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Determination of Organochlorine Pesticides in Sediments Using Gas Chromatography and Mass Spectrometry

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Abstract

Seventeen Organochlorine Pesticides (OCPs) were evaluated in 14 surface sediment samples from a dam lake in Northwestern Turkey. As analytical tool GC-Mass system; HP (Hewlett Packard) 6890 series gas chromatograph coupled with HP 5973 mass spectrometer was used. The HP 5MS capillary column had 30 m length with 0.32 mm internal diameter. A 0.25 mm film thickness cross-linked with stationary phase of 5% Phenyl methyl siloxane and Ultra-pure Helium gas was used as mobile phase. Ultrasonic bath extraction method was applied and cleaned up process were carried up with anhydrous Na_2SO_4 and Florisil column. Total pesticides concentrations were ranged from=0.237-2.39 mg/kg for dry weight. Percent total organic carbon (TOC) were observed between 1 and 3%. Average total OCP concentrations was 58.00 ± 45.44 mg/kg. The total concentrations of OCPs in sediment samples ranged from 12.9 to 169.9 mg/kg, with a mean value of 58.00 mg/kg. Although organochlorine pesticides have been banned in Turkey still the residues can be seen in sediment samples indicating the use of prohibited pesticides in the country. Comparison of organochlorine pesticides concentrations in sediment samples with other lakes in Turkey implies the higher concentration therefore higher usage of synthetic chemicals.

Keywords: Organochlorine pesticides; Persistent organic pollutants; Gas chromatography-mass spectrometry; Pollutants

Introduction

The concerened about OCPs on environment and human health rised up last four decades. The Stockholm Convention on persistent organic pollutants (POPs) ratified by many countries in order to reduce the use of OCPs [1]. In the period of 1940s-1980s a wide range of application seen in the field of agriculture, domestic and public health sectors [2,3]. The OCPS residues are still seen due to their high photochemical, biological and chemical resistance to degradation in the environment [4]. OCPs are toxic, environmentally persistent and able to undergo long range environmental transport [5-8]. Organochlorine pesticides are commonly classified in persistent organic pollutants as a result of high chemical stability, poor water solubility and low vapor pressures in the environment [9]. Due to higher hydrophobicity, OCPs can adsorb to sedimentary surfaces which leds to accumulate in estuaries and lakes [10], and they may be transferred to food chain. These synthetic chemicals are banned by many countries starting from 1970s and still are used in some developing countries [11]. Turkey signed the Stockholm Convention on Persistent Organic Polluters in 2001 in order to provide the withdrawal and reduction of the releases of some organochlorine pesticides including DDT, aldrin, endrin, dieldrin, heptachlor and hexachlorobenzene [12]. However, these pollutants still exist in the environment of Turkey [13,14] Distribution and sources of organochlorinated contaminants in sediments from Izmir Bay [15]. The studies on pesticides residue are elevating in Turkey day by day. Some of these researches are conducted with the samples from central Anatolia [16], along the coast of mid Black Sea [17], from Uluabat Lake [18], from Meriç Delta [19]. The results show a different variation due to different times and different places, vary in terms of concentration.

Pesticide usage has soared in past decades around the world, 21 different types of organophosphates, organochlorines and carbamates insecticides available in the market to boost agricultural production and combat disease are banned [20]. Some researches on pesticides have estimated the potential risk to human health [21-25] which arised from the consumption of pesticide contaminated food in dietary intake. The nondietary exposures of pesticides effects should also be researched. Model studies should be conducted to understand the risks which include the chronic daily intake (CDI) [26].

Experimental

Study area and sampling

Balıkesir is among one of the cultivated area in Turkey. Agricultural activities are high and the sources of income are from olive production. Besides cereals, sugar beets, tomatoes, melons and cotton. The farmer is trying their best to increase the yield and the over use of pesticides is unavoidable. The study area which is a dam lake called İkizcetepeler, was an agricultural field before the dam was constructed in 1991. The dam lake water is major source for drinking and irrigation. İkizcetepeler Reservoir, is only 25 km far away from the city center. The area is under the influence of the urban pollution. The dense traffic on Balıkesir-İzmir-Bursa link highway passing near the dam lake, and the industrial zone of the city pollute the dam lake. Figure 1 shows the sampling area and the Geographical Positioning System (GPS) image of the Dam Lake. The dam lake should be under controlled since the water is used for drinking and irrigation. The sampling was done by grids of 0.123 km^2 and total 44 sampling points were determined. Fourteen samples were used for OCPs determination to figure out the OCP pollution distribution. The samples were collected by Van Veen grab sampler, an amount of 200-500 grams collected and were stored in a refrigerator

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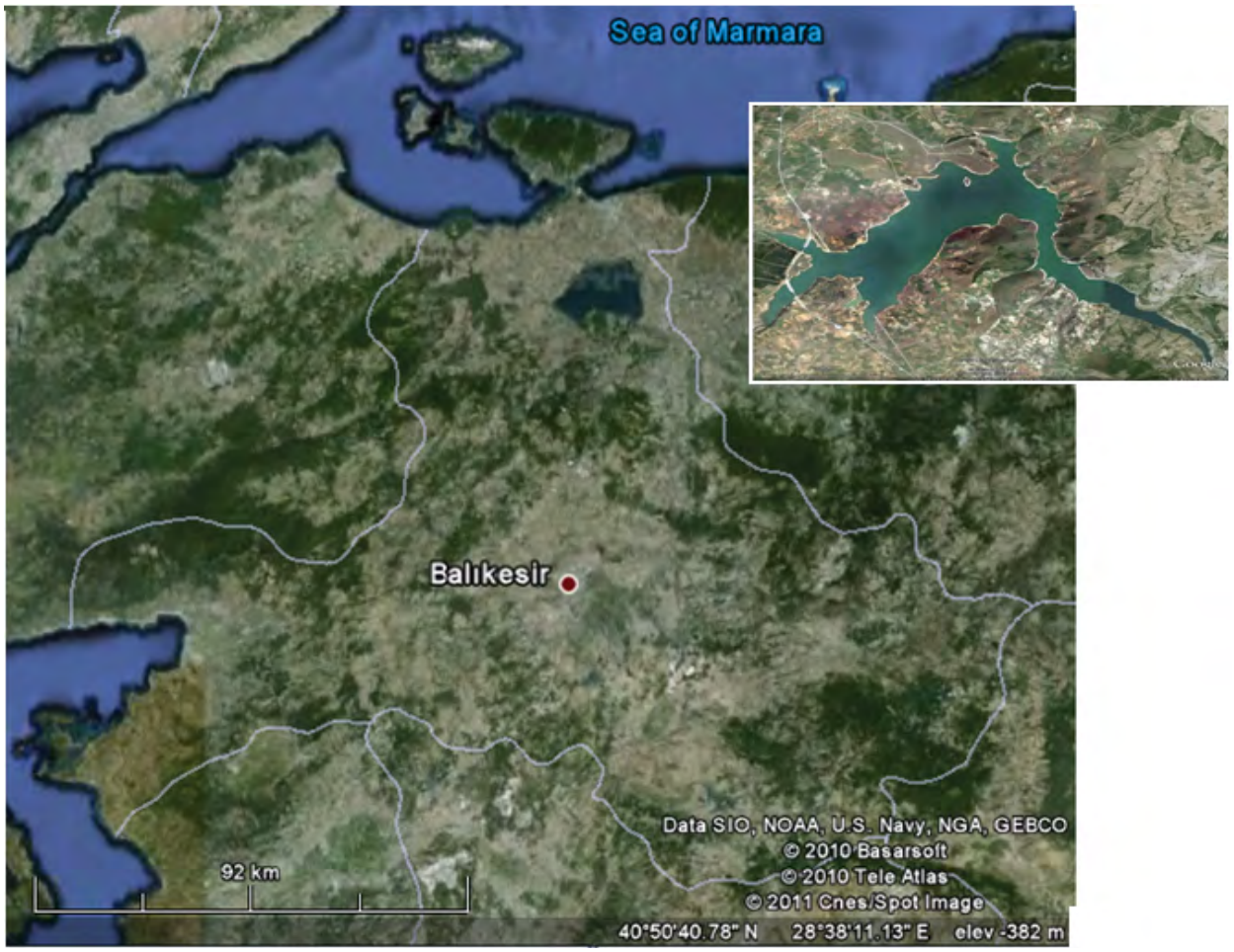

Figure 1: The sampling area and the GPS image of the Dam Lake.

at -20°C in nylon bags, dark medium was preferred to prevent the samples from the sunlight.

Materials and methods

All the solvents and standards used in the experiments were extra pure analytical grade. The organochlorine pesticide standards (EPA Method 508-Chlorinated Pesticide Mix 1, 1000 µg/ml), internal standards (Accustandard, Pentachloronitrobenzene, 1.0 mg/ml) and surrogate standards (2,4,5,6-Tetrachloro-m-xylene, 10 ng/µl and Decachlorobiphenyl, 0.5 mg/ml) were purchased from Dr. Ehrenstorfer (Ausburg, Germany. Standard reference materials (SRM 2261 and SRM 2275) were purchased from National Institute of Standards (NIST).

The materials used for the extraction were cleaned. The sodium sulfate, Florisil were pre-cleaned with hexane and acetone solvents by passing through glass column and then they were conditioned in an oven at 400°C for 4 h. For column packing glass wool was used, it was pre-cleaned with hexane and acetone then were conditioned overnight in an oven at 200°C.

All of the glassware used in laboratory were cleaned with Alconox detergent (Supelco) in hot water and deionized water. They were left to dry in an oven after rinsing with hexane and acetone at 100°C.

Analytical procedures

Sediments samples are dried in room condition and grinned with mortar. Two grams of sediment samples were weighed in an amber glass bottle with Teflon cap and 100 µL of 1.0 µg/L surrogate standards (2,4,5,6 Tetrachloro-m-xylene and Decachlorobiphenyl) were added. Sixty milliliters of hexane:acetone mixture (3:1) was added to the bottle and closed for ultrasonic bath extraction for two hours. Then, the extracts were firstly eluted through Na_2SO_4 column to eliminate the water moisture and then cleaned up with Florisil column to remove the polar compounds that interfere with the analytes. The mixture was evaporated with rotary evaporator and 100 µL internal standard (Pentachloronitrobenzene) having a concentration of 0.5 µg/L was added. The final solution was pre-concentrated to 1 mL with a minivap evaporator under pure nitrogen gas and taken into a 2 ml amber glass vial and kept in refrigerator at 4°C before the analysis with GC-MS [27].

Surrogate standards, internal standards and standard reference materials were used for Quality assurance /Quality control studies. An HP (Hewlett Packard) 6890 series gas chromatograph coupled with HP 5973 mass spectrometer was used for the analysis. A 30 m, 0.32 mm id., 0.25 mm film thickness, crosslinked 5% Phenyl methyl siloxane, HP 5MS, capillary column (Agilent Tech.) was used for the separation of OCPs throughout the study.

For Total Organic Carbon studies Method 5310 was applied to the sediment samples. Procedure is as follows; 25 grams of the sediment sample was treated with 25 ml 0.1 M H_2SO_4 for 2 hours on hot plate. The samples then dried in oven at 200°C for 4 hours.

Results and Discussion

Organochlorine pesticides in sediments

Dry weight concentrations of the organochlorine pesticides were determined from sediment samples and results are shown in Table 1. In this table, the concentrations that are below limit of detection are mentioned as n.d. (not detectable). β-HCH and p-p'-DDT was found in all fourteen samples. Whereas, Heptachlor Epoxide was not observed in any samples. Sample 8 was located in the middle of sampling area and contain all analytes except p-p'-DDD. It was concluded is the most contaminated position with fifteen organochlorine pesticides. This sampling point was in the middle of the lake. Likewise sampling points 2, 4 and 14 are in the middle region and contain nearly all of the pesticides in the sediment samples. Total pesticides concentration in these sediments samples were ranged from 236.82-2388.59 µg/kg for dry weight. The highest concentration were seen in sample 8 and 11 with 2159.96 and 2388.59 µg/kg.

The pollution contains natural and anthropogenic origin sources beside organics synthetic also observed, most natural waters and sediment samples contain organic substances with natural origin (Env. Chem. Analysis). The toxicity of the contaminants and the concentrations of these organic substances have been found to be well correlated [28]. However, it is not correlated with the dry weight concentration of the chemicals present in sediments. Therefore, the Sediment Management Standards criteria for organic chemicals have been set on an OC-normalized basis. The treatment of the sediment samples for Total Organic Carbon analysis was explained in the Experimental section. According to this treatment, OC normalization is applied to sample by sample basis since TOC values vary from station to station. The data set for the TOC concentrations was given in Table 2.

Standard values for TOC is different for every environmental medium. The values greater than 3% are common satiated as near shore sedimental environment. This value is higher in boggy areas and other wetland environments. The contamination with the organic substances with artificial factors such as sewage, petroleum hydrocarbons, wood chips raise the percent TOC values. Sandy and erosional areas or areas with fast flowing currents such as rivers have percent TOC values smaller than 1%.

From the Table 2 it can be seen that except sampling points 10 and 13 the %TOC values in the sediments are between 1 and 3%.

Total pesticide concentrations and %TOC values show no significant correlation therefore this suggests that the distributions of these organochlorine pesticides in the lake are not controlled by the distribution of organic matter. This situation also shows that the TOC and pesticides have different sources.

The dry weight concentrations are converted to OC-normalized concentrations via dividing the mg/kg dry weight concentrations by percent TOC values as shown in the following equation:

$$mg/kg\ OC = \frac{mg/kg\ dry\ weight}{kg\ TOC/kg\ dry\ weight}$$

Where, mg/kg OC=milligrams of the chemical per kilogram of organic carbon; mg/kg dry weight=milligrams of the chemical per kilogram of dry weight sample; kg TOC/kg dry weight=percent

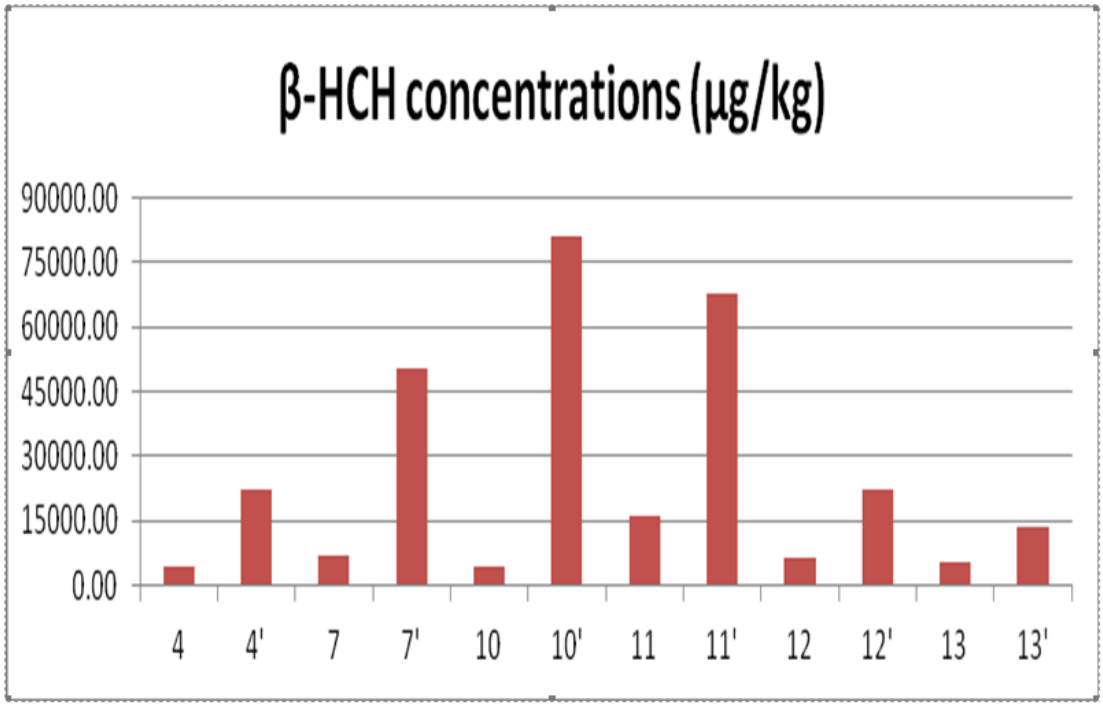

Figure 2: Comparison of dry weight concentrations and OC-normalized concentration of β-HCH at selected sampling points. The points with a "prime" sign show the concentrations after the TOC correction and dry weight concentrations were multiplied by 10 to obtain values in similar scale.

Pesticide / Sample	1	2	3	4	5	6	7	8	9	10	11	12	13	14
α-HCH	0.68	0.44	n.d.	n.d.	0.29	0.48	3.10	9.68	0.40	1.34	11.36	3.30	n.d.	4.71
β-HCH	26.07	74.00	115.44	450.49	138.72	23.15	698.19	196.69	152.46	421.49	1630.00	621.52	541.87	349.63
γ-HCH	2.88	n.d.	n.d.	88.58	3.39	1.82	n.d.	157.45	n.d.	n.d.	n.d.	n.d.	4.50	11.18
δ-HCH	52.35	62.81	n.d.	12.34	73.73	n.d.	153.15	56.53	n.d.	n.d.	221.39	n.d.	n.d.	n.d.
p,p'-DDD	n.d.	n.d.	n.d.	n.d.	1.53	0.07	n.d.	n.d.	n.d.	0.88	n.d.	2.01	2.74	6.90
p,p'-DDE	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	22.77	n.d.	0.11	n.d.	0.15	n.d.	9.05
p,p'-DDT	34.91	39.42	49.57	48.55	65.23	65.07	259.94	30.25	20.04	49.13	289.04	46.22	20.71	32.56
Methoxychlor	0.16	25.87	n.d.	1.49	n.d.	0.47	2.54	1.75	2.36	n.d.	78.19	38.30	2.14	0.97
Aldrin	n.d.	n.d.	n.d.	0.71	n.d.	n.d.	n.d.	282.95	n.d.	n.d.	6.00	n.d.	n.d.	102.24
Dieldrin	3.59	19.26	0.33	17.44	n.d.	n.d.	67.13	508.64	n.d.	6.33	57.57	17.08	n.d.	242.93
Endrin	n.d.	1.86	1.90	2.44	n.d.	n.d.	n.d.	305.74	n.d.	1.00	2.19	2.89	n.d.	243.99
Endrin Aldehyde	n.d.	n.d.	n.d.	96.70	n.d.	n.d.	n.d.	29.63	n.d.	n.d.	60.57	n.d.	n.d.	n.d.
Heptachlor	n.d.	14.25	168.70	606.17	2.81	1.16	10.47	18.73	0.30	3.12	13.23	3.29	n.d.	3.69
Endosulfan	4.63	1.34	n.d.	4.85	11.06	1.22	n.d.	91.51	3.36	9.49	19.04	n.d.	n.d.	n.d.
Endosulfan II	n.d.	n.d.	26.40	17.32	n.d.	n.d.	n.d.	23.14	11.14	n.d.	n.d.	n.d.	50.75	n.d.
Endosulfan sulfate	232.78	237.48	74.58	176.94	365.26	143.40	n.d.	421.46	259.13	390.33	n.d.	121.94	194.74	139.74
Total	358.06	476.72	436.90	1524.02	662.04	236.82	1194.52	2156.91	449.18	883.32	2388.59	856.68	817,46	1147.62

Table 1: Concentrations of the organochlorine pesticides according to the sampling points (µg/kg, dry weight).

Sampling Point	%TOC
1	2.01
2	2.22
3	1.97
4	2.02
5	1.42
6	1.83
7	1.38
8	2.03
9	1.27
10	0.52
11	2.4
12	2.81
13	4.01
14	1.71

Table 2: The total organic Carbon for fourteen samples.

total organic carbon in dry weight sample (expressed as decimal; for example, 1% TOC=0.01).

The concentrations of the pesticides in the samples showed significant change after the OC normalization was applied. Total sediment concentration ranged from 12941.20 µg/kg, OC normalized (sampling point 6) to 169850.81 OC normalized (sampling point 10) were given in Table 3.

The dry weight concentration and TOC corrected concentrations were compared for pesticides. The results were given in the Figure 2. Sampling point with higher TOC percentage and samller dry weight concentration becomes fairly important. When we compare with a group of pesticides. The individual pesticide corrected of TOC will be smaller. Sample 10 Beta-HCH concentration is not significant without TOC corrected. However after the TOC correction it results became fairly high concentration.

The data set obtained in whole study period for Organochlorine Pesticides (OCPs) in 14 sediment samples are displayed in Table 4. The values presented are for the concentrations above the Limit of Quantification, which is determined as the concentration at S/N value of 10. Heptachlor epoxide concentration was found below limit of quantification for all of the samples therefore not shown in the Table 4. Injection of each fourteen sample solutions was performed as three replicates in order to evaluate the precision of the data set. The average, minimum and maximum concentrations and standard deviations of organochlorine pesticides are given in Table 4. According to this table, beta- HCH is the prominent pesticide in the lake and Endosulfan Sulfate, Aldrin, Heptachlor and Dieldrin have the highest concentrations among the other organochlorine pesticides.

The standard deviations of the concentrations between the samples of the different sampling stations are high, since the pollution level may not be the same in different locations due to different sources effective near the environment of the study area.

Average total OCP concentration in İkizcetepeler sediment Samples Lake is 58.00 ± 45.44 mg/kg. The concentration distribution of sampling stations is shown in Table 5. Two OCPs (β-HCH, and p,p-DDT) were detected at all 14 sites. The total concentrations of OCPs in sediment ranged from 12.94 to 169.87 mg/kg, with a mean value of 58.00 mg/kg. The greatest concentration was observed at site 10. Among OCPs, residues of total HCHs (ΣHCHs, the summation of α-, β-, γ- and δ-HCH), DDTs (ΣDDTs, summation of p,p'-DDD, p,p'-DDE and p,p'-DDT), total cyclodienes (Σ Cyclodienes, summation of Aldrin, Dieldrin, Endrin, Endrin Aldehyde, Heptachlor)and total endosulfan derivatives (Σ Endosulfans summation of Endosulfan, Endosulfan II, Endosulfan sulfate) in sediment varied from 1.39 to 81.31 µg/g, 0.64 to 19.02 µg/g, 0.02 to 34.67 µg/g, 0.79 to 76.89 µg/g, respectively. Among cyclodienes, aldrin and heptachlor have the highest concentration in their subgroup. Among DDTs, p, p'-DDT is the prominent derivative.

Pesticides / samples	1	2	3	4	5	6	7	8	9	10	11	12	13	14
α-HCH	33.85	19.90	n.d	n.d.	20.70	26.29	224.30	476.76	31.26	257.47	473.43	117.44	n.d.	275.69
β-HCH	1297.09	3333.22	5859.75	22301.52	9769.02	1264.90	50593.67	9689.32	12004.62	81055.02	67916.60	22118.08	13513.06	20446.18
γ-HCH	143.34	n.d.	n.d.	4385.00	238.77	99.52	n.d.	7756.37	n.d.	n.d.	n.d.	n.d.	112.23	654.07
δ-HCH	2604.68	2829.10	n.d.	610.68	5192.34	n.d.	11097.91	2784.70	n.d.	n.d.	9224.41	n.d.	n.d.	n.d.
***p,p'*-DDD**	n.d.	n.d.	n.d.	n.d.	108.04	3.76	n.d.	n.d.	n.d.	170.15	n.d.	71.48	68.45	403.64
***p,p'*-DDE**	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	1121.59	n.d.	20.49	n.d.	5.43	n.d.	529.52
***p,p'*-DDT**	1736.69	1775.65	2516.07	2403.55	4593.98	3555.75	18836.09	1490.18	1577.59	9447.91	12043.14	1644.68	516.42	1904.22
Methoxychlor	8.17	1165.09	n.d.	73.91	n.d.	25.58	183.77	86.21	186.05	n.d.	3257.92	1362.90	53.42	56.98
Aldrin	n.d.	n.d.	n.d.	35.36	n.d.	n.d.	n.d.	13938.46	n.d.	n.d.	250.11	n.d.	n.d.	5979.12
Dieldrin	178.36	867.60	16.50	863.30	n.d.	n.d.	4864.76	25055.94	n.d.	1217.93	2398.90	607.67	n.d.	14206.72
Endrin	n.d.	83.73	96.21	120.73	n.d.	n.d.	n.d.	15060.94	n.d.	192.54	91.31	102.73	n.d.	14268.14
Endrin Aldehyde	n.d.	n.d.	n.d.	4787.02	n.d.	n.d.	n.d.	1459.46	n.d.	n.d.	2523.96	n.d.	n.d.	n.d.
Heptachlor	n.d.	641.94	8563.49	30008.47	198.22	63.14	758.65	922.60	23.98	600.01	551.34	116.98	n.d.	215.98
Endosulfan	230.19	60.36	n.d.	239.88	778.99	66.43	n.d.	4507.97	264.35	1825.40	793.51	n.d.	n.d.	n.d.
Endosulfan II	n.d.	n.d.	1340.27	857.57	n.d.	n.d.	n.d.	1139.95	876.81	n.d.	n.d.	n.d.	1265.64	n.d.
Endosulfan sulfate	11581.34	10697.20	3785.58	8759.48	25722.34	7835.84	n.d.	20761.48	20403.96	75063.88	n.d.	4339.45	4856.30	8171.99
Total concentration	17813.71	21473.81	22177.87	75446.47	46622.41	12941.20	86559.14	106251.93	35368.61	169850.81	99524.63	30486.86	20385.52	67112.24

Table 3: Concentrations of the organochlorine pesticides according to the sampling points (µg/kg, OC normalized).

Compounds	Concentrations (µg/g OC normalized)		
	Mean	**SD**	**Range**
α-HCH	0.18	1.24	0.02- 0.48
β-HCH	22.94	25.33	1.26- 67.92
γ-HCH	1.91	3.01	0.10- 7.76
δ-HCH	4.9	3.87	0.61- 11.10
∑HCHs	**29.93**	**26.92**	**1.39-81.31**
p,p'-DDD	0.16	0.14	0.070-9.18
p,p'-DDE	0.42	0.53	0.110-22.8
p,p'-DDT	4.58	5.26	20.0-346
Methoxychlor	0.59	1.01	0.160-78.2
∑DDTs	**5.75**	**5.52**	**0.64-19.02**
Aldrin	5.05	6.53	0.04- 5.98
Dieldrin	5.03	8.23	0.02- 25.06
Endrin	3.75	6.74	0.08-15.06
Endrin Aldehyde	2.92	1.70	1.46-4.79
Heptachlor	5.05	8.66	0.06-30.01
∑Cyclodienes	**21.8**	**17.91**	**0.02-34.67**
Endosulfan	0.97	1.44	0.06-4.51
Endosulfan II	1.1	0.22	11.1-50.8
Endosulfan sulfate	16.83	19.67	74.6-422
∑Endosulfans	**18.9**	**19.88**	**0.79-76.89**

Table 4: Statistics for Organochlorine Pesticides (Values in mg/kg, OC normalized).

Among HCHs, β- HCH isomer was much higher than α-, γ- and δ-HCH. Endosulfan sulfate has the highest concentration among endosulfan subgroup.

The p,p'- DDT isomer covers the 87.72% of the total DDTs. Dieldrin is responsible for the 33.09% share of cyclodiene pollution. Endosulfan sulfate has a share of 93.41% in its subgroup and in HCH subgroup β- HCH has the greatest share with 86.60%. This finding indicates that HCH derivatives of the area are residues of former usages since β-HCH was the most stable isomer in HCHs due to low vapor pressure [11].

Sampling points 4, 7, 8, 10, 11, 14 have greater average total concentration than the total average concentration around the lake. The most polluted site is the sampling point 10 with 169.87 mg/kg concentration (Table 5).

The data obtained in this study are compared to the other studies performed in some regions in Turkey and China. The data are seen in Table 6. Concentrations are the dry weight measurements and given in µg/g.

The comparison is made between the studies that include lakes since the fluvial effects become important when the rivers or seas are in discussion. Hirfanlı Dam Lake and Tuz Lake have higher pesticide concentrations than İkizcetepeler Dam Lake however Meriç Delta, Black Sea and Taihu Lake were found to be less contaminated than the sampling region in this study. Tuz Lake is the most polluted lake in terms of organochlorine pesticides, Hirfanlı Dam Lake follows it and İkizcetepeler Dam Lake is the third most contaminated region among these six selected sampling regions.

Sampling point	Total 17 OCP (ppm)
1	17.81
2	21.47
3	22.18
4	75.45
5	36.99
6	12.94
7	86.56
8	106.25
9	35.37
10	169.87
11	99.52
12	30.49
13	20.39
14	67.11
Average	58.00

Table 5: Concentrations of seventeen organochlorine pesticides from fourteen sampling points (concentrations in mg/kg).

	∑ HCHs (µg/g)	∑ DDTs (µg/g)	∑ Cyclodienes (µg/g)	∑ Endosulfans (µg/g)	Number of sampling points	Sampling Time	References
Hirfanlı Dam Lake	2.33	1.23	3.65	-	10	1999-2000	[16]
Tuz Lake	4.26	3.70	3.02	-	10	1999-2000	[16]
Meriç Delta	0.0023	0.0017	0.0018	0.0017	8	2002-2003	[19]
Black Sea, wet wt.	0.012	0.011	0.069	-	14	1998-2000	[17]
Taihu Lake, China	0.018	0.00079	0.0074	-	20	2006	[10]
İkizcetepeler Dam Lake	0.48	0.11	0.22	0.20	14	2009	This study

Table 6: Different studies and the sum of OCP analytes concentrations.

Conclusion

Fourteen surface sediment samples were analysed from Balıkesir (İkizcetepeler) Dam Lake for seventeen Organochlorine Pesticides (OCPs) analytes. The dry weight concentrations and OC-normalized concentrations were calculated. Concentrations of organochlorine pesticides in sediments were found higher. This made us to conclude that further studies can direct how harmful is using of the water from Dam Lake. It indicates that there is an environmental pollution related to pesticides contamination. Besides this, there may be PAH and other organics contaminations are also possible.

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