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Pollution status, distribution, source analysis, and risk assessment of OCPs in soil from the Hexi Corridor in Northwest China

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Abstract

Since OCPs with different historical usage and atmospheric input may show varied environmental behaviors and risks to the local residents and relevant research on underdeveloped areas in the middle latitudes of China is lacking, it is essential to classify the contamination status and sources of OCPs from these middle latitudes areas and to evaluate the related health risks to humans. Fifty soil samples were collected and analyzed within five cities in the Hexi Corridor in Northwest China. The ranges of $\sum_{24}$OCPs, ∑DDTs, and ∑HCHs concentrations in the soil samples were 23.1-393 ng/g, 4.96-167 ng/g, and 3.40-97.5 ng/g, respectively. The residual OCPs in soil were dominated by DDTs and HCHs, accounting for 38.7% and 16.1% of $\sum_{24}$OCPs. Source analysis shows that the HCHs come from historical application and possible recent pesticide use, and DDTs are mainly from early application residues that formed in aerobic environments created by agriculture ploughing, which aerates the soil. The risk assessment showed that the soil in the Hexi Corridor may have a potential risk of residual OCPs, and the carcinogenic risk (CR) was 1.90×10^{-7}-6.12×10^{-7} for adults and 5.6×10^{-7}-1.8×10^{-6} for children, but the hazardous index (HI) was 0.0093 to 0.2817 for adults and 0.032 to 0.932 for children. Only a few samples showed values higher than the acceptable range for children. Therefore, in this study area, there is a low health risk to local

residents. Nevertheless, our results provide a strong rationale for ongoing risk assessment and management and, hopefully, eradication of OCPs in the environment.

Keywords: OCPs, Hexi Corridor, pollution characteristics, residual levels, carcinogenic risk

INTRODUCTION

Organochlorine pesticides (OCPs) are mostly derivatives of chlorine and contain one or more benzene rings. These pesticides are semi-volatile and have high toxicity, environmental persistence, bioaccumulation, and long-range migration[1]. They are also considered to be typical persistent organic pollutants (POPs)[2-4]. OCPs accumulate through the enrichment and amplification effects that happen in food chains, and eventually enter the human body, endangering human health. As a big cultivated country, China has been a major producer and consumer of OCPs, but it has gradually banned OCP usage since 1983[5-7]. However, studies have found that the residual concentrations of OCPs are still at a high level in areas where OCPs were once widely used. Soil is an important reservoir of OCPs. The half-life of OCPs in soil lasts for several years to several decades, and they can be adsorbed into soil organic matter and enter food chains, resulting in biological accumulation and enrichment[8]. Simultaneously, the contaminated soil is also part of the global cycle of OCPs. From soil, they can be transferred to the atmosphere and bodies of water through volatile diffusion, forming secondary pollution[9,10]. Due to their stable chemical structure, higher toxicity, and resistance to degradation, the environmental issues of OCPs in different regions and countries have become a global concern[7,8,11-14].

At present, the studies on OCP contamination have been mainly concentrated in the economically developed provinces and regions of China (e.g., the southeastern coast of China)[15,16], and the plateau areas such as the Tibetan Plateau of China[17,18]. However, there are few studies that have examined the northwest of China[19,20]. The Hexi Corridor, in western Gansu Province, is an important grain-producing area located in a key part of the Silk Road Economic Belt[21]; it provides more than 2/3 of the commercial grain and cash crops for Gansu Province. The area is remote from the sea and deep in the hinterland of Eurasia. OCPs were widely applied in this area before they were banned[22]. Geographically, the Hexi Corridor is a typical semiarid area with a unique valley and basin terrain and complex meteorological conditions. Data show that before the prohibition of OCPs, the proportion of HCHs and DDTs in the region accounted for more than 50% of the total pesticide use[23]. In summary, the Hexi Corridor is unique from other areas that have been studied regarding OCPs because of its unique climate, topography, and historical application of OCPs and because the source and input of OCPs in this middle-high latitude area are expected to be different from the developed regions and plateau areas. Therefore, the data on pollution levels and geographical spread of OCPs obtained through this study can offer valuable insights to policymakers, allowing them to implement suitable measures for mitigating potential ecological and health hazards.

MATERIAL AND METHODS

Study area and sample collection

The study region (92°13′-108°46′ E and 32°31′-42°57′ N) was represented by five main cities. Fifty soil samples were collected in November 2018 from Jiuquan (A1-A10), Jiayuguan (B1-B10), Zhangye (C1-C10), Jinchan (D1-D10), and Wuwei (E1-E10) [Figure 1]. Collect soil samples using the uniform distribution method, refer to GB/T 36197-2018 for specific operations. Five subsamples were gathered from each sampling site, taken from the same 100 m^2 area with a depth range of 0-20 cm, which were then combined to create a composite sample. After eliminating stones and residual roots, the samples were air-dried at room temperature and filtered through a 1-mm mesh screen. These samples were subsequently placed in

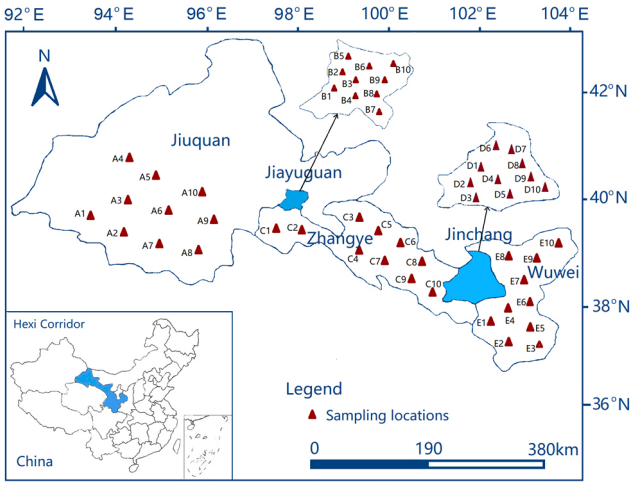

Figure 1. Sampling locations in the study area.

glass bottles and kept at 20 °C below zero until they were analyzed. Soil pH and organic carbon content were evaluated using previously described techniques[24].

Chemicals and reagents

OCPs mixed standards were purchased from AccuStandard and included 24 individual compounds: *α*-HCH, *β*-HCH, δ-HCH, *γ*-HCH, *p,p′*-DDD, *p,p′*-DDT, *p,p′*-DDE, *o,p′*-DDT, dicofol (DIC), HCB, heptachlor (HE), heptachlor epoxide (HPE), *cis*-chlordane (*cis*-CD), *trans*-chlordane (*trans*-CD), *α*-endosulfan (*α*-ED), *β*-endosulfan (*β*-ED), endosulfan sulfate (EDS), aldrin (AD), dieldrin endrin (DE), endrin aldehyde (EDA), endrin ketone (EDK), mirex (ME), and methoxychlor (MEC). The 100-200 mesh silica gel was purchased from Qingdao Haiyang Chemical Co. (Shandong, China). It was activated by heating it at 130 °C for 16 h, then deactivated by adding purified water (3.3%), and stored in a desiccator. To use, anhydrous Na_2SO_4 was baked at 600 °C for 6 hours. All solvents used were of HPLC grade (Fisher Chemical Company, USA) and all glassware was thoroughly cleaned with detergent, $K_2Cr_2O_7$-H_2SO_4 solution, running water, deionized water, and acetone. It was then baked at 400 °C for 3.5 h before reusing.

Sample pretreatment and instrumental analysis

Our team followed the procedure detailed in our previous report to evaluate the target OCPs[25]. First, 10 g soil samples were weighed for each group and mixed with 10 g of anhydrous Na_2SO_4. Next, we spiked this mixture with a surrogate (4-Chloro-m-xylene with a concentration of 2.5 × 10^{-4}μg/g) and used a low-temperature ultrasonic extractor to extract it with a 200-mL combination of hexane/acetone (1:1 v/v). The resulting extracts were concentrated, and their solvent was exchanged to hexane. We utilized a silica gel column of dimensions 25 cm × 1 cm I.D. containing 6 g of deactivated silica gel topped with 2 cm of anhydrous sodium sulfate. To prepare the column, we first eluted it with 40 mL of hexane before injecting the extract. Then, we proceeded by eluting the column with 80 mL of n-hexane, followed by 35 mL of *n*-hexane/DCM (2:3 v/v). The process used to determine OCPs using gas chromatography with a micro-electron capture detector (GC-μECD). The detector was equipped with a fused silica capillary column and helium was used as a carrier gas. The temperature program involved maintaining an initial temperature of 60 °C for 2 min, followed by an increase to 190 °C at a rate of 5 °C/min, and then an increase to 280 °C at a rate of 10 °C/min. Quantification of OCPs was performed using a five-point calibration method, and the results were confirmed with an Agilent 6890GC-5975MSD system, which utilized electron impact ionization mode and selected ion monitoring (SIM) mode.

Quality assurance

Table 1 presents the limits of detection (LOD), analysts' recoveries, and relative standard deviations (RSD) obtained by testing natural soil spiked with individual OCP standards (n = 5) to assess the recovery efficiency of the procedure. To ensure proper procedural performance and monitor matrix effects, different blank and triplicate samples were evaluated for each of the five samples, with additional surrogates added to each sample. Soil samples spiked with surrogates showed recoveries ranging from 63.4% to 92.0%, while the OCP variabilities in triplicate samples were no more than 10%. The results were presented on a dry weight basis, and were not adjusted for recoveries.

RESULTS AND DISCUSSION

Pollution characteristics and residual levels of OCPs

In this paper, we collected 50 soil samples from five cities located in the Hexi Corridor and determined the concentrations for 24 individual OCPs. The statistical results of OCPs in the soils are shown in Table 1, and the residual contents of OCPs in the soil of five cities are shown in [Supplementary Table 1]. From Table 1, except for a few sampling locations for individual OCPs, such as EDS, EDA, EDK, ME, and MEC, other individual OCPs were detected in the soil at all sampling locations, which indicated that the OCP contamination in the Hexi Corridor was diverse and ubiquitous. Based on historical reports, the usage of DDTs and HCHs was 15,589 tons and 64,991 tons, respectively, from the 1950s to the 1980s in Gansu Province[26]. As may be expected, the entire study location, as a main grain and vegetable production location, has relatively widespread OCP residues, mostly likely due to historical usage as well as recent input in the study areas. The contents of $\sum_{24}$OCPs ranged from 23.1-393 ng/g (average: 94.7 ng/g), the ∑DDTs contents ranged from 4.96-167 ng/g, and the ∑HCHs contents were in a range of 3.40-97.5 ng/g. The sum of AD, DE, EDA, and EDK (∑Drins) was in a range of 4.04-64.3 ng/g, accounting for 12.6% of $\sum_{24}$OCPs. The total concentration of *cis*-CD and *trans*-CD (ΣCHLs) ranged from 0.740-24.1 ng/g. The order of the dominant individual OCPs in the Hexi Corridor was as follows: DDTs (average: 35.8 ng/g) > HCHs (average: 16.0 ng/g) > ∑Drins (average: 11.6 ng/g) > Nonachlor (average: 6.27 ng/g) > Methoxychlor (average: 4.86 ng/g) > CHLs (average: 4.32 ng/g) > HCB (average: 4.28 ng/g) > Endrin aldehyde (average: 4.18 ng/g). The average concentration of heptachlor epoxide (3.30 ng/g) was higher than that of heptachlor (1.75 ng/g), indicating an existing aerobic degradation environment in the areas sampled. Among 24 individual OCPs, the contents of ΣDDTs and ΣHCHs were much higher than that of the other OCPs, accounting for 38.7% and 16.1% of OCPs, respectively. The OCPs in soils from the Hexi Corridor were mainly HCHs and DDTs, and the concentration of DDTs was higher than that of HCHs, which may be attributed to (1) larger amounts of DDTs used in agricultural activities compared to HCHs, and (2) the higher resistance to bio-degradation of DDTs than HCHs[27]. Among individual OCPs, EDS was relatively low (average: 0.34 ng/g), but it was detected in 98% of the soil samples.

There are many studies on OCP contamination in soil from China and worldwide [Table 2]. The ∑OCPs levels in urban soils vary considerably, ranging from 7.6 to 37,331 ng/g with a median value of 53.7 ng/g. The average content of OCPs is 2,861 ng/g, which is several orders of magnitude higher than the values (58.9 ng/g) reported for agricultural soils in China[12]. Compared with other regions and countries in the world, the content of ∑HCHs in this study was lower than that of Kalashah Kaku, Pakistan[28], Spain[29], and Tianjin[30] in China. ∑HCHs were comparable to the results from Urumqi[31], Taiyuan[32], and Fujian Province[8] in China. But ∑HCHs were higher than those reported for Hong Kong[33], Beijing[34], Guangzhou[22], and Shanghai[25]; seven cities in India[35]; Punjab Province of Pakistan[36]; Canada; and the UK[37]. The content of ∑DDTs in soil was lower than that in Romanian soil[38,39], Germany[40], Beijing[34] and Guangzhou[22] in China; but higher than that reported for the Pearl River Delta[41], Urumqi[31] and Hong Kong[33] in China as well as that reported for India[42], Nepal[43], Italian[13,14], and Poland[44]. In general, compared with other areas in China and worldwide, the contents of HCHs and DDTs in the soil in the Hexi

Table 1. Statistics results and the detected limit, recovery and RSD of the method of OCPs in soil (*n* = 50, ng/g)

Analysts	LOD	Recovery	RSD	Min	Max	Mean	SD	DF	RV
α- HCH	0.016	94.15	6.72	0.121	14.4	1.71	2.64	100	2.5
β- HCH	0.019	84.39	8.79	0.303	80.1	6.20	12.38	100	1.0
γ- HCH	0.027	89.58	5.36	0.791	35.0	4.33	5.25	100	0.5
δ-HCH	0.015	86.21	8.13	0.164	16.5	3.79	2.92	100	-
ΣHCHs	-	-	-	3.40	97.5	16.1	13.5	100	10
p,p′-DDD	0.007	91.43	6.66	1.09	37.6	5.98	5.43	100	-
p,p′-DDE	0.005	82.37	7.32	1.54	40.7	8.83	6.76	100	-
p,p′-DDT	0.010	89.89	7.98	1.35	93.6	18.3	15.7	100	-
o,p′-DDT	0.012	81.32	5.32	0.262	14.6	2.62	2.64	100	-
ΣDDTs	-	-	-	4.96	167	35.8	47.5	100	2.5
HCB	0.005	92.35	4.74	0.391	83.9	4.28	12.0	100	2.5
Nonachlor	0.035	71.47	9.80	1.03	35.0	6.27	8.24	100	-
Heptachlor (HE)	0.025	84.36	4.38	0.113	4.92	1.05	1.12	100	-
Heptachlor epoxide (HPE)	0.015	94.32	4.58	0.293	21.1	3.30	3.38	100	-
trans-Nonachlor	0.020	68.87	7.82	0.181	8.19	1.46	1.68	100	-
cis-Chlordane (cis-CD)	0.020	78.34	6.68	nd	6.41	1.43	1.33	98.0	2.5
trans-Chlordane (trans-CD)	0.015	78.80	5.54	nd	8.35	1.97	1.50	98.0	2.5
ΣCHLs	-	-	-	0.270	8.16	2.58	1.01	100	
Endrin aldehyde (EDA)	0.030	81.34	6.25	0.924	35.2	4.18	5.77	100	-
Endrin ketone (EDK)	0.032	78.18	8.17	0.351	11.7	2.33	2.07	100	-
α-endosulfan (α-ED)	0.016	69.27	6.15	0.362	9.83	2.01	2.29	100	-
β-endosulfan (β-ED)	0.018	89.54	6.42	nd	19.63	2.74	3.16	96.0	-
Endosulfan sulfate (EDS)	0.025	85.25	7.47	nd	1.60	0.34	0.31	98.0	-
Endrin (ED)	0.030	75.54	9.88	nd	9.48	2.25	1.80	92.0	1.0
Aldrin (AD)	0.046	78.84	5.35	0.293	14.9	2.35	2.40	100	-
Diedrin endrin (DE)	0.036	72.45	9.78	0.222	17.2	2.03	2.47	100	-
Methoxychlor (MEC)	0.025	78.84	6.88	nd	17.59	4.86	3.73	96.0	-
Σ24OCPs	-	-	-	23.1	393	94.7	45.3	100	

LOD: the limits of detection; RSD: relative standard deviations; nd: not detected; SD: Standard Deviation; RV: reference values of unpolluted soil according to soil protection guideline of the Netherlands.

Corridor fell in the upper middle level. The study area is located in the temperate continental climate, which is characterized by arid and semiarid areas with less wind and rain, and fewer microorganisms in soil, resulting in a slower degradation of HCHs and DDTs.

Spatial distribution of OCPs

Figure 2 and [Supplementary Table 1] show the distribution characteristics and spatial distribution of OCPs in the soils from the study area. OCPs are normally associated with agricultural application contamination[25], which may explain the differences in OCP levels obtained among the five cities [Supplementary Table 1]. From [Supplementary Table 1], Jiuquan is known to have a long and intensive agricultural and pesticide production history, and therefore the highest average level with a mean of 179 ng/g (ranging from 49.2-393 ng/g) is not surprising. The other three cities, despite some extreme values, had relatively low contents, with Jiayuguan showing a mean of 75.3 ng/g (ranging from 45.8-153 ng/g), very similar to Wuwei with a mean of 74.9 ng/g (ranging from 23.1-129 ng/g) and Jinchang with a mean of 84.1 ng/g (ranging from 56.9-125 ng/g). very similar to Wuwei with a mean of 74.9 ng/g (ranging from 23.1-129 ng/g) and Jinchang with a mean of 84.1 ng/g (ranging from 56.9-125 ng/g). Zhangye showed the lowest mean value (60.4 ng/g and ranging from 38.8-96.7 ng/g). A little surprising, as the major agricultural

Table 2. Distribution of DDTs and HCHs (ng/g) in soils of different regions

Location	Sample time	∑HCH (ng/g)	∑DDT (ng/g)	References
Kalashah Kaku, Pakistan	2008	25-122	-	[28]
Spain	2003-2004	200	-	[29]
Tianjin	2008	77.67	-	[30]
Urumqi	-	nd-30.86 ng/g	nd-40.03	[31]
Taiyuan	2006	5.24-97.81	-	[32]
Fujian Province	-	0.97-247	-	[8]
Hong Kong	-	2.5-11	nd-5.7	[33]
Beijing	2007	0.64-32.32ng/g	1.42-5,910.80	[34]
Guangzhou	-	nd-17.79 ng/g	3.58-831	[22]
Shanghai	2007	nd-10.38 ng/g	-	[25]
seven cities in India	-	0.01-60	-	[35]
Punjab Province of Pakistan	2014	1.7-20	-	[36]
UK	-	< 0.01-0.07	-	[37]
rural soil of Romania	2009	-	226.9 ± 157.2	[38-39]
urban soil of Romania	2009	-	113.1 ± 151.8	[38-39]
Germany	1999	-	23.7-173	[40]
the Pearl River Delta	2006	-	0.27-414	[41]
Hexi Corridor	2019	3.40-97.48	4.96-166.97	This study

nd: not detected.

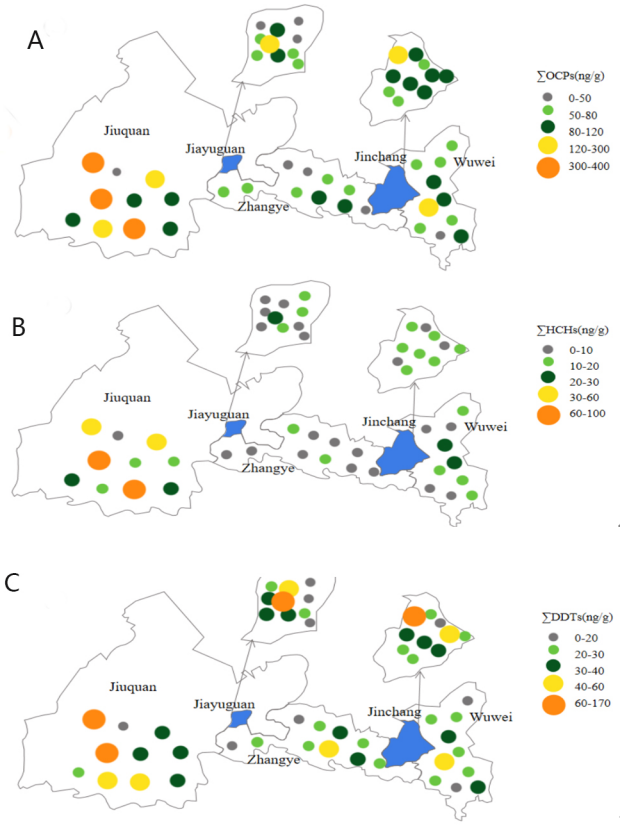

Figure 2. Spatial distribution of (A) for $\sum_{24}$OCPs; (B) for ∑HCHs; and (C) for ∑DDTs in soil from Hexi Corridor in Northwest China.

production area, this low level might be attributed to the rise of organic agriculture and facilities farming, resulting in a high degradation of OCPs. However, this hypothesis needs further investigation. For individual sampling locations, it can be seen from [Supplementary Figure 1] that the highest value (393 ng/g) of OCPs is located in A3 (Jiuquan), and the lowest value (23.1 ng/g) is located in E2 (Wuwei). The highest HCHs value (29.2 ng/g) was in A7 (Jiuquan), and the lowest HCHs value (14.7 ng/g) was in E1 (Wuwei). The highest value of DDTs (42.9 ng/g) was in A4 (Jiuquan), and the lowest value (6.93 ng/g) was in C3 (Zhangye).

The contents of *p,p′*- DDT and *p,p′*-DDD were the highest among detected individual OCPs, which were 1.35-93.6 ng/g and 1.54-40.7 ng/g with a mean of 18.3 ng/g and 8.33 ng/g, respectively. As can be seen from Figure 2, the highest contents of DDTs appear at sampling locations A3, A4, and A7 (Jiuquan City). The order of content of $\sum_{24}$OCPs in the soils of five cities from highest to lowest in Hexi was Jiuquan > Jinchang > Jiayuguan > Wuwei > Zhangye. The order of ∑HCHs residual content was Jiuquan > Wuwei > Jinchang > Jiayuguan > Zhangye. The order of ∑DDTs residual content was Jiuquan > Jinchang > Jiayuguan > Wuwei > Zhangye. Most of the sampling locations in Jiuquan were near cultivated land and farmland. Previous research data indicates that before the prohibition of OCPs, the proportion of HCHs and DDTs in the region accounted for more than 50% of the total pesticide use[23]. Therefore, there were high residual amounts of HCHs and DDTs. In addition, Jiuquan is a city where industry and agriculture jointly develop, while Zhangye is an agricultural city. Therefore, the sources of OCPs in Jiuquan soil are more extensive than those in Zhangye. Thus, the residual OCPs in the soil of Jiuquan are higher compared to other cities.

The composition of OCPs

Composition characteristics of HCHs

Figure 3A displays the HCH composition present in the soil of the Hexi Corridor. All four HCH isomers (*α*, *β*, *γ*, and δ) were detected with a 100% detection rate, and the total residual concentration of HCHs ranged from 3.40 ng/g to 97.5 ng/g, with an average of 16.0 ng/g. The residual concentration of individual isomers was as follows from highest to lowest: δ-HCH (6.20 ng/g) > *β*-HCH (4.33 ng/g) > *γ*-HCH (3.79 ng/g) > *α*-HCH (1.71 ng/g). The percentage of HCH isomers found in soil samples was as follows: δ-HCH (30.1%) > *β*-HCH (29.9%) > *γ*-HCH (27.0%) > *α*-HCH (9.03%). The percentage of δ-HCH found in Jiuquan soil (30.1%) was the highest among all sampling sites, indicating the possibility of historical residues in the study areas in the Hexi Corridor. Data show that before the prohibition of OCPs, the proportion of HCHs and DDTs in the region accounted for more than 50% of the total pesticide use[23]. Moreover, δ-HCH, as a difficult-to-biodegradable isomer of HCHs, has a high residual amount in soil.

Composition patterns of the DDTs

The compositions of four isomers of DDTs are presented in Figure 3B. The detection frequencies of individual isomers of DDT in the soil was 100%, and the total contents of DDTs in the soil were 4.96-167 ng/g with a mean of 35.8 ng/g. The amounts of *p,p′*- DDT, *p,p′* - DDD, and *p,p′*- DDE accounted for 48.67%, 26.56%, and 17.38%, respectively, while *o,p′*- DDT accounted for only 7.40% of the ∑DDTs [Figure 3B]. The lowest content of DDT was *o,p′*-DDT, with a mean of 2.62 ng/g. Judging from Figure 3B, the DDT contamination in the study area was mainly DDT and DDD, indicating that the soil DDTs in the Hexi Corridor were mainly derived from early pesticide residues. This may be due to the fact that the study area is arid and semiarid, and the annual average temperature is low, so DDT had the highest contribution to the residual amount of OCPs.

Composition patterns of the other OCPs

The high levels of HCB originate from industrial and agricultural applications, as well as from the

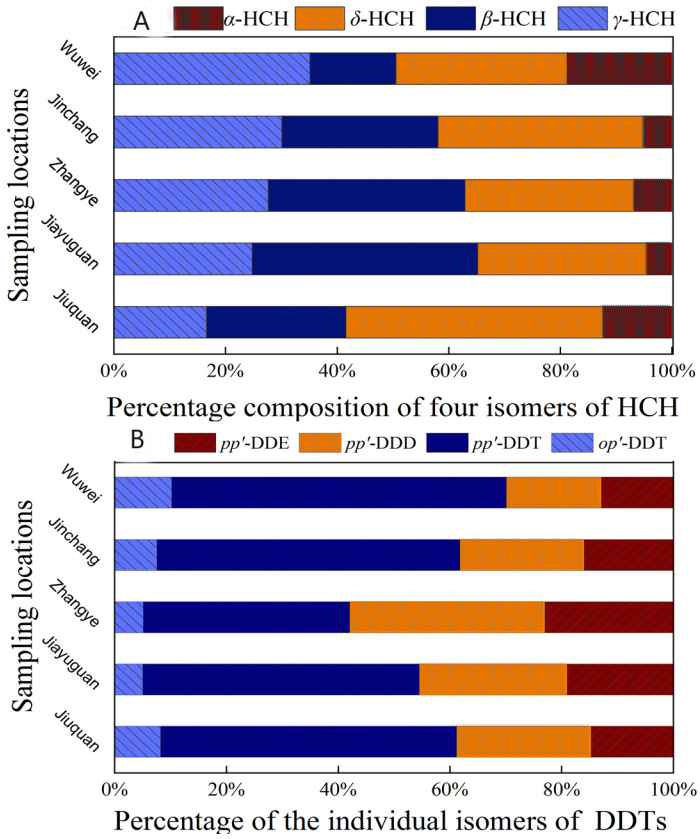

Figure 3. Percentage of the individual isomers of HCH (A) and DDTs (B) in soils. DDTs: Dichlorodiphenyltrichloroethanes; HCH: Hexachlorocyclohexane.

incomplete burning of waste, fossil fuels, and biomass[45]. Additionally, HCB is capable of getting released into the surroundings when different production procedures linked to the byproducts of chlorinated solvents, aromatics, and pesticides occur. The historical production of HCB was 3,522 tons/year until it was banned in 2004 in China[16]. HCB was found to be prevalent in all locations that were studied, with an average of 4.28 ng/g (ranging from 0.389-83.9 ng/g) [Table 1]. The higher levels of HCB were distributed in Jiuquan and Wuwei. The soil samples indicated the presence of the highest concentration of HCB at sites A8, A3, and A4 in Jiuquan and at E3, E4, E6, and E7 in Wuwei. ED comprised 3.1-9.7% of Σ_{24}OCPs in the collected samples [Figure 2]. The higher levels of *cis*- and *trans*-ED were found in Jiuquan in the range of 0.79-7.23 ng/g (mean 2.85 ng/g). The contents of *cis*- and *trans*-ED detected in Jiayuguan, Jinchang, and Wuwei were similar--0.36-2.27 ng/g, 0.41-2.57 ng/g, and 0.17-3.15 ng/g, respectively [Supplementary Table 1]. The highest concentrations of EDs were detected at A3, A1, and A7 in Jiuquan [Supplementary Figure 1]. Industrial ED is still applied in cotton and other crops in China. As such, it is found in more than 90% of agricultural soils in China and is the most chemically rich OCP. This is consistent with the fact that ED is currently being utilized in China and that extensive cotton cultivation exists around the study area in the upwind areas, causing it to accumulate in soils over time. The historical production of chlordane was 363 tons/year[16]. Chlordane was not widely applied in agricultural practice and was banned in China in 2009. Technical chlordane is dominated by TC (13%), CC (11%), HEPT (5%), and *trans*-nonachlor (5%)[15]. Chlordane chemicals were highly detected OCPs and accounted for 4.31%-11.7% of $\sum_{24}$OCPs [Figure 2]. The relative abundance of chlordane was found in all study sites and showed a stable concentration irrespective of sampling sites [Supplementary Figure 1 and Supplementary Table 2]. The CC and TC concentrations were in the range of 1.52-1.62 ng/g and 1.50-1.69 ng/g, respectively [Table 1]. Higher levels of CC and TC

were detected at A2, A3, and A4 in Jiuquan, and at E3, E4, and E7 in Wuwei [Supplementary Figure 1]. Despite being banned in China for years, the higher detection of Chlordane in many areas suggests that its presence in soil is a result of secondary distribution and specific sources.

The relationship between total organic carbon and OCPs

TOC in soil has the ability to adsorb OCPs due to its hydrophobicity properties. Furthermore, TOC provides a sufficient amount of carbon to reduce the levels of OCPs through bio-degradation[46,47]. Accordingly, TOC can affect the behavior and risk of OCPs in soil. In the present study, TOC in soil was in a range of 4.43-28.4 g/kg with a mean of 11.9 g/kg. To get insights into the role of TOC, the contents of Σ_{24} OCPs, ΣHCHs, and ΣDDTs were plotted with TOC concentration using a scatter plot [Supplementary Figure 2]. The results showed that the OCPs were positively correlated with TOC concentrations in soil (Σ_{24} OCPs with $R^2 = 0.609$, ΣDDTs with $R^2 = 0.671$, and ΣHCHs with $R^2 = 0.409$ at $P < 0.05$ [Supplementary Figure 2]. This suggests TOC in soil plays a crucial role in adsorbing OCPs[25,48]. In an equilibrated soil-air system, there is a proportional relationship between the residual OCP concentrations and the soil organic matter (SOM)[49]. However, if the system is imbalanced or if other complex soil conditions exist, deficient correlations may result[50]. In a previous report, we also noticed a weak correlation between TOC and OCPs[25]. Nevertheless, our current study has found a stable linear correlation between OCPs and TOC, which agrees with prior investigations in Central Europe[17] and the Central Tibetan Plateau[51].

Source apportionment of OCPs in soil

The proportion of parent compound to its metabolites can offer valuable insights into the origin of contamination[52]. The technical HCH compound typically contains around 60%-70% α-HCH, 5%-12% β-HCH, 10%-12% γ-HCH, and 6%-10% δ-HCH (UNEP). In addition, lindane (> 99% β-HCH) was also used, despite the ban on technical HCH[53]. β-HCH is the most easily absorbed by SOM and less likely to evaporate compared to other isomers. Meanwhile, α-HCH and γ-HCH can transform into β-HCH in soil, and the molecules' chlorine atoms in β-HCH resist degradation from microorganisms in soil, causing its accumulation. This study employed the ratios of α-HCH/γ-HCH and β-HCH/(α-HCH+γ-HCH) to trace the source of HCHs. Ratios of α-HCH/γ-HCH greater than 3 but less than 7 indicate technical HCH contamination, whereas a ratio less than 3 suggests new lindane input[54]. For the β-HCH/(α-HCH + γ-HCH) ratio, values over 0.5 denote historical contamination, while lower values indicate pollution from recent pesticide use and atmospheric deposition. Results reveal the presence of HCH contamination in all sampling sites, with low ratios of α-HCH/γ-HCH, indicating early-stage lindane input. The β-HCH/(α-HCH+γ-HCH) ratios are in a range of 0.12-3.57, with 70% of sampling locations > 0.5 [Figure 4A], signifying historical application, mainly in the Zhangye region, indicating contamination of the Hexi corridor with HCHs from past sources and possible new contamination.

Studies indicate that DDT is a commonly used insecticide, with *p,p′*-DDE and *p,p′*-DDD being the primary products resulting from the dechlorination of *p,p′*-DDT[55]. The ratio of *p,p′*-DDT to (*p,p′*-DDE + *p,p′*-DDD) is utilized to determine whether the input of DDT in the environment is increasing or decreasing. A low ratio of *p,p′*-DDT/(*p,p′*-DDE + *p,p′*-DDD) suggests historical DDT use, while a value > 2.0 denotes recent application[56]. As observed in Figure 4B, the majority of values of *p,p′*-DDT/(*p,p′*-DDE + *p,p′*-DDD) were < 2.0 (ranging from 0.204 to 3.70), indicating that DDT in the soil of the sample points primarily originated from the past and had been bio-converted to DDD and DDE. Additionally, a DDD/DDE ratio of less than 1 was observed in 24% of sampling sites, suggesting that DDT had undergone aerobic bio-degradation, influenced by the environment's oxygen content and soil porosity. Notably, the technical endosulfan accounted for 70% and 30% of *α* and *β*-ED, correspondingly, with a ratio of almost 2.33[25]. A ratio of *α*-ED/*β*-ED < 2.33 indicates aged soil residues as the *α*-isomer decomposes more effortlessly in comparison to the β-isomer in soil. The ratio of *cis*-CD/*trans*-CD in technical mixtures is reported to be about 0.77[57].

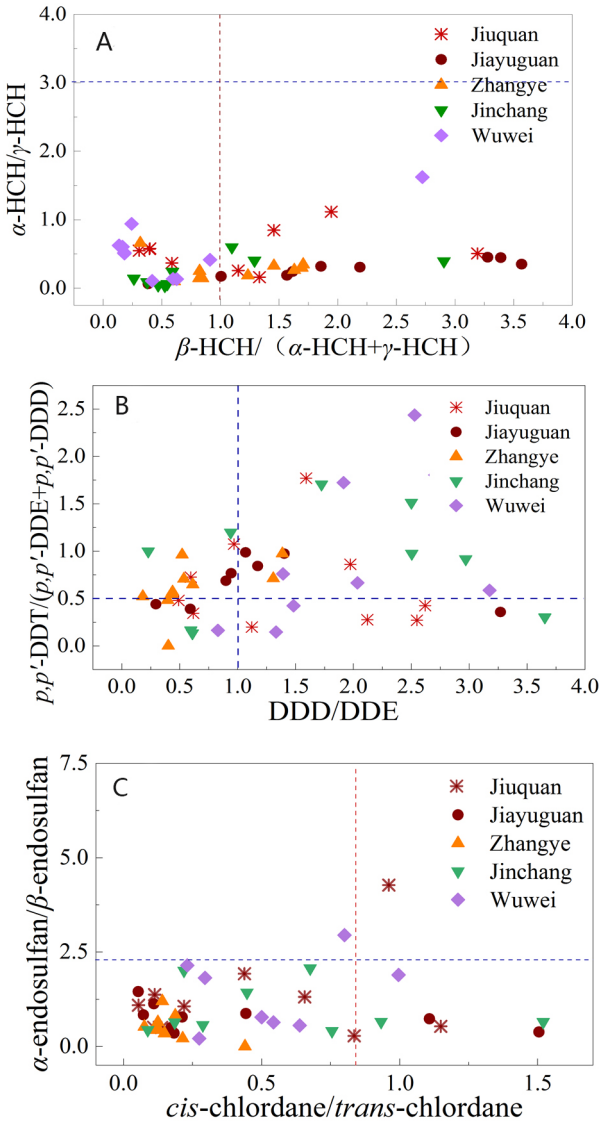

Figure 4. Ratios of the individual isomers of HCH(A); DDT(B); CDs, and EDs (C) in soils

Previous reports showed that *trans*-CD is easier to degrade than *cis*-CD in the environment[58], and the ratio of *cis*-CD/*trans*-CD > 1 is generally indicative of aged chlordane[59]. The concentration range of *cis*-CD was 0.113-4.92 ng/g, with an average of 1.05 ng/g, while *trans*-CD ranged from 0.292 to 21.1 ng/g, with an average of 3.30 ng/g. The *cis*-CD/*trans*-CD ratio range was 0.66-18.8, with a mean value of 5.16 [Figure 4C], indicating that CD residues mainly resulted from past use. Additionally, the level of HE was commonly lower than that of HPE, indicating no recent input in the study areas in Northwest China's Hexi Corridor. Nonetheless, a high level of HE was found in soils of numerous locations, suggesting HE is still being introduced into the study area's soil.

Pollution and risk assessment

Pollution assessment

The soil quality in this study was assessed according to the national standards of China (GB15618-1995), which classifies soil into three levels based on the residual concentrations of ΣDDTs and ΣHCHs. The first

level is less than 50 ng/g, the second level ranges from 50-500 ng/g, and the third level ranges from 500-1,000 ng/g. Out of the 43 soil samples collected, the ΣHCHs content was below the first-level guideline. Additionally, out of the 50 total soil samples, only two collected from A3 and A7 were in the second-level category for ΣDDTs, while none of the samples exceeded the third level. Compared to the soil protection guidelines of the Netherlands [Table 1], the levels of ΣHCHs in all collected soil samples were higher than the target value for uncontaminated soil, but the concentrations of β-HCH and γ-HCH were lower than the target value for unpolluted soil. Similarly, the levels of ΣDDTs were higher than the target value of uncontaminated soil. The target values of HCB, endrin, and CHLs in the soil protection guideline were higher than corresponding OCP levels in some of the collected soil samples. Overall, the HCH pollution of the soil in the Hexi Corridor may be categorized as existing, while the level of DDTs was considered low pollution. HCB, endrin, and CHLs were classified as low contamination for half of the soil samples, with very few samples considered non-contaminated. Although no remediation strategies were deemed necessary for soil OCP pollution, further research is necessary on the adverse ecological and health effects of OCPs' endocrine-disruptive properties, which may lead to biological magnifications in the food chain, including human beings.

Ecotoxicological risk assessment for OCPs

The study evaluates the risk of OCP pollution to soil biota in the Hexi Corridor in Northwestern China. To accomplish this, the Sediment Quality Guidelines (SQG) were used to assess the potential ecological risk of OCPs in the soil[60]. These guidelines utilize two sets of values: the Effect Range Low (ERL) and the Effect Range Median (ERM). The values of ERL, ERM, Threshold Effect Level (TEL), and Probable Effect Level (PEL) for different OCP compounds were analyzed, and the findings were listed in Table 3. The results indicated that some elements did not exceed ERL, but a considerable number of soil samples had a higher concentration of *p,p′*-DDT, *p,p′*-DDE, HCH, and endrin as compared to ERL. 50% of soil samples for *p,p′*-DDE and endrin exceeded the ERL value, while 88% of *p,p′*-DDD and 40% of endrin were between ERL and ERM. Furthermore, 74% of HCH exceeded the ERM value, which suggests that there is an ecological risk to the soil biota due to OCP pollution in the area. Such pollution could potentially harm the ecological environment.

Health risk assessment

Exposure risk assessment of OCPs in soils was conducted by the USEPA method[61]. The non-carcinogenic and carcinogenic risks for children and adults under three exposure pathways (ingestion, dermal contact, and inhalation) were estimated and established upon Equations (1)-(5). The applied equations of OCP exposure in different exposure pathways were presented as follows:

$$CDI_{Ing} = \frac{C \times OSIR \times ED \times EF \times CF}{BW \times AT} \quad (1)$$

$$CDI_{Dermal} = \frac{C \times CF \times SAE \times SSAR \times EF \times ED \times ABS}{BW \times AT} \quad (2)$$

$$CDI_{Inh} = \frac{C_s \times DAIR \times EF \times ED}{PEF \times BW \times AT} \quad (3)$$

where C is the concentration of OCPs, CDI_{Ing} is ingestion exposure, OSIR is daily soil intake, ED is exposure years, EF is the rate of exposure, CF is conversion factor, BW is average weight, AT is average contact time, SAE is skin area touching the soil, SSAR is soil-skin adhesion coefficient, ABS is absorption factor, DAIR is daily air volume, PEF is formation coefficient of soil dust, CDI_{Dermal} refers to dermal contact exposure, and CDI_{Inh} is inhalation exposure. The exposure risk is divided into the carcinogenic risk (CR) and the non-carcinogenic effect (HI).

$$CR = \sum CR_i = \sum (CDI_i \times SF_i) \quad (4)$$

$$HI = \sum HQ_i = \sum \left(CDI_i / RfD_i \right) \quad (5)$$

where CR is the composite index of carcinogenic risk, CR_i represents the three exposure routes, HI is the composite non-carcinogenic risk index, HQ_i expresses the non-carcinogenic risk of an exposure path, CDI_i is the soil exposure amount under a certain exposure path, R_fD_i is the reference dose of a non-carcinogen for an exposure route. The related parameter used in the Eq(1)-Eq(5) is presented in Supplementary Table 2 and Supplementary Table 3.

The CR (HCHs, DDTs, HCB, aldrin, and chlordane) and the HI (HCHs, DDTs, HCB, aldrin, CDs, and EDs) were evaluated in the soil of the Hexi Corridor [Supplementary Table 4]. Generally, CR < 1.0×10^{-6} is an acceptable risk level, while CR ranging from 1.0×10^{-6} to 1.0×10^{-4} indicates a medium cancer risk to humans. CR above 1.0×10^{-4} is considered to be a relatively high cancer risk[14]. Meanwhile, HI > 1 indicates a higher health damage risk, while < 1 indicates a low health risk to humans. Based on the calculated results, the total CR fluctuated in a range of 1.86×10^{-7} to 5.62×10^{-6} for children and 1.21×10^{-7} to 1.91×10^{-6} for adults. Moreover, for the three exposure pathways, the CR through ingestion was higher or equal to the risk through dermal contact, but two or three orders of magnitude higher than the risk through inhalation. The order of the means of carcinogenic risk of OCPs in soil was Jiuquan (1.80×10^{-6} for children, 6.12×10^{-7} for adults) > Jinchang (7.60×10^{-7} for children, 2.58×10^{-7} for adults) > Jiayuguan (7.26×10^{-7} for children, 2.46×10^{-7} for adults) > Wuwei (7.31×10^{-7} for children, 2.48×10^{-7} for adults) > Zhangye (5.60×10^{-7} for children, 1.90×10^{-7} for adults) [Supplementary Table 4]. According to the CR index [Supplementary Table 4], the CR index of Jiuquan is higher than that for other regions, and the carcinogenic risk of OCPs in the soil was higher for children than for adults. HI of OCPs for children and adults ranged from 0.032 to 0.932 (average: 0.458) and 0.093 to 0.281 (average: 0.155), respectively, and all of these were < 1, indicating a low health risk. For HI, HCHs contributed the most, accounting for more than 50% on average, followed by DDT, and CHL [Figure 5]. In summary, fortunately, the levels of OCPs in very few of the collected soil samples have certain carcinogenic and other health risks to humans; however, these should receive further research and management attention. According to the risk index of different exposure routes, the main exposure pathway was ingestion, followed by dermal contact and inhalation. These findings align with prior research indicating that children are a demographic that is more susceptible to the adverse health effects of these pollutants[8,19].

CONCLUSIONS

The results exhibited the content of $\sum_{24}$OCPs ranged from 23.1 to 393 ng/g, with a mean of 94.7 ng/g. The order of the dominant OCPs in the Hexi Corridor soil was as follows: ∑DDTs > ∑HCHs > Heptachlor > HCB > Chlordane > Endosulfan. HCHs and DDTs were much higher than other OCPs, accounting for 38.7% and 16.1% of $\sum_{24}$OCPs, respectively. The order of content of ∑24OCPs (from high to low) in the Hexi Corridor was Jiuquan > Jinchang > Jiayuguan > Wuwei > Zhangye. Source analysis showed that there was recent pesticide use in these areas, and the higher levels of OCPs and unused OCPs suggest that a certain amount of OCPs were, in fact, recently input into the study areas. The results indicated that there was a certain ecological risk of OCP contamination in the soil of the Hexi Corridor, which may cause harm to the ecological environment. The results showed that the main health risk of OCPs in the different areas of the Hexi Corridor was ingestion, followed by dermal contact and oral inhalation, and OCPs in the soil in Jiuquan, in particular, pose carcinogenic and other health risks to humans, demanding vigilant attention.

Table 3. Bio-toxicity risk assessment of OCPs in surface soil of Hexi Corridor

OCPs	ERL	ERM	Range	Sample number ratio /%		
				< ERL	ERM-ERL	> ERM
p,p'-DDE	2.20	27.00	1.09-37.58	16%	82%	2%
p,p'-DDD	2.00	20.00	1.54-40.71	6%	88%	6%
p,p'-DDT	1.00	7.00	1.35-93.58	0	18%	82%
∑DDT	1.58	46.1	4.96-166.97	0	84%	16%
∑HCH	0.02	8.00	3.40-97.48	0	26%	74%
Endrin	1.58	46.10	0.36-9.83	60%	40%	0

ERL: the Effect Range Low; ERM: the Effect Range Median.

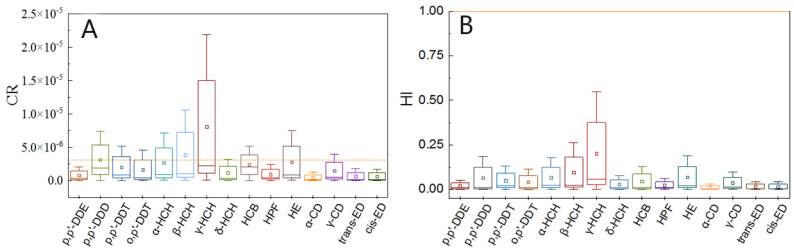

Figure 5. Carcinogenic and non-carcinogenic risk of OCPs in soils. (A): Carcinogenic risk of OCPs in soils; (B): non-carcinogenic risk of OCPs in soils. CR: the composite index of carcinogenic risk; HI: the composite non-carcinogenic risk index.

The present results provide contamination information and a strong rationale for taking measures to control these pollutants in the contaminated sites in order to further guarantee the health of the residents living around the study areas. Future studies should focus on these unique high-contamination sites in the middle latitudes and the input and output sources of the pollutants in the cities in this area.

DECLARATIONS

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Authors' contributions

Writing - original Manuscript Preparation: He R
Experiment, data analysis: Yuan L
Writing - manuscript preparation, reviewing and editing, funding acquisition: Jiang Y
Software, validation analysis: Jia Z
Soil sampling and treatment: Ding W
Instrumental analysis, reviewing and editing: Yang Z

Availability of data and materials

Data published as supplementary information in the journal.

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Conflicts of interest

All authors declared that there are no conflicts of interest.

Ethical approval and consent to participate

Not applicable.

Consent for publication

Not applicable.

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