

Distribution and Accumulation Characteristics of Heavy Metals in Sediments in Southern Sea Area of Huludao City, China

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Abstract: The southern sea area of the Huludao City, Liaoning Province might be polluted by heavy metals because it is close to the Jinzhou Bay, one of the heaviest sea area polluted by heavy metals in China. The undisturbed modern sediment core can be used to analyze the accumulation and source of the pollutants using ^{137}Cs and $^{210}\text{Pb}_{\text{ex}}$. Thirty-five samples of surface sediment and two core sediments were collected from the southern sea area of Huludao City. The concentrations of copper (Cu), lead (Pb), chrome (Cr), zinc (Zn), arsenic (As) and mercury (Hg) in the surface sediments as well as Cu, Pb, Zn, Cr, ^{137}Cs and $^{210}\text{Pb}_{\text{ex}}$ in the core sediments were determined to research the spatial distribution and accumulation characteristics, and to analyze the sources and the potential risks of heavy metals. The results show that the pollution levels of Zn and Hg are serious, and 26 stations are at moderate or heavy ecological risks. The concentrations of the heavy metals increase from east to west, as well as from open sea to offshore marine area. The concentrations of heavy metals are not high in the sediments adjacent to the Jinzhou Bay, and the influence caused by the seawater exchange with the Jinzhou Bay is little. The concentrations of the heavy metals in the core sediments show low-high-low characteristic, and it coincides with the pollution history of Huludao City. The atmospheric deposition of heavy metals from the Huludao Zinc Plant is likely to be the main source of pollution without direct discharge of wastewater. The high concentrations of heavy metals appear on the upper sediment of 20 cm. The shallow sediment with high heavy metal contents might be exposed to surface when it was disturbed by the ocean engineering and big storm surge, then cause risk to the safety of aquaculture and human healthy.

Keywords: heavy metals; accumulation characteristic; sediment; Huludao City; China

Citation: Wang Yan, Liu Ruhai, Fan Dejiang, Yu Ping, Wang Jinyu, Tang Aikun, 2013. Distribution and accumulation characteristics of heavy metals in sediments in southern sea area of Huludao City, China. *Chinese Geographical Science*, 23(2): 194–202. doi: 10.1007/s11769-012-0579-0

1 Introduction

Heavy metals continue to be introduced into estuarine and coastal environment through rivers, runoff and land-based point sources, and thus, heavy metal contamination is still a major environmental problem throughout the world. Most heavy metals that enter the marine environment will settle and become incorporated into sediments with other organic matter, along with Fe/Mn oxides, sulfides, and clay acting as a sink (Salomons and Stigliani, 1995; Wang and Chen, 2000; Peng *et al.*,

2009). These sediment-bound metals may be subsequently released to the overlying water as a result of either physical disturbance or diagenesis (Boughriet *et al.*, 1992), and can affect the health of the marine ecosystem (Bryan and Langston, 1992). Heavy metals would be released and distributed again and produce 'secondary pollution' due to the change of hydrodynamic conditions and the influence of biological activities (Theofanis *et al.*, 2001). The heavy metals in the sediments could not only do harm to aquatic organisms, but also affect human health through food chain. Thus,

Received date: 2012-02-23; accepted date: 2012-05-11

Foundation item: Under the auspices of National Natural Science Foundation of China (No. 40806045), Ocean Public Welfare Scientific Research Project, State Oceanic Administration People's Republic of China (No. 201105005)

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the metal content in sediments can reflect the pollution level of the water and geochemical behavior of the metals. The core sediments recorded the pollution history of aquatic ecosystem in previous researches (Lopez and Lluch, 2000; Mohamed, 2005; Nabi-Bidhendi and Bayati, 2005). The radioisotopes ^{137}Cs and $^{210}\text{Pb}_{\text{ex}}$ (half-life is 22.3 yr) have been widely used in the modern sediments to determine core chronology of sedimentary compartments including rivers, estuaries and oceans on scales of several decades to 100 years (Wan *et al.*, 1987; Baskaran and Naidu, 1995; Appleby, 1998; Sarin, 2000; Su and Hu, 2002; Roos and Valeur, 2006; Du *et al.*, 2010).

The Jinzhou Bay of Liaoning Province is one of the most serious heavy-metal polluted sea areas in China because of the large amounts of pollutant discharges (Ma and Shao, 1994; Zhang *et al.*, 2008). The contents of heavy metals in the sediments and water decreased due to the decrease of the pollutants contents in the Wulihe River. However, in recent years, the heavy metals had a tendency of migrating to outside of the bay (Wan *et al.*, 2009). The seawater of the Jinzhou Bay flowed out from the northwest to the southeast at ebb tide, then imported to the southwest ebb-tide of the Liaodong Bay (Fu and Liu, 1994; Liu, 1995), so the high polluted seawater of the Jinzhou Bay might be carried to the southern sea area of Huludao City. The results of previous researches show that the contents of zinc (Zn) and lead (Pb) in the sediments of the Jinzhou Bay were primarily associated with the non-residual fraction, and were easily dissolved into the water or used by living beings (Zhang, 2008).

During 1988–2006, the area of land reclamation at the Jinzhou Bay reached 23.63 km² (Li *et al.*, 2011), and the coastal environment has been changed severely, then the sediment might be re-suspended and heavy metals in the sediment might be released to seawater. The southern sea area of Huludao City is separated into two parts by Huludao Peninsula, the north sea area is the Jinzhou Bay polluted by the heavy metals, and the southern sea area is an important aquaculture farm and seaside tourism holiday zone of Huludao City. There is less information available in the literature regarding the spatial distribution, sources and the potential hazards of heavy metals.

In this study, we report the content and distribution of heavy metals in surface sediments of the southern sea area of Huludao City. Our primary goals are: 1) to characterize the spatial distribution and concentrations

of heavy metals in the southern sea area of Huludao City; 2) to explore accumulation characteristics, pollution history of the heavy metals using isotope analysis of ^{37}Cs and $^{210}\text{Pb}_{\text{ex}}$; and 3) to analyze the sources and the potential hazards of heavy metals.

2 Materials and Methods

2.1 Sampling and determination of heavy metals

Thirty-five samples of the surface sediments were collected in the southern sea area of Huludao City using grab-bucket sampler (Fig. 1). Two core sediments were collected at the Station 5 and the Station 28 by diver using precleaned cylindrical plastic tube. The sediment samples were carefully shipped to the Key Laboratory of the Marine Environment and Ecology of the Education Ministry at the Ocean University of China, where they were processed and preserved. In the laboratory, the surface sediment samples were air-dried and homogenized to pass 60-mesh nylon sieve.

The samples were digested by $\text{HNO}_3+\text{HClO}_4+\text{HF}$ in a Teflon tube using heating block. The digestion liquid was heated until the solid residue disappeared and the solution turned into white or light yellow-greenish pasta like material. The concentrations of Cu, Pb, Zn and Cr in the sediments were determined by atomic absorption spectrophotometer (Thermo, USA, M6). Hg and As were determined by atomic fluorescence spectrophotometry (BTI, CHN, AFS-920) after the samples were digested by $\text{HNO}_3\text{-HCl}$ (1 : 3 v/v).

The stream sediment standard material (GBW07302) and the offshore marine sediments standard material (GBW07314) were used for quality control. Three parallel samples were determined, and the relative standard deviations were all less 5%.

Two core sediments were collected at the Station 5 and the Station 28 by the diver using precleaned cylindrical plastic tube. The depths of the core sediments were 76 cm and 56 cm at the Station 5 and at the Station 28, respectively. Every 2 cm of the sediments was taken as a sample to determine Cu, Pb, Zn and Cr. There were 66 samples totally. The determination of the metals was same to the surface sediment.

2.2 Determination of $^{210}\text{Pb}_{\text{ex}}$ and ^{137}Cs in core sediment

Every 2 cm of the core sediments was taken as a sample for the upper 16 cm of the sediments, while every 6 cm

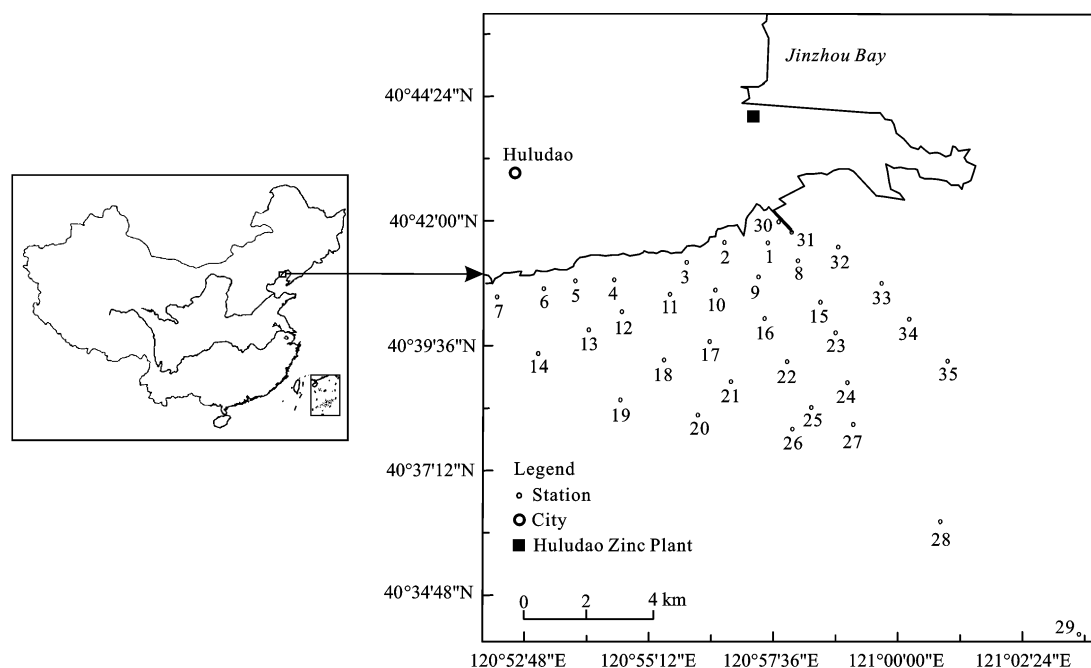


Fig. 1 Sampling sites in southern sea area of Huludao City

of the sediments was taken as a sample for the other depth, then there were 26 samples to determine $^{210}\text{Pb}_{\text{ex}}$ and ^{137}Cs . The activities of $^{210}\text{Pb}_{\text{ex}}$, ^{137}Cs were determined by gamma ray spectrometer analysis system consisted of high purity germanium detector (Ortec HPCe GWL) produced by EG&G Ortec Company of United States and Ortec 919 controller and 16k multi-channel parsers constituted of IBM microcomputer.

Before testing, water content of the samples was determined, and then the dry density of each sample was calculated, then organic matter was removed. After the samples were dried and grinded to pass 0.15 mm aperture sieve, 2–5 g of dry samples were put into specialized testing sample box for sealing 15 days. A variety of nuclide contents of the samples were determined simultaneously by gamma analysis method. The standard sample of ^{137}Cs was provided by China Institute of Atomic Energy, and the standard samples of $^{210}\text{Pb}_{\text{ex}}$ were checked in British Liverpool University.

3 Results and Analyses

3.1 Distribution characteristics of heavy metals in sediment

Zn and Hg were the main pollutants according to the data shown in Table 1. The rates over first class standard of Marine Sediment Quality (GB 18668-2002) were

above 30%. The concentrations of Pb met to the sediment quality standard. The rates of Cr, As were 11.43% and 2.86%, respectively, and were lower compared to Zn and Hg. Potential ecological risk index proposed by Hakanson (1980) was calculated according to the method used in the Jinzhou Bay (Chen, 1992; Fan *et al.*, 2006). The results showed that 10 stations at the west of the sea area were at heavy ecological risk and 16 stations at the middle of the sea area were at moderate ecological risk, the left 9 stations were at lower ecological risk. So the heavy metals at the west stations close to the land might threaten the safety of fishery production.

Table 1 Concentrations of heavy metals in surface sediments

Heavy metal	Concentration range (mg/kg)	Average value (mg/kg)	Rate over first class standard of Marine Sediment Quality (%)
Pb	5.17–51.10	32.20	0.0
Cr	32.3–127.9	75.0	11.4
Zn	56.0–237.9	143.9	34.3
Cu	13.9–39.9	25.9	5.7
As	6.29–23.20	12.00	2.9
Hg	0.035–0.323	0.169	31.4

As shown in Fig. 2, the concentrations of heavy metals increased from the east to the west, as well as from open sea to offshore marine area. The concentrations of heavy metals were low in the sediments adjacent to the

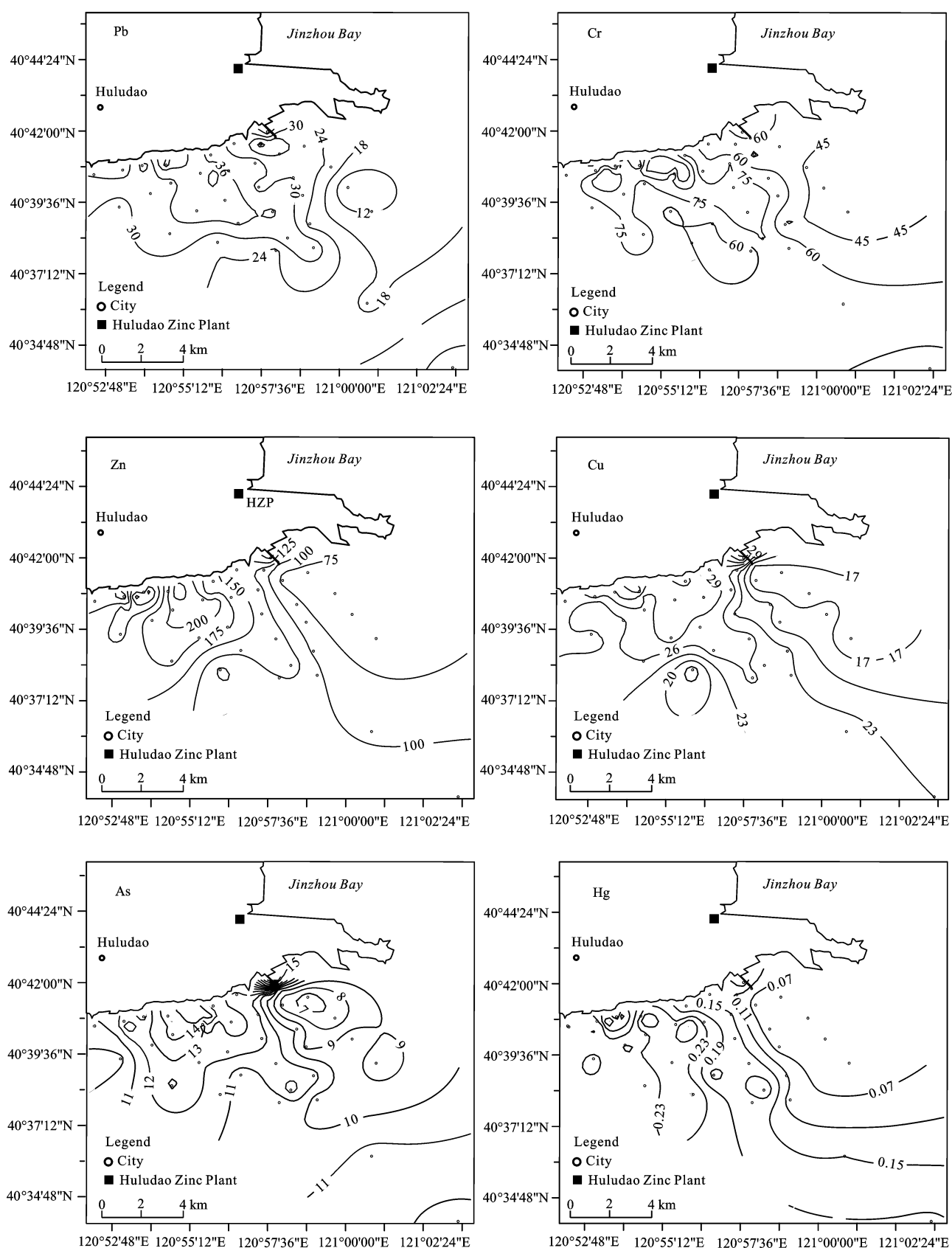


Fig. 2 Horizontal distribution of heavy metal concentrations in surface sediments (mg/kg)

Jinzhou Bay, and the influence caused by the seawater exchange with the Jinzhou Bay was little. The heavy metals in this sea area might come from the discharges from coast and offshore aquaculture pollution.

There is no river flowing into the sea on the southern coast of Huludao City, and the industrial and municipal wastewater was mostly discharged to the Jinzhou Bay, North of Huludao Peninsula, so the discharged wastewater was not the main source of heavy metal pollution in the sea area. The results of previous studies showed that the main source of soil pollution of heavy metals in Huludao City was atmospheric emissions of the Huludao Zinc Plant (Zheng *et al.*, 2009). The heavy metals in the air would deposit to the adjacent soil and sea area, and those heavy metals in the soil were carried to the offing by runoff from the adjacent land. According to the research on the pollution of soil in Huludao City (Lu *et al.*, 2010), dust precipitation from the Huludao Zinc Plant was the primary source of the pollutants. They estimated that the average deposition rates of Cd, Pb and Zn were 0.33 g/(m²·yr), 1.75 g/(m²·yr), and 30.97 g/(m²·yr), respectively at 1 km away from the Huludao Zinc Plant, and 0.0048 g/(m²·yr), 0.035 g/(m²·yr), and 0.20 g/(m²·yr), respectively at 10 km away from the Huludao Zinc Plant. Runoff carried the soil particle to offshore, and then settled in sediment. The metals also were brought to the sea by the atmospheric deposition directly. Therefore, it could be concluded that the Huludao Zinc Plant was the source of the heavy metal content in sediment in the southern sea area of Huludao City.

3.2 Accumulation characteristics of heavy metals in sediment

3.2.1 Sedimentation rate and accumulation characteristics of heavy metals at Station 5

As shown in Fig. 3, at the Station 5, the concentration of radionuclide ²¹⁰Pb_{ex} was the highest in the surface, then it decreased gradually from the surface to the bottom, and the distribution pattern of ²¹⁰Pb_{ex} showed a typical decay structure. The concentrations of radionuclide ¹³⁷Cs showed a low-high-low pattern from the surface to the bottom, which conformed to human nuclear test in the atmosphere. Both ²¹⁰Pb_{ex} and ¹³⁷Cs concentrations indicated that the Station 5 had good sedimentary continuity and there was no abnormally rapid depositon at the station recently.

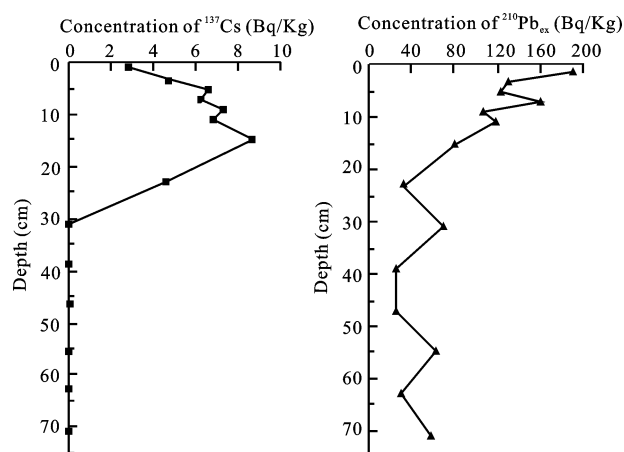


Fig. 3 Concentrations of radionuclide ¹³⁷Cs and ²¹⁰Pb_{ex} in core samples at Station 5

It was apparent that ¹³⁷Cs presented the residues (4.596 Bq/kg) at the depth of 23 cm and the accumulation peak (8.637 Bq/kg) at the depth of 16 cm (Fig. 3). According to the distribution pattern of ¹³⁷Cs in lake sedimentary core of the northern hemisphere, the residual layer of ¹³⁷Cs corresponded to the beginning of global nuclear tests in 1954, while the main accumulation peak layer corresponded to the global scattering peak of ¹³⁷Cs in 1963. However, the results could be checked by ²¹⁰Pb_{ex} dating because of the sampling densities. ²¹⁰Pb_{ex} dating required the content of ²¹⁰Pb_{ex} in the core with exponential decay, at least, sectionally exponential decay. This distribution pattern is the premise of estimating deposition rate using ²¹⁰Pb_{ex} because it could prove that core or one section of rocks did not be disturbed (Liu, 2006). According to the distribution of ²¹⁰Pb_{ex}, segmentationally Constant Initial of Concentration model (CIC) could be used to calculate the sediment deposition rate (Shi and Qiu, 2008).

For the first three sites, the fitted curve between the depth and ²¹⁰Pb_{ex} activity was $y = 202.99e^{-0.1102x}$ ($R^2 = 0.85$), and the average sediment deposition rate calculated by the equation was 0.36 cm/yr. From the fourth site to eighth site, the fitted curve is $y = 298.83e^{-0.0936x}$ ($R^2 = 0.96$), and the average sediment deposition rate was 0.31 cm/yr. According to the results, the sedimentary era of each layer was calculated (Table 2). Because of long sampling interval, ²¹⁰Pb_{ex} dating was adopted in this study.

The vertical distribution of heavy metals at the Station 5 is shown in Fig. 4. The concentrations of Pb, Cu and Zn increased obviously firstly, then decreased gradually, and they presented a small peak at the depth

of 23 cm (1944), and then decreased a little, then increased to the peak value at the depth of 11 cm (1986). The Cr concentration at the depth of 11 cm was 61.70 mg/kg, and the Cr concentrations at the other depths were not higher than the background of the Bohai Bay sediment (60 mg/kg) (Chen, 1992). There is no clear increase in the surface layers, which indicates that Cr might be not enriched.

Table 2 Specific activity layer and dating year of ^{137}Cs and $^{210}\text{Pb}_{\text{ex}}$ at Station 5

Depth (cm)	^{137}Cs specific activity (Bq/kg)	Dating (yr)	$^{210}\text{Pb}_{\text{ex}}$ specific activity (Bq/kg)	Dating (yr)
15	8.637	1963	81.146	1973
23	4.596	1954	32.849	1944

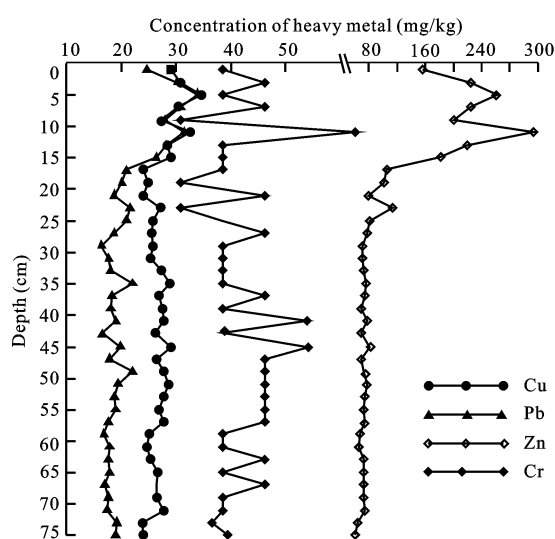


Fig. 4 Concentrations of heavy metals in core samples at Station 5

Huludao Zinc Plant put into production in November 1942. The results of this study showed that the Zn concentration in this core sample at Station 5 increased obviously in 1944, which coincided with the time that zinc system started production in the Huludao Zinc Plant. The plant was all affected by the war during 1945–1948, which was corresponding to the little decrease of heavy metals concentrations. In the 1980s, with the rapid economic development of China, the output of large state-owned enterprises like the Huludao Zinc Plant in Huludao City increased successively. In this study, the Zn concentration reached the peak value in 1986, which was directly related to the rapid increase of zinc output at this period. During the period of 1996–2000, 1.157×10^{10} yuan (RMB) was invested to prevent intensive land-source pollution in Huludao City. The work on

pollution prevention and control achieved significant results. From Fig. 4, it could be seen that the concentrations of heavy metals in core sample decreased gradually from the depth of 11 cm to the surface layer. The accumulation pattern of the Zn, Cu and Pb was corresponding closely with the production of the Huludao Zinc Plant.

3.2.2 Sedimentation rate and accumulation characteristics of heavy metals at Station 28

At the Station 28, little change of radionuclide $^{210}\text{Pb}_{\text{ex}}$ from 0 cm to 7 cm and lower values than underlying sediments were found (Fig. 5). Below 7 cm, the concentrations of radioactive nuclide $^{210}\text{Pb}_{\text{ex}}$ were normal, which accorded with radioactive decay process. The concentrations of radionuclide ^{137}Cs showed a low-high-low pattern from the surface to the bottom, which conformed to human nuclear test in the atmosphere (Fig. 5). According to the curve of $^{210}\text{Pb}_{\text{ex}}$, the surface sediment samples (0–7 cm) might be disturbed by external sediment. The dredging sediment of neighboring channel according to on-site investigation might be dumped at the Station 28 before sampling.

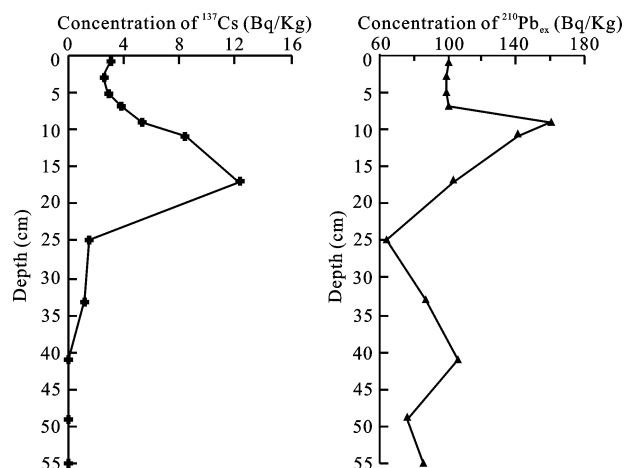


Fig. 5 Concentrations of radionuclide ^{137}Cs and $^{210}\text{Pb}_{\text{ex}}$ in core samples at Station 28

According to the points from 5 to 8 at Station 28, we adopted CIC mode to date using $^{210}\text{Pb}_{\text{ex}}$. The fitted curve was $y = 264.66e^{-0.0561x}$ ($R^2 = 0.99$) between the depth and $^{210}\text{Pb}_{\text{ex}}$ activity, so the average sediment deposition rate was 0.55 cm/yr. The sedimentary era of the core was listed in Table 3.

There were similar vertical distribution pattern for the heavy metals between the Station 5 and the Station 28, although different deposition rates exist (Fig. 6). The concentrations of Zn increased obviously from the depth

Table 3 Specific activity layer and dating year of ^{137}Cs and $^{210}\text{Pb}_{\text{ex}}$ at Station 28

Depth (cm)	^{137}Cs specific activity (Bq/kg)	Dating (yr)	$^{210}\text{Pb}_{\text{ex}}$ specific activity (Bq/kg)	Dating (yr)
17	12.384	1963	103.705	1979
33	1.265	1954	86.422	1949

of 23 cm, reaching peak value (132.0 mg/kg) at 15 cm depth, and then gradually decreased. The accumulation patterns of Pb, Cu and Cr were similar to Zn, especially from 25 cm to the surface. Compared with the Station 5, the concentrations of heavy metals were lower at the Station 28 for the farther distance to the source. At most of the depths, the concentrations of Cu, Pb, Zn and Cr in the sediments met the first class standard of Marine Sediment Quality (GB 18668-2002), and the Cr concentration was lower than its background value of the Bohai Bay (60 mg/kg), but there was an increasing trend with the depth which is probably related to the accelerated modern deposition rate of the Bohai Sea (Wang *et al.*, 2003), high deposition rate causes coarsen particulate matter to increase, and absorption capacity to heavy metals to decline (Kang *et al.*, 2003). Because of the different existing state of the heavy metals, the variation trend of Cu was not similar to Cr, and the state of Cr mainly was residual, the 'particle effect' was more significant than other metals (Jin and Wang, 1984; Dltoro *et al.*, 1986), and Cu mainly existed in organic-bound, carbonate salt and Fe-Mn oxides-bound forms (Yang *et al.*, 2007).

The long sampling interval for determining radionuclide caused the inaccuracy of dating, however, accord-

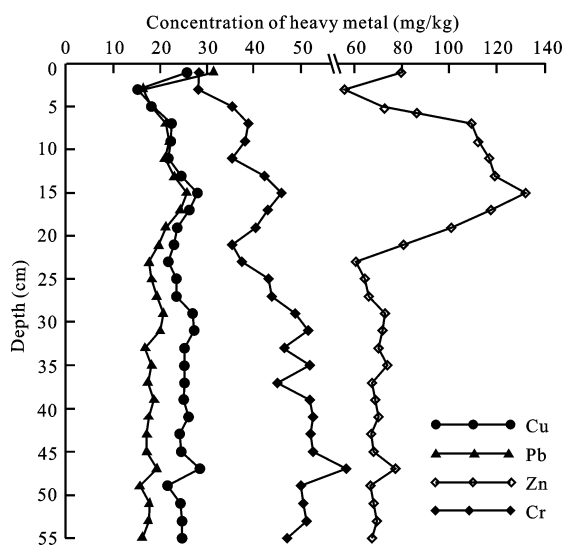
ing to vertical distribution of the heavy metals in the two core samples, heavy metals concentrations, especially Zn, all increased and then decreased. The Station 5 is closer to the coast and pollution source compared with the Station 28, so the starting time of pollution increase for the Station 5 was earlier, which also reflected indirectly that the pollution sources came mainly from land-sourced input.

At the Station 28, the concentrations of Zn, Cu, Pb and Cr in the surface sediments were 79.6 mg/kg, 25.7 mg/kg, 31.4 mg/kg and 28.6 mg/kg, respectively (Fig. 6), and the concentrations of Cu, Pb and Zn were higher slightly than those in the underlying sediments, while the concentrations of Cr was almost the same as that in the underlying sediments. From the vertical distribution pattern of radionuclide concentrations, external older sediment deposition at the Station 28 caused high concentrations in the surface sediments. The high concentrations of heavy metals at the two core sediments all appeared at the upper 20 cm. The sediment layer with high heavy metals concentrations could be exposed to the surface by ocean engineering and big storm surge, so the risk to the safety of aquaculture still exist.

4 Conclusions

The southern sea area of Huludao City might be polluted by heavy metals because it is close to the Jinzhou Bay, one of the sea area seriously polluted by heavy metals in China, and the soil in Huludao City is also polluted by heavy metals came from the Huludao Zinc Plant. The heavy metals are determined in surface and core sediments to study the distribution of heavy metals, the accumulation and source of the heavy metals are studied by using isotope analysis of ^{137}Cs and $^{210}\text{Pb}_{\text{ex}}$.

The concentration of heavy metals increases from east to west, as well as from open sea to offshore marine area. There is moderate or heavy ecological risk in the offshore area. The concentrations of heavy metals are low in the sediments adjacent to the Jinzhou Bay, and the influence caused by the seawater exchange with the Jinzhou Bay is little. The atmospheric deposition of the heavy metals from the Huludao Zinc Plant is likely to be the main source of pollution. The heavy metals deposited to the soil in the past could be carried to the sea by runoff, although the emission of pollutant by the Huludao Zinc Plant is cut down.

**Fig. 6** Concentrations of heavy metals in core samples at Station 28

The heavy metal concentrations in the two core sediments show the distribution pattern of low-high-low from the surface to the bottom which coincides with the history of the emission and treatment of pollution of the Huludao Zinc Plant. The pollution control measures had caused the decrease of heavy metal concentrations in the surface sediment. The high heavy metal concentrations of the two core samples all appear in the upper 20 cm of the sediments, so the risks of heavy metals in the sediment to the safety of aquaculture still exist.

To determine the characteristics of heavy metals in the sediment will be helpful to assess the ecological risk to aquatic ecosystem. The research on the heavy metals in runoff coming from the Huludao Peninsula will be useful to estimate the flux to the sea and to check the source of metals in future.

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