

Persistent Toxic Substances in Agricultural Soils of Lishui County, Jiangsu Province, China

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Abstract The contamination status and distribution of PTS, including HCHs, DDTs, PCBs, and PAHs, were studied in the agricultural soil of Lishui County, one of China's nationally designated eco-demonstration region. The PTS was analyzed by GC, GC/MS, and HPLC coupled with microwave extraction. The concentrations of the sum of HCHs, DDTs, and PCBs ranged from $<0.1 \text{ ng g}^{-1}$ to 133 ng g^{-1} , 93.8 ng g^{-1} , and 81.0 ng g^{-1} (dry weight), respectively; while the contents of the sum of PAHs ranged from 3.26 ng g^{-1} to 91.0 ng g^{-1} (dry weight). The soil investigation revealed that there were no recent HCHs and DDTs pollution; none of the soil PCBs concentrations suggested a serious biological threat in the investigated area; the extent of pollution of PAHs was low compared to other investigations. In summary, levels of PTS contamination in the soil of the Lishui County were relatively low, but caution should be taken in industrial zone.

Keywords Agricultural soils · HCH · DDT · PCB · PAH · Nationally designated eco-demonstration region · East China

Persistent toxic substances (PTS) are of great environmental concern due to their bioaccumulation, persistence, toxicity, and long-distance transport issues. The behavior of PTS in

soil has been a hot topic of research (Barra et al. 2005). In China research on this topic includes dichloro-diphenyl-trichloroethane (DDT) (Cao et al. 2007), polychlorobiphenyl (PCB) (Mai et al. 2005), and polyaromatic hydrocarbon (PAH) (Ding et al. 2005). The results show that it is necessary to pay attention to PTS pollution in China.

In 1995, China initiated the Nationally Designated Eco-Demonstration Region (NDEDR) Project. One goal was to produce clean and green food supplies for domestic and international consumption. To date, more than 300 counties in China have participated in the NDEDR Project. The assessment index of eco-environmental protection for NDEDRs, announced by China State Environmental Protection Administration in 1995, ignores the factor of 'soil quality', which strongly influences the fate and distribution of pollutants in the environment (Barra et al. 2005), as well as the quality of agriculture products. Also, the study of PTS soil contamination in China rarely focuses on NDEDRs. In this paper the contamination status and distribution of PTS in the agricultural soil of Lishui County, one of China's NDEDRs, were investigated and soil quality was assessed.

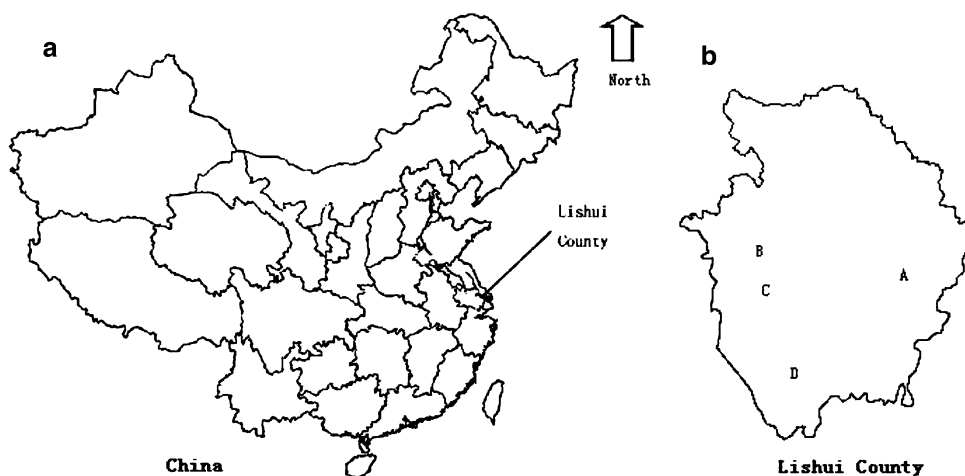
Materials and Methods

Lishui County is located in southern Jiangsu Province, China (Fig. 1). After the rice harvest in November, 2004, 74 soil samples were collected in four agricultural subregions in Lishui: an organic crop production area (Subregion A), the provincial agricultural science and technology park (Subregion B), a non-hazardous rice production area (Subregion C), and a non-hazardous vegetable oil production site (Subregion D) (Fig. 1). Fourteen samples were collected from Subregion A, 30 samples from Subregion B, nine samples from Subregion C, and 21

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Fig. 1 Study area: (a) China; (b) Lishui County with the four subregions studied



samples from Subregion D. All samples were collected from surface soil (0–20 cm depth), and each sample was a composite of three to five sub-samples.

Hexachlorocyclohexane (HCHs) and DDTs standard solution, containing eight components (α -HCH, β -HCH, γ -HCH, δ -HCH, *p,p'*-DDD, *o,p'*-DDT, *p,p'*-DDE, and *p,p'*-DDT) each at 100 mg L⁻¹ in hexane, was obtained from the National Research Center for Certified Reference Materials in China. The PCBs standard solution, containing 20 congeners (Table 1) each at 100 mg L⁻¹, was obtained from Accu-standard, Inc., USA. The PAHs standard solution, containing 16 US EPA priority PAHs (Table 1) each at 100 mg L⁻¹, was obtained from Supelco, USA. Solvents and other chemicals used in this study were hexane (pesticide grade, Merck, Germany), acetone (pesticide grade, Merck, Germany), methanol (HPLC grade, Merck, Germany), and sodium sulfate (analytical grade, Nanjing Chemical Reagent Factory), stored in sealed containers after baking at 600°C for 5 h.

PTS were extracted following the EPA SW-846 microwave extraction Method 3546. About 10 g of the wet soil sample was weighed before being transferred into an extraction vessel with 30 mL of hexane/acetone (1:1, v/v), and then placed into the microwave extraction system MES-1000 (CEM, US) and extracted at 100°C (1,200 w) for 20 min. After extraction, the vessel was cooled down to room temperature. The extract was filtered through a glass funnel with 5 g anhydrous sodium sulphate followed by washing with *n*-hexane/acetone (1:1, v/v) three times. The extract was concentrated in a water bath at 30°C and the solvent was exchanged to hexane during the concentration step. The extract was then purified using a Florisil column (LC-Florisil, 1 g, 6 mL, SUPELCO, USA) and the column eluted with 10 mL *n*-hexane/acetone (9:1, v/v) to obtain the fraction containing HCHs, DDTs, PCBs, and PAHs. The eluate was concentrated by evaporation in a 30°C water bath under gentle nitrogen gas flow to a volume of 1.0 mL. The obtained extract (Extract 1) was first used for HCHs and

DDTs analysis and subsequently for PCBs analysis. The extract was then concentrated and the solvent was exchanged to methanol (Extract 2) and PAHs were analyzed using high performance liquid chromatography (HPLC). An equivalent of 10 g wet soil sample was used to measure water content and the final results were reported on a dry-weight basis.

HCHs and DDTs analysis was performed by gas chromatography with electron capture detection (GC-ECD) on a HP 6890 GC (Agilent, USA) with DB-1701 capillary column (30 m × 0.32 mm I.D., 0.25 μm film thickness). The GC operating conditions were as follows: nitrogen was used as carrier gas at a constant flow rate of 1.0 mL min⁻¹ and make-up gas at a constant flow rate of 29 mL min⁻¹. Injector and detector temperature were optimized at 250°C and 280°C, respectively. The column temperature program started at 50°C (held for 5 min), set at 35°C min⁻¹ to 220°C and was held for 20 min. The sample injection volume was 1.0 μL. The injection was splitless for 0.75 min with the split flow rate at 30 mL min⁻¹. The GC peaks were identified by accurate assignment of retention times for each standard within ±1%. HCHs and DDTs standard solutions were analyzed at concentration of 5, 20, 50, and 100 μg L⁻¹ to establish calibration curves.

PCBs analysis was performed by gas chromatography-mass spectrometry (GC/MS) on a HP 6890 GC/5973 MSD (mass selective detector) (Agilent, USA) equipped with a DB-5MS capillary column (30 m × 0.25 mm I.D., 0.25-μm film thickness). The GC operating conditions were as follows: helium was used as the carrier gas at a constant flow rate of 1.0 mL min⁻¹. The temperature program was set from 40°C to 280°C with an incremental rate of 35°C min⁻¹ and held at 280°C for 5 min. Injector and interface temperature were 300°C and 280°C, respectively. The sample injection volume was 1.0 μL. The injection was splitless for 0.75 min with a solvent delay of 5 min. The MS was operated in the electron impact ionization mode (electron energy 70 eV), and the ion source temperature was 230°C. The

Table 1 List of target PTS

PAHs	PCB congeners	HCHs	DDTs
Acenaphthene (Ane)	2,4'-dichlorobiphenyl (PCB8)	α -HCH	p,p'-DDD
Anthracene (Ant)	2,2',5-trichlorobiphenyl (PCB18)	β -HCH	o,p'-DDT
Benz[a]anthracene (Baa)	2,4,4'-trichlorobiphenyl (PCB28)	γ -HCH	p,p'-DDE
Benzo[a]pyrene (Bap)	2,2',3,5'-tetrachlorobiphenyl (PCB44)	δ -HCH	p,p'-DDT
Chrysene (Chr)	2,2',5,5'-tetrachlorobiphenyl (PCB52)		
Dibenz[a,h]anthracene (Daa)	2,3',4,4'-tetrachlorobiphenyl (PCB66)		
Fluoranthene (Fla)	3,3',4,4'-tetrachlorobiphenyl (PCB77)		
Fluorene (Fle)	2,2',4,5,5'-pentachlorobiphenyl (PCB101)		
Naphthalene (Nap)	2,3,3',4,4'-pentachlorobiphenyl (PCB105)		
Phenanthrene (Phe)	2,3,4,4',5-pentachlorobiphenyl (PCB118)		
Pyrene (Pyr)	3,3,4,4',5-pentachlorobiphenyl (PCB126)		
Benzo[b]fluoranthene (Bbf)	2,2',3,3',4,4'-hexachlorobiphenyl (PCB128)		
Acenaphthylene (Any)	2,2',3,4,4',5'-hexachlorobiphenyl (PCB138)		
Benzo[k]fluoranthene (Bkf)	2,2',4,4',5,5'-hexachlorobiphenyl (PCB153)		
Benzo[g,h,i]perylene (Bgp)	2,2',3,3',4,4',5-heptachlorobiphenyl (PCB170)		
Indeno[1,2,3-c,d]pyrene (I1p)	2,2',3,4,4',5,5'-heptachlorobiphenyl (PCB180)		
	2,2',3,4',5,5',6-heptachlorobiphenyl (PCB187)		
	2,2',3,3',4,4',5,6-octachlorobiphenyl (PCB195)		
	2,2',3,3',4,4',5,5',6-nonachlorobiphenyl (PCB206)		
	2,2',3,3',4,4',5,5',6,6'-decachlorobiphenyl (PCB209)		

selective ion monitoring (SIM) mode was used to detect the target PCBs congeners according to their characteristic m/z values (Table 2).

PAHs analysis was performed using a Waters 2695 HPLC equipped with 2475 Fluorescence Detector and 2996 Diode Array Detector (Waters, US). The HPLC method used for the separation of PAHs consisted of a Waters PAH C18 column (4.6 × 250 mm, 5 μm-particle size) held at 27°C and mobile phase at a constant flow rate of 1.2 mL min⁻¹ with gradient elution program as follows: 60/40 (v/v) acetonitrile/water to 100% acetonitrile in 12 min. The sample injection volume was 20 μL. Both the fluorescence detector and the

UV detector were used for PAHs determination. The excitation wavelength (λ_{ex}) and emission wavelength (λ_{em}) of the fluorescence detector were 275 nm and 350 nm, respectively. The UV detector scanned from 200 nm to 380 nm and the quantification wavelength was 254 nm. Naphthalene (Nap), acenaphthalene (Ane), pyrene (Pyr), and indeno[1,2,3-cd]pyrene (I1p) were detected using diode array detector and the other twelve PAHs listed in Table 1 were detected using fluorescence detector.

Results and Discussion

The method detection limits (MDLs) for the target PTS were calculated based on the standard deviations of analytical results from seven parallel blank matrix samples spiked with low concentrations of target standards. The analytical procedure of MDLs was identical to that of sample analysis, including sample preparation. The MDLs and recoveries of PTS, listed in Table 3, were sufficient to meet the data quality requirements for this study.

The results of HCHs and DDTs measurement are shown in Table 4. In the 74 samples, the detectable frequencies of α -HCH, β -HCH, γ -HCH, and δ -HCH were 5.5%, 84.9%, 2.2%, and 2.2%, respectively, while those of p,p'-DDD, o,p'-DDT, p,p'-DDE, and p,p'-DDT were 33.1%, 1.2%, 2.5%, and 27.7%, respectively. The concentrations of HCHs and DDTs in the soil of Lishui County were far

Table 2 Selective m/z values of target PCB congeners

Compound	m/z*	Compound	m/z*
PCB8	222,152	PCB126	324
PCB18	256,186,150	PCB128	360,290
PCB28	256,186,150	PCB138	360,290
PCB44	292,255,127	PCB153	396,324
PCB52	292,255,127	PCB170	394,324
PCB66	292,254,127	PCB180	394,324,162
PCB77	292,254	PCB187	394,324,162
PCB101	326,254,127	PCB195	430,358,144
PCB105	326,254	PCB206	464,392,196
PCB118	326,254	PCB209	498,214,178

* The first m/z value was used for quantification

Table 3 MDLs and recoveries for target PTS

Targets	Analytical method	MDLs (ng g ⁻¹)	Recovery (%)	RSD (%)
HCHs	GC/ECD	0.10	55.0–75.3	0.9–2.4
DDTs	GC/ECD	0.10	71.5–98.5	1.5–3.1
PCBs	GC/MS	0.20	57.0–94.9	1.1–3.5
PAHs	HPLC/FL/DAD	0.01 (0.25*)	94.2–110	0.6–4.0

* Only for Nap, Ane, Pyr, and I1p

below the China National Soil Quality Standard (GB15618-95) level 2 value of 500 ng g⁻¹.

Results of correlation analysis indicated that significant relationships were found between the concentration of β -HCH and total soil HCHs, and between p,p'-DDT and total DDTs, with the correlation factors of 0.996 and 0.976, respectively ($p < 0.01$).

The relatively high content of β -HCH observed in this study is consistent with the results of the vegetable field investigation conducted in Guangzhou, China (Chen et al. 2005). The technical HCHs used in China contain 67% α -HCH, 10% β -HCH, 15% γ -HCH, and 8% δ -HCH. Note that all chlorine atoms in the β -HCH molecule occupy equatorial positions, which causes β -HCH to have the lowest evaporation pressure among the HCH isomers and thus be the most persistent isomer by accumulating in the soil. Although β -HCH is not the main component in the mixture of technical HCHs, the relatively high concentration of β -HCH indicates long-term accumulation of pollution.

DDT degrades to dichlorodiphenyldichloroethane (DDD) under anaerobic conditions and to dichlorodiphenyldichloroethylene (DDE) under aerobic conditions. In this study, among the samples in which DDT could be detected, generally

the proportions of DDE/DDT were higher than those of DDD/DDT at the same sampling site (Fig. 2). This indicates that DDEs are the predominant degradation products of DDT in Lishui County, which is consistent with other report (Lu et al. 2000) that under aerobic conditions, the major degradation products of DDTs are DDEs.

The concentration of HCHs in the soil samples was higher than that of DDTs, which may be due to the fact that in China HCHs are used more frequently (90% of total organochlorine pesticides) than DDTs, especially in paddy soils (Lu et al. 2000).

The average concentrations of the sum of all the twenty PCB congeners were 21.09, 12.21, 9.87, and 12.25 ng g⁻¹ for Subregions A, B, C, and D, respectively. Compared with the concentration of the sum of thirteen PCBs (4.13 ng g⁻¹) in agricultural soils of other counties in Jiangsu Province, China (Zhang et al. 2007), the contents of PCBs in the soil of Lishui County were much higher. But compared with target level of the sum of five PCBs (20 ng g⁻¹) in ambient soil approved by the Netherland in 1994 (Zhou and Song 2004), the soil of Lishui County was in the “clean soil” category. Conclusion could be drawn from the results above that there is no obvious PCBs pollution problem in the agricultural soil of Lishui County.

Long et al. (1995) developed an effects range-low (ERL) standard and an effects range-median (ERM) standard intending to assess the biological effects of pollutants in the environment. The ERL and ERM guideline values for PCBs were set at 22.7 ng g⁻¹ and 189 ng g⁻¹, respectively. According to the criteria, 17 samples collected in Lishui County contained PCBs at concentrations deemed to have potential adverse biological effects. The concentrations of PCBs in these 17 samples ranged from

Table 4 PTS detected in the 74 samples from agricultural soil

Targets	Median (μg/kg)	Minimum (μg/kg)	Maximum (μg/kg)	Skewness	Kurtosis	Standard Deviation (μg/kg)
α -HCH	0.00	ND*	3.09	1.29	0.48	0.81
γ -HCH	0.00	ND*	6.57	3.77	15.1	1.15
β -HCH	7.08	ND*	130	2.74	7.92	24.9
δ -HCH	0.00	ND*	6.57	3.77	15.1	1.15
Σ HCHs	8.46	ND*	133	2.74	7.99	25.8
pp'-DDD	0.00	ND*	8.77	1.39	2.60	1.79
op'-DDT	0.00	ND*	25.1	6.96	50.67	3.21
pp'-DDE	0.00	ND*	4.31	1.75	1.79	1.17
pp'-DDT	0.00	ND*	68.9	1.63	2.21	16.2
Σ DDTs	2.50	ND*	93.8	2.05	5.16	18.3
PCBs	5.75	ND*	81.0	1.76	2.47	18.5
PAHs	10.95	3.26	91.0	3.72	17.8	13.2

* Means less than MDLs

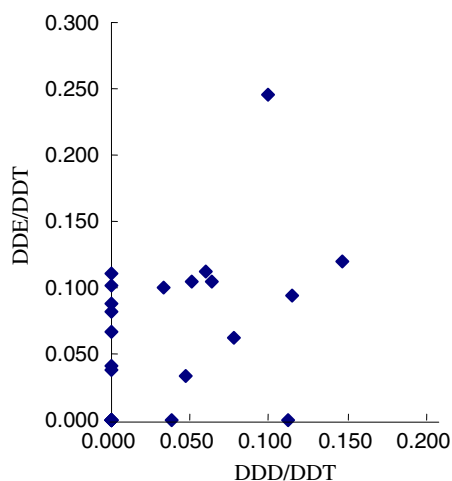


Fig. 2 Comparison of DDE/DDT to DDD/DDT

23.5 ng g⁻¹ to 81.0 ng g⁻¹, which are higher than the ERL guideline. Of the 17 samples, six were from Subregion A, five were from Subregion B, one was from Subregion C, and five were from Subregion D. The average ratio of PCBs concentration: ERL was 1.9, 2.2, 2.3, and 1.8, for Subregions A, B, C, and D, respectively. Since PCBs concentrations of all the samples were much lower than the ERM guideline value, none of the PCBs content is considered a serious biological threat.

It was notable that Subregion A showed relatively higher PCBs contamination than the other subregions in Lishui County. In Subregion A the average PCBs concentration was near the ERL guideline value, and there were more samples with PCBs content above the ERL guideline value than other subregions. This can be tentatively attributed to the adjacent industries which are potential sources of PCBs, suggesting that extra attention should be given to this location.

Of the twenty PCB target congeners, eight PCBs (PCB8, PCB 18, PCB101, PCB105, PCB118, PCB128, PCB138, and PCB153) were frequently detected. PCB118 showed the highest concentration among the congeners, followed by PCB105, PCB128, and PCB153 (Fig. 3). Both PCB118 and PCB105 are dioxin-like mono-ortho PCBs with toxic equivalent factors of 0.0001 established by the WHO (WHO-TEFs). In this study, Pentachlorinated and hexachlorinated PCBs make-up 75.6% of the total PCB congeners. This may be because PCB congeners with less chlorine atoms are more prone to leaching because of their higher solubility, while congeners with more chlorine atoms tend to accumulate in the soil and do not readily degrade (Mai et al. 2005).

The average sum of PAH concentrations were 18.58, 8.52, 18.92, and 16.05 ng g⁻¹ for Subregions A, B, C, and D, respectively. Acenaphthylene (Any) and Pyr were not detected in any of the samples. Dibenz[ah]anthracene

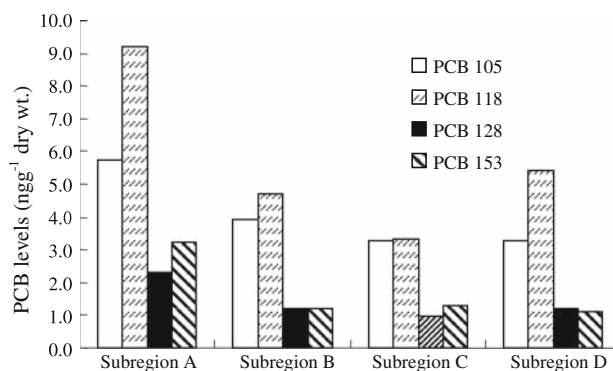


Fig. 3 Priority PCBs detected in soil samples of Lishui County

(Daa) had the highest concentration among the PAHs, followed by benzo[b]fluoranthene (Bbf), benzo[a]anthracene (Baa) and benzo[k]fluoranthene (Bkf), with average concentrations of 5.16, 2.52, 2.26, and 2.02 ng g⁻¹ for Subregions A, B, C, and D, respectively (Figs. 4 and 5).

The concentrations of most PAHs in the sampling sites were above the background level (1–10 ng g⁻¹), but were far below those reported for Wujiang County paddy soils (219.5–1,628.6 ng g⁻¹) (Ding et al. 2005) and those reported for Guangzhou vegetable soils (42–3,077 ng g⁻¹) (Chen et al. 2005). As soil with PAHs concentration between 200 ng g⁻¹ and 600 ng g⁻¹ (dry-weight) is considered weakly contaminated (Maliszewska-Kordybach 1996), soil contamination by PAH in Lishui County is minor. PAHs with four aromatic rings were mostly detected in this study, followed by PAHs with five, six, three, and two aromatic rings, respectively. PAHs with four and five aromatic rings constituted more than 80% of the total PAHs, of which four-ring PAHs constituted 43–50%, and five-ring PAHs constituted 31–43%, indicating that PAHs in soil of Lishui County originated from the combustion of coal. Since five- and six-ring PAHs are proved to be strongly carcinogenic (Ding et al. 2005), the PAH contamination sources in Lishui County should be closely monitored.

The concentrations of PAHs with four rings such as Baa and chrysene (Chr) had a close relationship and those of PAHs with five rings such as Bbf, Benzo(a)pyrene (Bap), Daa, Bkf, I1p, and Bbf also showed good correlations, indicating they might come from the same source. The results of primary component analysis showed that in the first component, the coefficients for Baa, Bbf, Bkf, Bap, I1p, and Daa are high (see factor 1 of Table 5), also indicating they have the same source.

Adami et al. (2000) proposed that in river sediments, if the concentration ratio of phenanthrene (Phe) to acenaphthene (Ant) was greater than 10, PAHs were mainly from petroleum sources, while if Phe/Ant < 10,

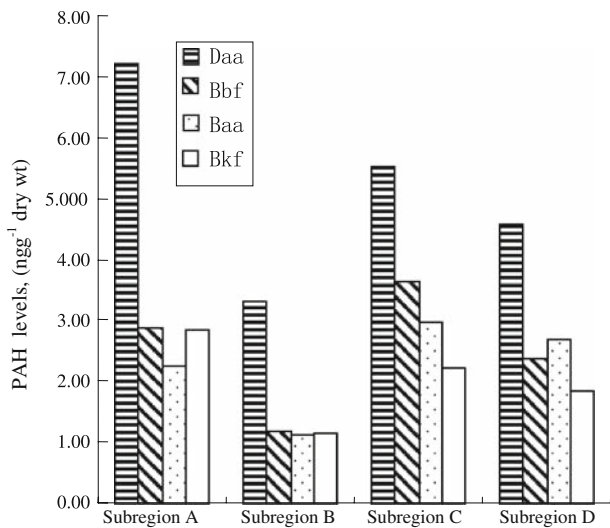


Fig. 4 The priority PAHs detected in soil samples of Lishui County

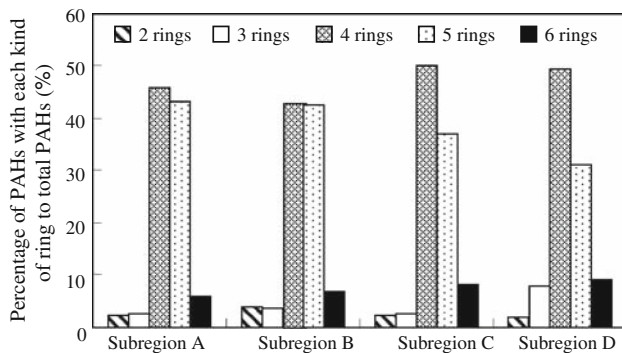


Fig. 5 Percentages of PAHs with different rings in soil samples of Lishui County

PAHs were mainly from coal combustion. Rogge et al. (1993) discovered that if the concentration ratio of fluoranthene (Fla)/Pyr >1, the combustion source is mobile exhaust, while Fla/Pyr <1 means the main source is from petroleum. Phe/Ant ratio in most of the soil samples in Lishui County were 0 and Pyr was not

detected in any sample, indicating that PAHs in Lishui County soil originated from coal combustion. The concentrations of the sum of PAHs were relatively higher in Subregion A and C. There is a pharmaceutical factory with coal consumption of 1,100 tons acre⁻¹ near Subregion A and a kiln factory with coal consumption of 800 tons acre⁻¹ near Subregion C, which could be the main sources of PAHs pollution.

Generally, the soil investigation in Lishui County, China revealed that the concentrations of the sum of HCHs ranged from <0.1 ng g⁻¹ to 133 ng g⁻¹, in which β-HCH was the most readily detected residue, indicating there was no recent HCHs pollution. The levels of the sum of DDTs ranged from <0.1 ng g⁻¹ to 93.8 ng g⁻¹. DDE was the predominantly degradation product of DDT under aerobic conditions. The content of β-HCH showed good correlation with the concentration of the sum of HCHs, while the concentrations of p,p'-DDT and the sum of DDTs also had a good correlation. The concentrations of the sum of PCBs ranged from <0.1 ng g⁻¹ to 81.0 ng g⁻¹. Pentachlorinated and hexachlorinated PCBs made up 75.6% of the total PCBs. The concentration of PCB118 was the highest among the congeners detected, followed by concentrations of PCB105, PCB128, and PCB153. However, none of the soil PCBs concentrations suggested a serious biological threat in the investigated area. The contents of the sum of PAHs ranged from 3.26 ng g⁻¹ to 91.0 ng g⁻¹, with a median of 10.95 ng g⁻¹. The extent of pollution of PAHs was low compared to other investigations. Among all PAHs detected Daa had the highest concentration, followed by Bbf, Baa, and Bkf. PAHs with four aromatic rings were dominant in this study, followed by PAHs with five, six, three, and two rings. Primary component analysis showed that PAHs with five rings such as Baa, Bbf, Bkf, Bap, I1p, and Daa had the same source. The ratio of Phe/Ant suggested that PAHs were mainly from combustion of coal. In summary, levels of PTS contamination in the soil of the Lishui County were relatively low, but caution should be taken in industrial zone.

Table 5 Correlation factors between concentrations of seven PAHs with 4–5 rings and principal component analysis

	Baa	Chr	Bbf	Bkf	Bap	I1p	Daa	Factor 1	Factor 2
Baa	1.000**							0.882	0.410
Chr	0.803*	1.000**						0.482	0.872
Bbf	0.897*	0.475	1.000**					0.992	-0.016
Bkf	0.617	0.340	0.780	1.000**				0.850	-0.026
Bap	0.675	0.104	0.902**	0.576	1.000**			0.863	-0.391
I1p	0.689	0.122	0.910**	0.584	1.000**	1.000**		0.871	-0.374
Daa	0.529	0.247	0.726	0.994**	0.543	0.548	1.000**	0.801	-0.100

* *p* < 0.10

** *p* < 0.05

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