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Residues of Organochlorine Pesticides in Irrigated Sierozem-Meadow Soils around Buried Chemicals Stock

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Abstract

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The concentration and distribution of organochlorine pesticides (OCPs) were studied in sierozem-meadow soils around a buried obsolete chemicals stock in Mirzaabad district of Syrdarya region of Uzbekistan. 23 soil samples were collected from the topsoil (0–30 cm of soil depth) and 15 samples were collected from three soil profiles (down to 125 cm of soil depth) located in nearby vicinity of the stock. They were extracted using an automated Soxhlet extractor and analyses were done by gas chromatography-mass spectrometry. The detected OCPs were DDTs, HCHs, aldrin, dieldrin, endrin, chlorpyrifos, and endosulfans. The concentration of HCH was detected only as α-HCH in the range of 0–3.159 μg/kg. Only two isomers of DDT: *p,p*'-DDE and *p,p*'-DDD were found to be dominant, with the range of their values of 0.068-4.941 and 0-13.63 μg/kg, respectively. The concentrations of aldrin, dieldrin, endrin and chlorpyrifos in the samples collected around the source were in the range of 0–0.504, 0–1.117, 2.414–20.87 μg/kg and 0–3.819 μg/kg. β-endosulfan varied from a non-detectable amount to 74.56 μg/kg. According to results, the sum of DDTs was observed to be higher in the upper horizons, especially at depths of 0–10 and 10–50 cm, with the values 543.15 and 212.22 μg/kg, respectively, which can be a cause of concern. Due to their ability to migrate in the environment, accumulate in the food chain and be harmful to human health, monitoring the level of residues of organochlorine pesticides in soil is of vital importance.

Keywords: DDT; distribution; pollution; soil; source

The accumulation and distribution of organochlorine pesticides (OCPs) in the environment, especially in soils, can cause significant stress on ecosystems. OCPs are considered insoluble in water and they are strongly retained by soil. Persistent pesticides can be decomposed by environmental factors into other more complex components contaminating the soil with even more toxic compounds (Bhattacharya *et al.* 2003). OCPs show a high bioaccumulation potential, ubiquity, persistence in the environment and high toxicity to non-target organisms. The negative effect of DDT and other organochlorines on human health was summarized e.g. by Longnecker *et al.* (1997).

Many studies confirm the frequent occurrence of OCPs in soil, yet few studies assess the presence of OCPs in deeper horizons of the soil profile. Some authors (Zhang *et al.* 2009; Weaver *et al.* 2012) reported that much higher concentrations of OCPs, such as DDT, HCH, and endosulfan were determined in cotton and rice fields. Another author (Haria *et al.* 1994) showed that the transport of OCPs, insoluble in water, might occur in irrigated and frequently precipitated areas due to the physical movement of soil particles that adsorb pesticides in organic fractions.

The migration and distribution of OCPs can be explained by different processes and factors such as site characteristics, chemical properties of soil, application history and environmental factors (Ruzickova *et al.* 2008). Transport of OCPs may occur by runoff and by eroded sediments and soil particles, depending on the landscape of the cropping area (Shivaramaiah *et al.* 1998, 2002). Weaver *et al.* (2012) and Silburn *et al.* (2002) confirmed that the detection of higher values of OCPs in deeper soil horizons might be characterized by movement of the residues adsorbed in clay and soil organic matter via different cracks and pores.

High concentrations of persistent OCPs in topsoil, which are detectable at the present time, may be a result of their much increased use in the past, in spite of their application having been banned in most countries for many decades. In Uzbekistan, particularly high values of persistent pesticides are detectable in the soils around the buried agrochemicals stocks as well as around former agricultural aerodromes (Report 2007; National Report 2008). According to the data from State Committees of Uzbekistan, the total concentrations of OCPs in soils tend to be declining.

A number of studies have been conducted in order to measure the residues of pesticides in arable soils of Uzbekistan (Karimov *et al.* 2016). Although the state committees such as Centre of Hydrometeorology and State Committee for Nature Protection provide monitoring of the agrochemicals in environmental components annually, there is a need for studies which will concentrate a wide range of research on long-term impact and fate of OCPs in an ecosystem of Uzbekistan.

Thus, the present study deals with the details of analyses of residual concentrations of OCPs in arable sierozem-meadow soils of Uzbekistan.

MATERIAL AND METHODS

Research area is situated in Mirzaabad district of Syrdarya region of Uzbekistan. The area is covered by irrigated arable land and partly abandoned land. Prevailing crops grown in the area are wheat, cotton, rice and maize.

The climate in Mirzaabad district is continental with hot, dry summers and cold winters. The area is situated in a semiarid zone of Central Asia. According to meteorological data from two meteorological stations Syrdarya and Yangiyer, average annual air temperature ranges from 12.9 to 14.9°C while maximum temperature in summer varies between 25.4 and 29.5°C and minimum temperature ranges between1.8 and 0.1°C in winter. Soil temperature is in the range of 16.1–17.4°C, annually. Average relative air humidity ranges from 51.7 to 65.7%, while rainfall amount varies within 324–340 mm annually (Supplementary Table S1 in Electronic Supplementary Material (ESM)). The main wind mass comes from the east and northeast with 3–4 m/s. Within May and June, wind is scarce and weak.

Soil sampling. Soils of the research area were classified as sierozem-meadow (according to WRB it corresponds to Calcic Xerosol). Soil samples were collected in October 2013 at three distances and directions around the contamination source. From three soil profiles located in the east, south and west of the stock, 15 samples were collected (down to 125 cm of depth). Additionally, between the distances where soil profiles were taken, 23 samples were collected from topsoil (within 0–30 cm of depth), separately. The first soil profile (SP1) is the closest (200 m) to the source, and the other two (SP2 and SP3) are 400 m east and 700 m west of the source, respectively. No samples were taken from the north of the stock because of a water basin being located there. The samples of soil horizons were collected with a shovel into polyethylene bags and then transported to the laboratory where they were kept frozen until laboratory analyses. Soil samples were analysed by GC-MS in order to determine the concentration of OCPs in the samples of sierozem-meadow soils.

Sample extraction and measuring process. Soil extractions were done using an automated Soxhlet extractor (SER 148, Vepl Scientifica, Italy) according to Method 3541 (1994). Approximately 10 g of soil sample was added to the extraction thimbles and then 100 µl of internal standard containing deuterated PAH (2000 ug/ml) was dropped to all thimbles. The samples were extracted for two hours; the 1st hour immersed in the solvent, the 2nd hour reflux washed with the solvent. 50 ml of the hexane/acetone (1:1) solvent was used. The extractant was concentrated into 5 ml by solvent evaporation on the same machine and then further concentrated to 1 ml under a gentle stream of nitrogen using Turbo Vap (Caliper, Hopkinton, USA). Extracts were stored in the freezer until GC/MS analysis.

The OCPs such as DDTs, HCHs, aldrin, dieldrin, endrin, chlorpyrifos, the endosulfans and their metabolites were detected in soil samples collected around the source and in soil profiles collected at various distances from the stock. The concentrations of OCPs were determined using Varian 450 GC coupled to a Varian 240 MS ion trap detector (Varian, Santa Clara, USA) with VF-5MS column (30 m × 0.25 mm, film thickness: 0.25 μm) and helium (as carrier gas). Initial oven temperature was set at 60°C (held for 2 min), increased to 150°C at a rate of 30°C/min and then raised to 310°C at 8°C/min, after that held for 13.5 min. Injection temperature was 290°C and detector temperature was 200°C. Injection volume was 1.0 μl and samples were injected in a splitless mode. Normal scan mode was used and scan was averaged to 2 microscans. Data rate was 1.18 Hz.

During analysis, all quality and safety requirements were observed according to the methodology and general laboratory safety rules. Standards were kept in the freezer. Quantification was performed on the principle of internal standardisation. The limits of quantification (LOQ) were calculated to be 0.01 and 0.05 mg/kg (Supplementary Table S6 in ESM). Non-detectable levels of OCPs were marked as not detected (ND).

Physicochemical properties of soil. The physicochemical characteristics of soil (humus content, soil pH, cation exchange capacity, humus quality, carbonate content and texture) are given in Table 1 and Supplementary Table S2 in ESM. Soil pH_{H_2O} and pH_{KCl} were measured in accordance with ISO 10390 (1994). Organic matter (OM) content was calculated as total organic carbon (TC) multiplied by 1.724 (Skjemstad & Baldock 2008).

Humus quality was measured as a ratio of absorbances at 400 and 600 nm. Soil cation exchange capacity (CEC) was measured according to Bower & Hetcher (1966). The soil carbonate contents were detected by a volumetric method (Klute 1996). Soil texture was analysed hydrometrically (Gee & Or 2002). Results suggest that soils have alkaline and weak alkaline pH, poor organic matter and low unsaturated CEC, and high carbonate content. Soil texture is silty loam.

RESULTS AND DISCUSSION

Residual concentrations of OCPs determined in soil samples are given in Tables 2–4 and Supplementary Tables S3–S5 in ESM in appendices, and in Figure 1. The concentrations of HCH residues were quite low in all of the measured topsoil samples and did not exceed the maximum allowable limits set by Ministry of Health of the Republic of Uzbekistan to be 0.1 mg/kg in soil. However, α-HCH was detected at the concentration of 3.159 μg/kg (Table 2). No residues were found in the remaining samples suggesting that those were completely decomposed by microorganisms or probably affected by such environmental factors as leaching and volatilization.

Table 1. Physicochemical properties of the samples of soil profiles

Sample	Depth (cm)	pH_{KCl}	pH_{H_2O}	Humus (%)	A_{400}/A_{600}	CEC (meq+/100 g)	Carbonate (%)
SP1	0–10	7.77	8.07	0.56	2.12	12.0	10.9
	10–50	7.00	8.01	0.12	2.44	13.7	10.0
	50–90	8.05	8.38	0.29	3.32	8.7	12.0
	90–100	8.20	8.83	0.30	2.52	7.5	11.5
	100–115	8.17	8.99	0.18	2.28	13.2	15.0
	115–125	7.90	8.88	0.32	1.74	8.0	13.5
SP2	0–10	8.13	8.99	0.52	5.89	13.7	14.5
	10–30	8.12	9.05	0.32	2.65	13.5	14.4
	30–60	7.94	8.91	0.33	3.94	14.2	13.7
	60–90	8.11	8.84	0.19	1.92	13.2	15.0
	90–120	7.83	8.52	0.24	1.79	14.2	17.0
SP3	0–30	7.93	8.50	0.55	4.50	15.0	14.0
	30–60	7.93	8.86	0.24	2.16	12.2	15.0
	60–90	8.27	9.04	0.12	3.06	14.5	15.0
	90–115	8.19	9.03	0.14	1.34	20.0	14.0

CEC – cation exchange capacity; A_{400}/A_{600} – humus substances colour quotient

Table 2. Residues of HCHs (in μg/kg) in the topsoil samples at the depth of 0–30 cm

Sample	α-HCH	β-HCH	δ-HCH	γ-HCH	Sum HCH
S1	ND	ND	ND	ND	0
S2	ND	ND	ND	ND	0
S3	3.159	ND	ND	ND	3.159
S4	ND	ND	ND	ND	0
S5	ND	ND	ND	ND	0
S6	ND	ND	ND	ND	0
S7	ND	ND	ND	ND	0
S8	ND	ND	ND	ND	0
Mean	0.451	ND	ND	ND	0.451
SD	1.194	ND	ND	ND	1.194
Minimum	0	ND	ND	ND	0
Maximum	3.159	ND	ND	ND	3.159
Range	0–3.159	ND	ND	ND	0–3.159

ND – not detected; SD – standard deviation

According to Willett *et al.* (1998) and Li (1999), technical HCH contains 60–70% of α-HCH, 5–12% of β-HCH, 10–12% of γ-HCH, 6–10% of δ-HCH and 3–4% of ε-HCH. Results of this study showed that α-HCH isomer was in a detectable range and this is probably a product of Lindane isomerization.

The presence of HCHs confirmed that only β-HCH isomer with 59.23 μg/kg was detectable at 0–30 cm depth of SP3 and no isomers were found in the rest of the samples, not even in the samples collected closest to the stock. On the one hand, degradation of HCHs in soil seems to be almost complete, which confirms no recent use. On the other hand, the appearance of β-HCH indicates its persistence against decay or adsorption onto soil particles (Congcong *et al.* 2013).

Levels of *p,p*'-DDE and *p,p*'-DDD in the samples collected around the source ranged from 0.07 to 4.94 (mean 1.06) and from 0 to 13.63 (mean 2.29) μg/kg, respectively.

Weaver *et al.* (2012) and Shivaramaiah *et al.* (2002) indicated that DDE concentrations up to 400 μg/kg can occur in the topsoil of cotton fields where agrochemicals had been used in the past. In our case, the detected concentrations of DDE are under no means an evidence of historical application of agrochemicals; they are rather caused by the

Table 3. Residues of DDTs (in μg/kg) in the topsoil samples at the depth of 0–30 cm

Sample	*o,p*'-DDD	*o,p*'-DDE	*o,p*'-DDT	*p,p*'-DDE	*p,p*'-DDD	Sum DDT
S1	ND	ND	ND	4.941	13.63	18.571
S2	ND	ND	ND	0.467	1.555	2.022
S3	ND	ND	ND	1.486	0.941	2.427
S4	ND	ND	ND	0.595	0.674	1.269
S5	ND	ND	ND	0.068	1.504	1.572
S6	ND	ND	ND	0.174	ND	0.174
S7	ND	ND	ND	0.505	ND	0.505
S8	ND	ND	ND	0.235	ND	0.235
Mean	ND	ND	ND	0.504	0.668	1.172
SD	ND	ND	ND	0.474	0.695	0.893
Minimum	ND	ND	ND	0.068	0	0.174
Maximum	ND	ND	ND	1.486	1.555	2.427
Range	ND	ND	ND	0.068-1.486	0–1.555	

ND – not detected; SD – standard deviation

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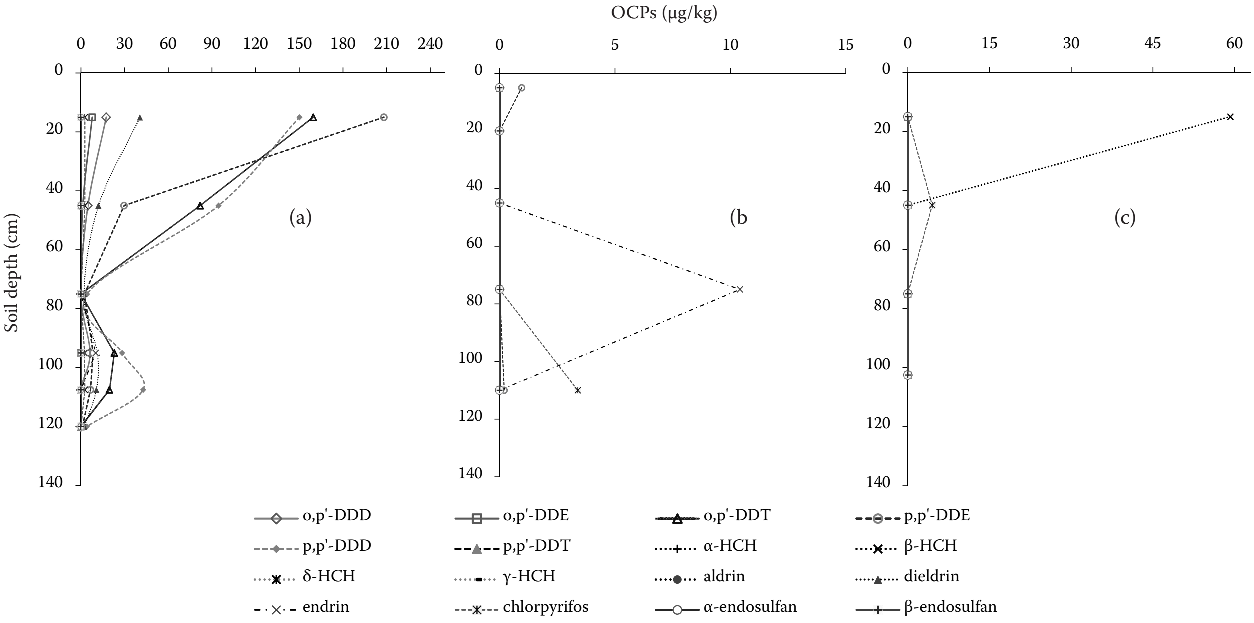

Figure 1. Distribution of organochlorine pesticides (OCPs) in soil profiles SP1 (a), SP2 (b) and SP3 (c)

storage of buried OCPs aged more than 30 years. No residues of DDT were detectable assuming there was no movement of residues across the source or it reflects their complete breaking down in time.

Analyses revealed that the highest amount of DDTs was detectable particularly within 100 cm of depth of SP1 (Figure 1 and Supplementary Table S3 in ESM). Levels of *p,p*'-DDE and *p,p*'-DDD with 208.19 and 150.15 μg/kg were mainly found within 0–10 cm depth of SP1, while the level of *o,p*'-DDT was 159.64 μg/kg in the same sample. *p,p*'-DDE, *p,p*'-DDD and *o,p*'-DDT were considerably higher than the maximum allowable limits which are 10 μg/kg. Their concentrations were rather high within 10–50 cm depth where the DDT sum accounted for 212.22 μg/kg. The measured levels of the residues in soil horizons are given in Figure 1. In SP1 DDT rates were greater within 0–50 cm depth than those in deeper horizons. The sum of OCP residues within 100–115 cm depth of SP1 was also measured to be 66–69 μg/kg (Figure 1, Supplementary Table S3 in ESM). In soil profile SP2 the concentration of *p,p*'-DDE was higher down to a depth of 10 cm in

Table 4. Residues of organochlorine pesticides (OCPs, μg/kg) in the topsoil samples at the depth of 0–30 cm

Sample	aldrin	dieldrin	endrin	chlorpyrifos	α-endosulfan	β-endosulfan
S1	0.50	1.12	11.72	ND	ND	ND
S2	ND	ND	5.28	3.82	ND	ND
S3	ND	0.39	10.87	3.09	ND	ND
S4	ND	ND	20.87	ND	ND	ND
S5	ND	0.55	11.36	ND	ND	74.56
S6	ND	ND	13.40	ND	ND	6.92
S7	ND	ND	2.41	ND	ND	2.32
S8	ND	ND	9.79	ND	ND	ND
Mean	0.06	0.26	10.71	0.86	ND	10.47
SD	0.18	0.41	5.49	1.61	ND	26.01
Minimum	ND	ND	2.41	ND	ND	ND
Maximum	0.50	1.12	20.87	3.82	ND	74.56
Range	0–0.50	0–1.12	2.41–20.87	0–3.82	ND	0–74.56

ND – not detected; SD – standard deviation comparison with the remaining residues. However, the presence of residual amounts in subsoil can indicate their movement throughout soil horizons.

Similar studies (Heberer & Deunnbier 1999) reported that the occurrence of higher levels of DDE and DDD in soil layers indicated an intensity of both aerobic and anaerobic degradation of parent DDT itself.

Our results confirmed those phenomena in which the occurrence of high levels of parent DDT decomposition in subsoil horizons has been measured as *p,p'*-DDD within 90–115 cm depth of SP1 ranging from 28.30 to 42.70 μg/kg. However, the level of *o,p'*-DDT was found to be high within 0–10 and 10–50 cm depths at 159.60 and 82.00 μg/kg, respectively. No *p,p'*-DDT was detected in the remaining soil profiles (Figure 1, Supplementary Table S3 in ESM). Interestingly, there were not any significant amounts of DDT in SP2 and SP3. This is because of their remoteness from the pollution source, whereas SP1 was directly in a neighbouring area with the stock.

Furthermore, we have measured the OCPs such as aldrin, dieldrin, endrin, chlorpyrifos, alpha and beta endosulfans from all collected samples, which have not been previously determined in that research area. The data analyses revealed the occurrence of aldrin, dieldrin, endrin, chlorpyrifos, and alpha and beta endosulfans in soil samples taken from buried land in which the amounts ranged within 0–0.5, 0–1.12, 2.4–20.9, 0–3.82 and 0–74.56 μg/kg, respectively (Table 4 and Supplementary Table S5 in ESM).

The concentration of aldrin was very small and alpha endosulfan was not in detectable values in those samples, indicating their almost complete breakdown. However, endrin and β-endosulfan were dominantly found in remote areas from the stock, which can be explained by their resistance to microbial decay. Downward movement of dieldrin and chlorpyrifos through soil profiles predominantly occurred in SP1 with measured values of 2.3–40.5 and 2.5–3.3 μg/kg, respectively. The endrin level was detected up to 10.4 μg/kg in both SP1 and SP2. No endosulfan isomers were measurable in those samples. However, the levels of dieldrin in SP1 showed an increasing tendency with greater depths of the soil horizon indicating that it has been moved downward either with soil particles or through cracks present as persistent residues.

CONCLUSION

Persistence of man-made chemical compounds is one of the key characteristics of pesticides which may ingenuously generate a resistance for such environmental reducers as microorganisms. Confirming this point of view, the analysis of measured data showed that despite more than three decades of the ban of persistent OCPs, significant amounts of pesticides were found even at risky levels which were predominated by DDT metabolites such as DDE and DDD in local topsoil and soil profiles. Although relatively high concentrations of OPCs, especially DDTs, were measured in the samples of soil profiles in the proximate area of the stock, it could not probably support the point that the mobility of residues down to soil layers has naturally occurred. The fact is that the chemical compounds in the stock area had been covered with soil until it has been disturbed by local residences. It is also of particular interest that the occurrence of OCPs in those topsoil samples collected around the stock indicates their slightly spatial distribution perhaps caused by wind rather than by anthropogenic influences. Overall, significant amounts of OCPs were found to be alarming, especially within 0–50 cm depth while insignificant levels were measured in the intermediate (50–90 cm) and deeper horizons (115–125 cm). The main distribution order of OCPs in soil was DDE > DDT > DDD > dieldrin. To sum up, the detected concentrations of residual OCPs may be a real threat to the environment and public health.

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