations VI and VIII, stage (2) with a distance of 3.53 between stations VI and VII, stage (3) with a distance of 5.89 between stations I and II, stage (4) with a distance of 6.76 between stations I and VI, stage (5) with a distance of 6.85 between stations I and III, stage (6) with a distance of 7.48 between stations I and V, stage (7) with a distance of 7.62 between stations I and IV, as illustrated in Fig. (7). Station VII recorded the most representative observation, and stations II and III recorded the least representative observation during spring and summer respectively.

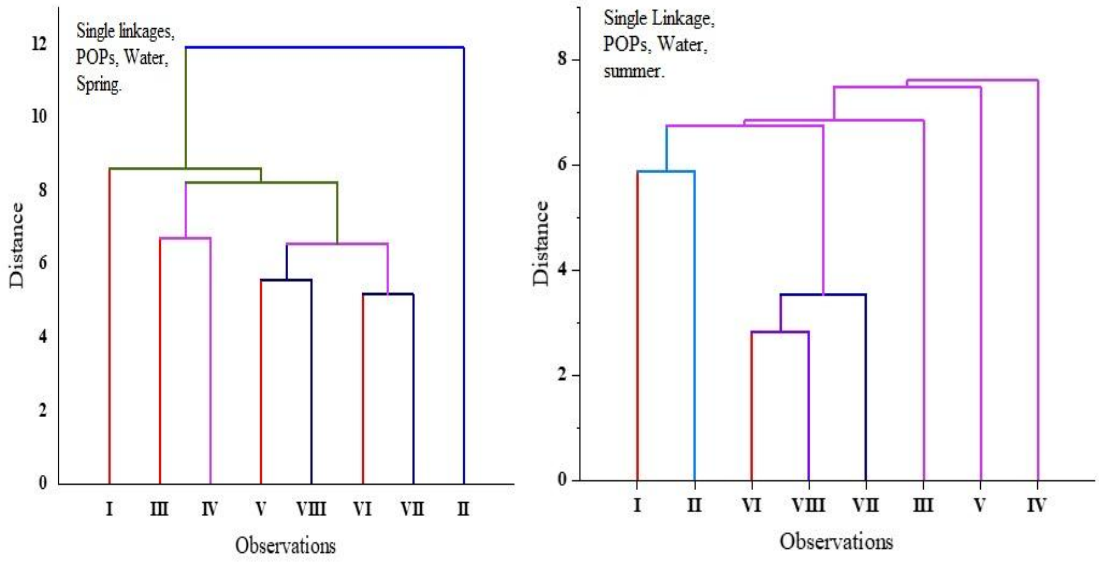


Fig. 7. Hierarchical cluster analysis dendrogram (single linkage) showing the spatial distribution of POPs (OCPs and PCBs) among different sampling sites in water from Lake Edku during spring and summer 2022

- **Complete linkage.** For the complete linkage records, stage (1) was recorded with a distance of 0.79 between α-HCH and p,p-DDD; stage (2) with a distance of 1.29 betweenα-HCH and PCB 180; stage (3) with a distance of 1.95 between γ-HCH and p,p-DDE; stage (4) with a distance of 2.47 between α-HCH and PCB 153; stage (5) with a distance of 2.57 between β-HCH and PCB 101; stage (6) with a distance of 3.06 between o,p-DDD and PCB 118; stage (7) with a distance of 3.54 between α-HCH and γ-HCH , and stage (8) with a distance of 3.59 between β-HCH and o,p-DDD, while stage (9) had a distance of 3.84 between β-HCH and o,p-DDE; stage (10) with a distance of 4.76 between α-HCH and β-HCH; stage (11) with a distance of 5.03 between Dieldrin and PCB 138; stage (12) with a distance of 5.44 between Aldrin and Dieldrin; stage (13) with a distance of 5.95 between Endrin and PCB 52; stage (14) with a distance of 6.06 between α-HCH and o,p-DDT; stage (15) with a distance of 6.23 between α-HCH and Aldrin; stage (16) with a distance of 7.72 between α-HCH and p,p-DDT; stage (17) with a distance of 8.08 between Endrin and PCB 28, and stage (18) with a distance of 10.75 between α-HCH and Endrin. During summer as illustrated in Fig. (6), the HCA showed results as follows: stage (1) with a distance of 1.16 between p,p-DDD and o,p-DDT; stage (2) with a distance of 1.44 between o,p-DDE and p,p-DDE; stage (3) with a distance of 1.78 between Dieldrin and PCB 118; stage (4) with a distance of 1.85 between o,p-DDD and PCB 101; stage (5) with a distance of 2.02 between p,p-DDD and PCB 180; stage (6) with a distance of 2.17 between o,p-DDE and p,p-DDD; stage (7) with a distance of 2.44 between α-HCH and p,p-DDT; stage (8) with a distance of 2.62 between o,p-DDE and o,p-DDD; stage (9), with a distance of 2.81 between PCB 28 and PCB 52; stage (10) with a distance of 2.88 between β-HCH and γ-HCH; stage (11) with a distance of 3.39 between α-HCH and β-HCH; stage (12) with a distance of 3.45 between

Dieldrin and PCB 138; stage (13) with a distance of 3.50 between o,p-DDE and PCB 153; stage (14) with a distance of 3.75 between Endrin and o,p-DDE; stage (15) with a distance of 4.08 between α-HCH and Endrin; stage (16) with a distance of 4.57 between Dieldrin and PCB 28; stage (17) with a distance of 6.16 between α-HCH and Dieldrin, and stage (18) with a distance of 7.97 between α-HCH and Aldrin. p,p-DDE recorded the most representative observation during both seasons, and PCB 28 and Aldrin recorded the least representative observation during spring and summer, respectively, as shown in Fig. (8).

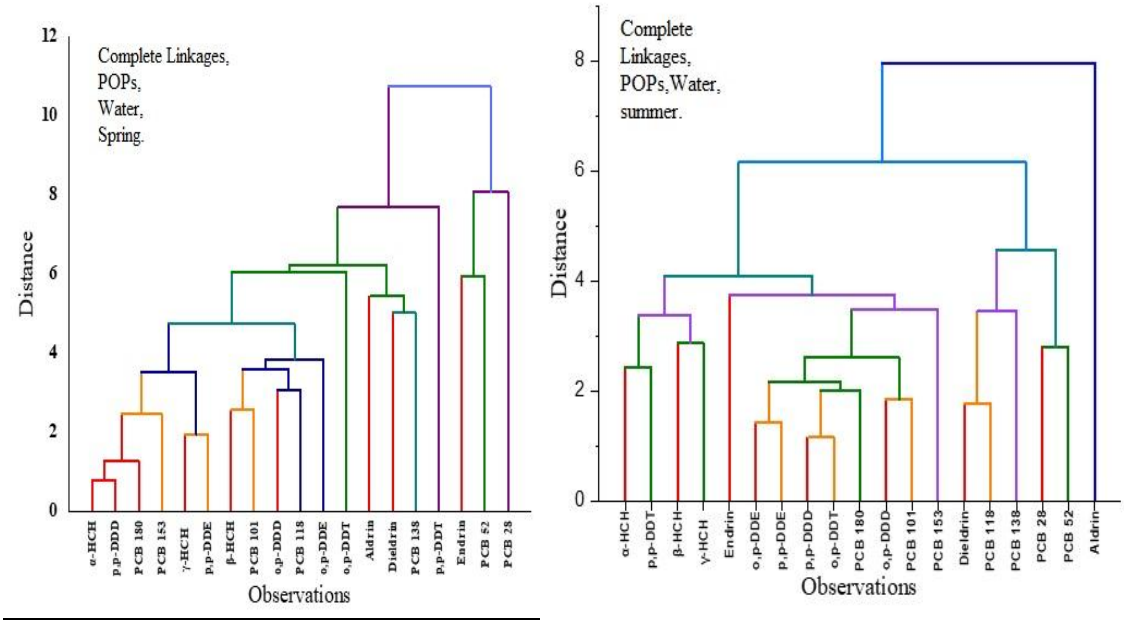


Fig. 8. Hierarchical cluster analysis dendrogram (complete linkage) between group for POPs (OCPs and PCBs) in water samples from Lake Edku during spring and summer 2022

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