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# Mercury contamination due to zinc smelting and chlor-alkali production in NE China

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ABSTRACT

Zinc smelting and chlor-alkali production are major sources of Hg contamination to the environment, potentially leading to serious impacts on the health of the local population. Huludao, NE China has been heavily contaminated by Hg due to long-term Zn smelting and chlor-alkali production. The aim of this work was to determine Hg accumulation in the aquatic and terrestrial environment, as well as in the human population of Huludao. The investigation included: (a) Hg accumulation in sediments, *Spirogyra* algae, crucian carp and shrimp, (b) Hg distribution in soil, vegetables and corn, and (c) assessment of potential health effects of Hg exposure associated with total Hg (T-Hg) concentrations in human hair. Measured T-Hg concentrations in sediments of Wuli River ranged from 0.15 to 15.4 mg $kg^{-1}$, with the maximum Hg concentration in sediment exceeding the background levels in Liaoning Province by 438 times. The maximum T-Hg levels in *Spirogyra*, crucian carp and shrimp were 13.6, 0.36, and 0.44 mg $kg^{-1}$, respectively. Total-Hg concentrations in hair of the human population varied from 0.05 to 3.25 mg $kg^{-1}$ (average 0.43 mg $kg^{-1}$). However, the frequency of paraesthesia to most inhabitants in Huludao was estimated to be lower than 5%, with only one person rated at 50%. The results indicated minimal adverse health effects of Hg exposure to the inhabitants of Huludao, despite the serious Hg contamination of the environment.



## 1. Introduction

Mercury is one of the most harmful environmental pollutants because of its high toxicity, ready uptake of organic forms of Hg by biota, and accumulation in the food chain (Li et al., 2008). High levels of Hg in the bloodstream may harm the developing central nervous systems of unborn babies and young children, ultimately making the child less able to think and learn (Obiri et al., 2010). Therefore, Hg contamination seriously threatens the health of ecosystems and human populations.

Zinc smelting is one of the most important anthropogenic Hg emission sources (Nriagu and Pacyna, 1988). During the Zn smelting process, almost all the Hg in the Zn ores is volatilized from the matrix, so that it is eventually emitted to the atmosphere, unless pollution control technology is applied (Pacyna and Pacyna, 2002). Chlor-alkali production is another important source of anthropogenic Hg (Gibičar et al., 2009). Although the Hg cell chlor-alkali (MCCA) technique has been replaced by alternative techniques in many locations, historical accumulation of Hg could still be a secondary contamination source to the environment (Olivero-Verbel et al., 2008).

Huludao is an industrial base in Liaoning Province, because of the Huludao Zn Plant (HZP). The plant is situated in the SE of the city of Huludao, which lies on the west coast of the Liaodong Gulf in Liaoning Province, China. Mercury has been emitted into the environment in large quantities through atmospheric deposition during Zn smelting since HZP was built in 1937. About 51 tons of Hg was emitted into the atmosphere from HZP from 1983 to 1989 (Zheng et al., 2007). Chlor-alkali production using a Hg cathode, was carried out in the Jinxi Petroleum Chemical Factory (JPCF) in Huludao, until 1998. The Wuli River flows through Huludao, and reaches the sea in the Jinzhou Gulf. About 265 tons Hg was discharged into Wuli River from 1952 to 1980, which led to 90 tons of Hg accumulating on the riverbed of Wuli River (Zhao and Yan, 1997). The ecosystems of Huludao are being impacted significantly by both historical and contemporary anthropogenic loadings of Hg (Zheng et al., 2008b). The objective of the present study was to investigate Hg accumulation in the environment of Huludao due to the combined contamination from Zn smelting and chlor-alkali production.

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## 2. Methods

### 2.1. Study area

Huludao (40°56′N, 120°28′E) is located in Liaoning Province, NE China (Fig. 1). The primary wind direction is from NE to SW. The industrial district spans an area of over 180 $km^2$ with an urban population of approximately 200,000 in 2007.

The Wuli River flows through Huludao, and reaches the sea in the Jinzhou Gulf. JPCF was located upstream of Wuli River, and the sewage outlet of JPCF was located between Sites W3 and W4 (Fig. 1). HZP is situated at the estuary of Wuli River. As a result, all Hg discharged from HZP and JPCF was into the Wuli River. The Lianshan District (LSD), Daochi District (DCD), Economic Developing District (EDD), New District (ND), Zn Plant District (ZPD), and Wanghaisi District (WHSD) represent different sections of the city. Most urban inhabitants in Huludao are concentrated in LSD, ND, ZPD and WHSD, and some villagers live in DCD. The main business centers and hospitals are distributed in LSD, EDD and ND, which have developed recently.

### 2.2. Sample collection and preparation

Samples of sediment, *Spirogyra* (*Spirogyra communis* (Hass.) Kutz.), shrimp (*Macrobranchium nipponense*), crucian (*Carassius auratus*), vegetables, corn and hair were collected from May 2006 to October 2008.

Approximately 300–400 g sediment was collected at various locations (Fig. 1) using a stainless steel shovel. About 100 g of *Spirogyra* algae were sampled at W2, W3, W4, W5, W7 and W8. Around 100 g shrimps and 100 g crucians were collected at W2, W3, W5, their lengths and weights were recorded.

Six types of vegetables were collected, 2 cereals and the corresponding soil samples at W6, W10, V1, V2 and V3. The amount of each product was chosen according to its fresh weight (FW) to dry weight ratio, in order to obtain approximately 20–30 g sample dry weight. The total number of vegetable and cereal samples was 124. The fresh vegetable samples were put in clean plastic bags and transported to the laboratory for sample treatment as soon as possible. After being cleaned with Mili-Q water, the fresh weights of the samples were recorded.

A total of 140 hair samples were collected from volunteers with ages from 3 to 75 a (76 males and 64 females) representing the population who have not been subject to occupational exposure to Hg in Huludao. Of these, 31, 29, 26, 26 and 28 inhabitants were selected from LSD, XD, DCD, ZPD and WHSD, respectively (Fig. 1). Hair samples (ranging from 100 to 300 mg) were collected near the scalp, in order to reduce contributions of exogenous contamination. Meanwhile, a questionnaire was collected from the participants designed to obtain information about age, sex, food consumption (home grown fruits and vegetables, fish and game animals), source of consumed water, smoking habits, beauty salon habits including hairdressing, alcohol consumption, health condition and medication, and work place. The samples were cut with clean stainless steel scissors and sealed in polyethylene bags before analysis.

### 2.3. Analytical procedure and methods

The hair samples were cleaned with mild detergent and water and rinsed three times with distilled water. The cleaned hair samples were air-dried and cut into 2-mm-long segments with clean stainless steel scissors. Dry subsamples (0.1 g) were digested with 4 mL $HNO_3$ (65%) and 2.5 mL (98%) $H_2SO_4$ ($H_2SO_4$–$HNO_3$–$V_2O_5$, GB/T 17136-1997) and were heated on a sand bath.

Sediments, *Spirogyra*, shrimp, crucian, vegetables, and corn samples were digested using $H_2SO_4$–$HNO_3$–$V_2O_5$ (GB/T 17136-1997). Total organic C in *Spirogyra* was determined by a combustion oxidation-non dispersive infrared absorption method (HJ/T 71-2001). An ultra-trace Hg analysis system (Tekran Series 2600, Canada) was used to determine the concentration of total Hg in samples.

### 2.4. Quality control

The average T-Hg concentrations of Standard Reference Materials of sediments (GBW 07304), of poplar leaves (GBW 07604) and of hair (GBW 07601) were 0.042 ± 0.01 mg $kg^{-1}$ ($n$ = 8), 0.027 ± 0.005 mg $kg^{-1}$ ($n$ = 8) and 0.38 ± 0.08 mg $kg^{-1}$ ($n$ = 8), which were comparable with the certified concentrations of 0.044 ± 0.008 mg $kg^{-1}$, 0.026 ± 0.003 mg $kg^{-1}$ and 0.36 ± 0.08 mg $kg^{-1}$, respectively. The percentage of recoveries on spiked samples ranged from 87% to 104% for T-Hg. Reagent blanks and internal standards were used wherever appropriate to ensure accuracy and precision in metal analyses.

## 3. Results and discussion

### 3.1. Mercury in the aquatic system

#### 3.1.1. Mercury in sediments

Total-Hg concentrations in the sediments of Wuli River ranged from 0.15 to 15.4 mg $kg^{-1}$, and the maximum Hg concentration

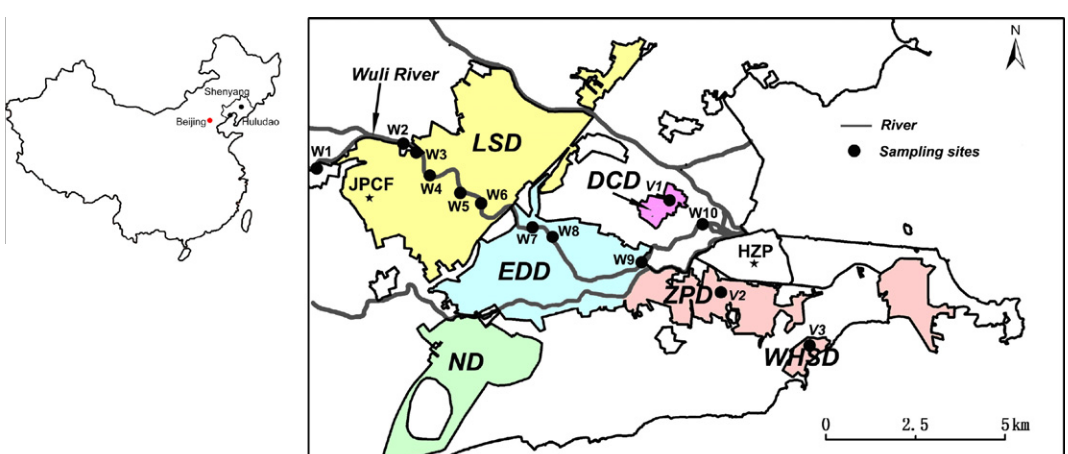


**Fig. 1.** The locations of the study area and sampling sites including Huludao Zn Plant (HZP), Jinxi Petroleum Chemical Factory (JPCF), Lianshan District (LSD), Daochi District (DCD), Economic Developing District (EDD), New District (ND), Zn Plant District (ZPD), and Wanghaisi District (WHSD).

occurred at sampling site W7, exceeding the background value in the sediment of Liaoning Province by 438 times (Fig. 2; Sun, 1992; Zheng et al., 2008b). Wuli River has suffered a long history of Hg contamination from JPCF, and has been contaminated by Hg derived from chlor-alkali production prior to 1998, when the use of Hg cathodes ceased. The sewage outlet of JPCF was located between Sites W3 and W4. Total-Hg levels in the sediments from Site W4 to W10 were higher than those from Sites W1 to W4. There is a waste water plant close to W10, and Zn smelting from HZP might also affect Hg levels in Site W10 due to the tide from Wuli Estuary (Wang et al., 2009; Zheng et al., 2008b). Mercury concentrations in the sediments were lower than other highly-impacted ecosystems, but they were similar to concentrations in the Hai River (Shi et al., 2005). Studies have indicated that Hg contamination from chlor-alkali production had been reduced since the termination of the use of Hg cathodes (Zhang et al., 2008) (see Table 1).

### 3.1.2. Mercury in Spirogyra, Crucian and Shrimp

For the *Spirogyra* samples, T-Hg concentrations varied from 0.28 to 13.6 mg $kg^{-1}$ (dry weight), and the maximal concentration was observed at W7 (Table 2). No *Spirogyra* were present in the W9 and W10 sites. Total-Hg contents in *Spirogyra* increased along Wuli River, but TOC in *Spirogyra* decreased. A significant correlation was observed between T-Hg concentrations in the sediments and in *Spirogyra* ($r = 0.839$, $p < 0.05$).

Total-Hg concentrations in the Crucian of Wuli River varied from 0.03 to 0.36 mg $kg^{-1}$, which were higher than other fish obtained from the markets in Huludao (Zheng et al., 2007a,b,c). Moreover, only 3 and 4 Crucian samples were collected at the W2 and W3 sites, respectively, and only 1 Crucian sample was obtained at the W5 site. Total-Hg in Crucian at the W5 site was higher than at site W2 and W3. Total-Hg concentrations in the shrimps of Wuli River varied from 0.06 to 0.44 mg $kg^{-1}$, and 28 and 43 shrimps were found in the sediment at the W2 and W5 sites, respectively. Total-Hg concentrations in shrimp at W5 were the highest in the study, similar to T-Hg concentrations in crucian. In Wuli River, no crucian and shrimp samples were collected except at W2, W3 and W5. Clearly, Hg contamination due to Zn smelting and chlor-alkali production has affected the aquatic environment of Wuli River, especially in the downstream reaches. Acute toxicity occurs in the sediments at sites where the Wuli River flows into the sea (Fan et al., 2006). The highest acute mortality, 100%, occurred in the amphipod species *Ampetisca abdita* at the mouth of Wuli River and Cishan River, where a 10-d flow-through sediment acute toxicity test was conducted (Yan et al., 1999).

### 3.1.3. Mercury in the river bank

Mercury contamination in the sediments not only affected the aquatic environment, but also threatened plant and animal species along the banks of the Wuli River. Zheng et al. (2007) reported that T-Hg levels in herb leaves along the Wuli River decreased in the following order: *Artemisia verbenacea* > *Polygonum hydropiper* > *Phragmites australis* > *Datura stramonium* > *Scirpus planiculmis Fr Schmidt* > *Xanthium sibiricum Patr.* > *Echinochloa Crusgalli.* Total-Hg concentrations in the stems and leaves of these species varied from 0.003 to 3.02 mg $kg^{-1}$ and the corresponding T-Hg concentrations in soil ranged from 0.51 to 17.6 mg $kg^{-1}$. Zheng et al. (2008a) reported that T-Hg levels in *Locusta migratoria manilensis* and *Acrida chinensis* from Wuli River banks were 0.013–0.154 and 0.009–0.138 mg $kg^{-1}$, respectively, while those of methylmercury were 0.001–0.012 and 0.001–0.006 mg $kg^{-1}$.

## 3.2. Mercury in the soil compartment

### 3.2.1. Mercury in surface soil

Previous research has demonstrated the presence of Hg contamination in soil due to Zn smelting and chlor-alkali production. Total-Hg concentrations in surface soil had a range of 0.05–14.6 mg $kg^{-1}$, and T-Hg concentrations in surface soil decreased radically from the centers around the factories for Zn smelting and chlor-alkai production. Mercury emissions from HZP and chlor-alkali industry are the main pollution sources of Hg to the soil (Zheng et al., 2007).

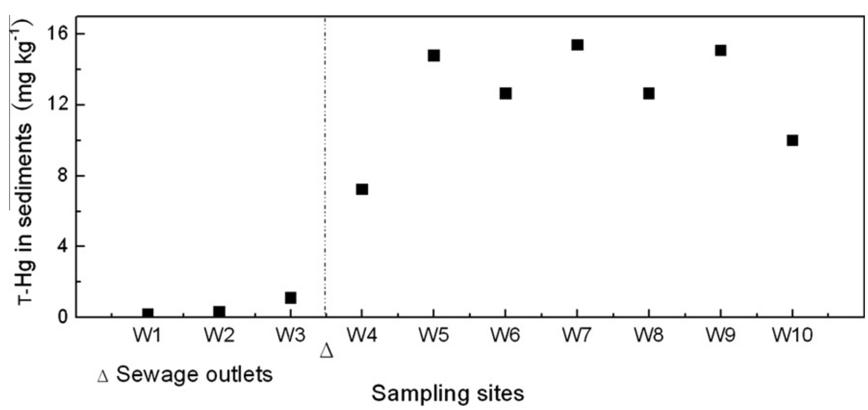


**Fig. 2.** T-Hg distribution in the sediments at each sampling sites along the Wuli River, NE China.

**Table 1**
Comparison of T-Hg concentrations in sediment in this study with other investigations worldwide.

| Aquatic environment | Hg source | Sediments (mg $kg^{-1}$) | References |
|---|---|---|---|
| Wuli River | Chlor-alkali Industry and Zn smelting | 0.15–15.4 | This study |
| Small streams, Wanshan, China | Hg mining activity | 90–930 | Qiu et al. (2005) |
| Idrijca River, Slovenia | Hg mining activity and Zn smelting | 5–1000 | Gosar et al. (1997) |
| Thur River, France | Chlor-alkali Industry | 5–500 | Hissier and Probst (2006) |
| Lake Balkyldak, Kazakhstan | Chlor-alkali Industry | 1500 | Ullrich et al. (2007) |
| Hai River, China | Industrial, domestic and agricultural effluents | 8.78 | Shi et al. (2005) |

**Table 2**
T-Hg contents in spirogyra, crucian and shrimp at sites of Wuli River.

| Sampling sites | *Spirogyra* | | Crucian | | Shrimp | |
|---|---|---|---|---|---|---|
| | T-Hg (mg kg$^{-1}$) | TOC | T-Hg (mg kg$^{-1}$) | Length (cm)/weight(g) | T-Hg (mg kg$^{-1}$) | Length (cm)/weight(g) |
| W2 | 0.28 | 21.3 | 0.03 | 8.01–8.54/4.87–5.32 | | |
| W3 | 1.85 | 35.0 | 0.06 | 7.11–7.25/4.37–5.74 | 0.07 | 1.10–4.70/0.13–0.68 |
| W4 | 4.76 | 31.9 | | | | |
| W5 | 5.74 | 32.8 | 0.36 | 6.5/4.12 | 0.44 | 0.90–2.61/0.14–0.41 |
| W7 | 13.6 | 27.7 | | | | |
| W8 | 13.1 | 25.5 | | | | |

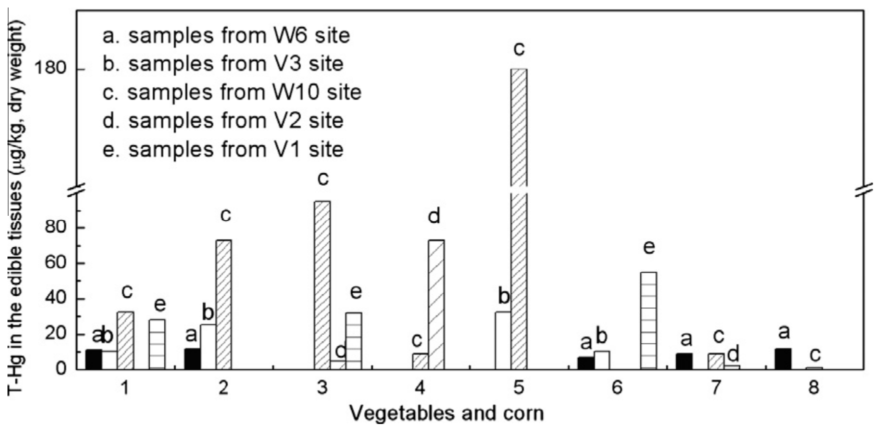


**Fig. 3.** T-Hg distribution in the edible tissues of vegetables and corn at different sites (1: Cowpea ($n$ = 17); 2: Chinese cabbage ($n$ = 35); 3: capsicum ($n$ = 12); 4: tomato ($n$ = 11); 5: leek ($n$ = 15); 6: carrot ($n$ = 11); 7: bean ($n$ = 12); 8: maize ($n$ = 11)).

*3.2.2. Mercury in soil-vegetables and corn*

The source of Hg in soil at the W6 site is mainly chlor-alkali production, and the Hg in soil at the W10 site is mainly derived from Hg deposition due to Zn smelting, chlor-alkali production and the waste water plant, while the Hg source in soil at the V1, V2 and V3 sites is mainly from deposition due to Zn smelting. Total-Hg concentrations in vegetables and corn samples increased in the order of W6, V3 sites < V1, V2 sites < W10 site (Fig. 3). Mercury deposition was the most important source of Hg to vegetables and corn. Zheng et al. (2007b) reported that Hg and Pb contents in leaves of vegetables around HZP are higher than those in roots, indicating that Hg and Pb in leaves of vegetables were mainly derived from atmospheric sources, most likely from dust particles. Therefore, Hg emission from HZP during Zn smelting is believed to be the main Hg source to vegetables and corn.

*3.2.3. Mercury in soil–plant–insect*

Zheng et al. (2007) reported T-Hg contents of 154 samples of woody plants from 13 different sites in Huludao, with Hg contents in the leaves of these plants ranging from 0.10 to 2.70 mg kg$^{-1}$. Zhang et al. (2008, 2009) reported that concentration factors of each trophic level of the soil–plant–herbivorous insect–carnivorous insect food chain were 0.18, 6.57, and 7.88 for Hg, respectively. On the whole, Hg was the most largely biomagnified

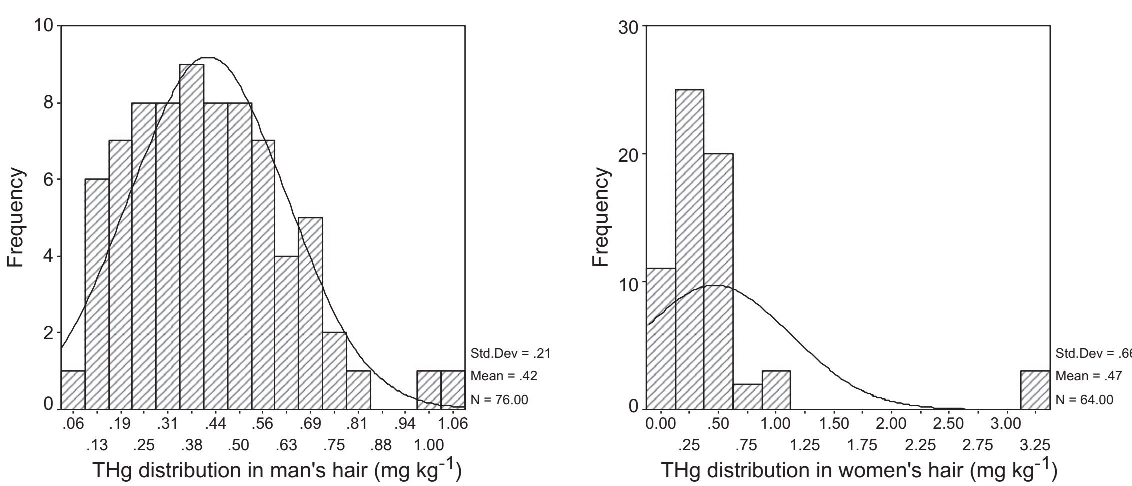


**Fig. 4.** Statistical frequencies of T-Hg in hair, Huladao City, NE China.

**Table 3**
T-Hg level of hair in different age, gender, sites (mg kg$^{-1}$).

| Factor | | n | Average | Range | S.D. |
|---|---|---|---|---|---|
| Age | <15 | 21 | 0.43 | 0.18–0.71 | 0.17 |
| | 16–25 | 34 | 0.33 | 0.07–1.05 | 0.23 |
| | 26–45 | 48 | 0.51 | 0.04–3.25 | 0.51 |
| | >45 | 37 | 0.39 | 0.09–0.68 | 0.17 |
| Sites | LSD | 31 | 0.37 | 0.11–0.63 | 0.16 |
| | XD | 29 | 0.34 | 0.24–1.01 | 0.29 |
| | DCD | 26 | 0.42 | 0.11–0.71 | 0.18 |
| | ZPD | 26 | 0.38 | 0.07–0.73 | 0.21 |
| | WHSD | 28 | 0.50 | 0.05–3.25 | 0.66 |

S.D.: standard deviation.

**Table 4**
Classification by hair Hg concentrations in paraesthesia.*

| T-Hg contents in hair (mg kg$^{-1}$) | Frequency of paraesthesia (%) | Number | Percentage (%) |
|---|---|---|---|
| <0.64 | <5 | 123 | 87.2 |
| 0.64–1.5 | 5 | 16 | 11.8 |
| 1.5–2.8 | 25 | 0 | 0 |
| 2.8–5.9 | 50 | 1 | 1 |
| 5.9–10.0 | 75 | 0 | 0 |
| >10.0 | 95 | 0 | 0 |

* Li (2003).

potentially harmful metal, while Cd and Pb were not as greatly accumulated in carnivorous insects as expected.

### 3.3. Mercury exposure in the general population

#### 3.3.1. Mercury in human hair

Mercury concentration in human hair has been widely used as a biomarker for human Hg exposure to reflect a steady state body burden. Compared with blood, hair is readily obtained and stored. Therefore, hair was selected to estimate Hg exposure in Huludao. Fig. 4 shows T-Hg concentrations in hair of the Huludao population, the values varying from 0.05 to 3.25 mg kg$^{-1}$. Age is an important factor influencing T-Hg concentrations in hair, although no significant correlation between age and T-Hg in hair ($r = 0.024$, $p > 0.05$) was observed. The highest mean value for T-Hg concentration was 0.51 mg kg$^{-1}$, and occurred in the 26–45 age group. The lowest values were 0.33 and 0.39 mg kg$^{-1}$ in the16–25 and >45 age groups, respectively. Total-Hg levels in females were higher than in males (Table 3). Total-Hg contents in hair decreased in the order of WHSD > DCD > ZPD > LSD, ND. However, there was no significant difference between T-Hg in hair from different districts based on a *T* test ($P > 0.05$).

#### 3.3.2. Mercury exposure

Paraesthesia is caused by Hg poisoning and has symptom in adults, even in subgroups suffering from low Hg exposure (Li, 2003). Based on their research work, USEPA classified hair Hg concentrations into groups associated with the symptoms of paraesthesia and adverse effects on the central nervous system. Considering the T-Hg level in hair of inhabitants in Huludao, the symptoms of paraesthesia were used to judge the health levels (Table 4). The results indicate that the frequency of paraesthesia to most inhabitants in Huludao is lower than 5%, with only one person from the selected group showing a frequency of 50%. Moreover, T-Hg contents in hair of Huludao inhabitants are lower than those of other cities or countries (Table 5). Therefore, there is a negligible health effect on Huludao inhabitants due to Hg exposure.

In general, food consumption was identified as the major pathway of human exposure, accounting for >90% compared to other exposure pathways such as inhalation and dermal contact (Loutfy et al., 2006; Feng et al., 2008). There is no significant potential health risk for adults and children in Huludao from the dietary Hg intake. The dietary intake of Hg was estimated to be $2.135 \times 10^{-3}$ mg d$^{-1}$ (Zheng et al., 2007a,b,c), which is less than the tolerable daily intake limit. Therefore, although the environment of Huludao City is seriously contaminated with Hg according to T-Hg levels in sediment, soil, *Spirogyra*, crucian and shrimp, the health effect of Hg exposure to local inhabitants in Huludao is not serious.

## 4. Conclusions

Mercury contamination derived from chlor-alkali production has affected the ecosystem of the Wuli River. Mercury deposition derived from HZP is an important source of Hg in soil, vegetables and corn. There is a relatively trivial health risk to the local population based on the assessment of hair Hg. Further work is needed not only to investigate T-Hg in blood and urine in order to affirm the health effect, but also to investigate Hg exposure in relation to dietary habits and focus on susceptible subgroups. Though there is little health effect of Hg based on the T-Hg levels in hair, Hg contamination in the estuary of the Wuli River and Bohai gulf definitely pose a health risk to fisherman. The ecological and health implications for Hg exposure in the residential environment of the smelting district needs further detailed investigation.

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**Table 5**
Comparison of T-Hg concentrations in hair in this study with other investigations worldwide.

| Sites | Type of residents | Hg source | T-Hg in hair (mg kg$^{-1}$) | References |
|---|---|---|---|---|
| Huludao city | Urban residents | Chlor-alkali Industry and Zn smelting | 0.43 (0.05–3.25)* | This study |
| Changchun city | Urban residents | Domestic effluents | 0.43 (0.05–10.46) | Li et al. (2006) |
| Di'er Songhua River | Fishermen | Industrial wastewater | 3.41(0.16–199) | Zhang and Wang (2006) |
| Wanshan, China | Urban residents | Hg mine | (0.32–58.50) | Feng et al. (2008) |
| Cambodia | Urban residents | Industrial wastes | 7.30 (0.54–190) | Agusa et al. (2005) |
| Apokon, Philippines | School children | Gold processing and refining plant | 0.99 (0.28–20.39) | Akagi et al. (2003) |
| Colombia | Urban residents | Abandoned chlor-alkali plant | 1.52 (0.10–21.80) | Olivero-Verbel et al. (2008) |

* Average (min–max).

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