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Occurrence, speciation and bioaccessibility of lead in Chinese rural household dust and the associated health risk to children

Zhixuan Han [a,b], Xiangyang Bi [a,b,*], Zhonggen Li [c], Wenlin Yang [b], Lixing Wang [b], Huan Yang [b], Fanglin Li [b], Zhendong Ma [b]

[a] State Key Laboratory of Biogeology and Environmental Geology, China University of Geosciences, Wuhan 430074, China
[b] Faculty of Earth Science, China University of Geosciences, Wuhan 430074, China
[c] State Key Laboratory of Environmental Geochemistry, Institute of Geochemistry, Chinese Academy of Sciences, Guiyang 550002, China

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ABSTRACT

Lead (Pb) concentration, speciation and bioaccessibility were measured in 122 household dust samples collected in rural areas of eight provinces of China. The mean Pb concentration in the household dust was 208 mg kg^{-1}, of which samples from sites in Hunan (538 mg kg^{-1}) and Yunnan (280 mg kg^{-1}) provinces exhibited the highest Pb concentrations while those from Shaanxi (96 mg kg^{-1}) and Fujian (80 mg kg^{-1}) provinces had a relatively low Pb content. The major fraction of Pb in the household dust samples was found to be strongly bound to Fe–Mn oxide phases (37%) while Pb present in minor fractions individually making up between 14 and 18% was characterized in falling orders as residual, carbonate, organic/sulphide, and exchangeable fractions by the sequential extraction method applied. Bioaccessible Pb making up an average proportion of 53% in the household dusts was significantly correlated to the Fe–Mn oxide phases of Pb. According to the Hazard Quotient (HQ), the ingestion of dust particles pose the highest risk to children in Chinese rural areas, followed by dermal contact and inhalation. Hazard Index (HI) values for most samples were lower than 1, indicating that the domestic Pb exposure in rural areas of China were relatively safe for children when they exposure to the household dust. However, dust Pb in 4.1% of the studied families having HI values higher than 1 may pose adverse health effect to the children.

1. Introduction

Lead (Pb) has received particular attention in recent decades for its ubiquitous pollution from the use of lead in gasoline and paints and the improperly controlled non-ferrous metallurgy. Exposure of Pb can result in serious toxic reactions in the infant human body, influencing children's nervous system and reducing children's intelligence. The World Health Organization (WHO) found a linear relationship of 1.3 IQ points lost per 5 μg dl^{-1} of blood lead between 5–20 μg dl^{-1} (WHO, 2003). At present, the blood lead levels (BLLs) of children from developed countries have been reduced. BLLs of American children (<6 years) reduced from 14.1∼15.8 μg dl^{-1} in 1970s to 2.0∼2.5 μg dl^{-1} in 2000 (CDC, 2004). In contrast, in developing countries Pb pollution situation is often more serious. In China, He et al. (2009) observed that more than 20% of children's BLLs were higher than 10 μg dl^{-1}, in addition the mean BLLs of children living in suburban and rural areas were higher than in urban areas.

The WHO estimated that the sources of Pb exposure in children are dust and soil (45%), food (47%), water (6%) and air (1%), respectively. Dietary Pb exposure has gradually decreased with increasing of food security and effective control measurements, and direct exposure to dust has become more dominant, especially for children with hand-to-mouth activity. The USEPA (1994) reported that the average dust intake of children through ingestion was 135 mg day^{-1}. In some cases, dust and soil intake via ingestion by a child may be even as high as of 60 g day^{-1} (Van-Wijnen et al., 1990; Calabrese et al., 1999). Many studies indicated that there is a significant correlation between BLLs and Pb concentration in dust/soil (Laidlaw et al., 2005; Ren et al., 2006; Laidlaw and Taylor, 2011). Pb exposure via hand-to-mouth actions may be higher for the children in Chinese rural areas, given that they may spend more time on the ground. However, there is little information regarding Pb contamination in Chinese rural areas. Consequently, it is important to study the rural lead contamination and find solution to minimize its severity.

* Corresponding author. State Key Laboratory of Biogeology and Environmental Geology, China University of Geosciences, Wuhan 430074, China. Tel.: +86 27 67883001; fax: +86 27 67883002.
E-mail address: bixy@cug.edu.cn (X. Bi).

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Therefore, in order to gather information and initial survey the contamination levels of lead in Chinese rural areas, the primary objectives of the present study were: 1) to investigate the concentration, speciation and bioaccessibility of Pb in household dust from Chinese rural areas; and 2) to assess health risk of exposure to Pb via household dust for Chinese rural children.

2. Materials and methods

2.1. Sample collection

Sampling was conducted in August 2009. 122 household dust samples were randomly collected in rural Chinese areas of 8 provinces, including Pingyi County in Shandong Province, Ezhou city in Hubei Province, Lianyuan City in Hunan Province, Zhangzhou City in Fujian Province, Hanyin County in Shaanxi Province, Minhe County in Qinghai Province, Yuxi City in Yunnan Province and Jarud Banner in Inner Mongolia. Dust samples were collected inside from the floor using a brush and plastic spatula, stored in polyethylene bags, then transported to the laboratory.

2.2. Sample analytical procedure

After removal of pebbles, plants, hair and other large impurities, the samples were air-dried at room temperature and then sieved to a mesh <250 μm. About 250 mg of dust sample was digested with US-EPA Method-3050B for measuring total Pb. A sequential extraction procedure was employed to investigate the chemical speciation of Pb, which contained the following five phases (Tessier et al., 1979). F1: exchangeable phase (extracted by $MgCl_2$), F2: carbonate phase (extracted by NaOAc), F3: Fe–Mn oxide phases (extracted by $NH_2OH\cdot HCl$), F4: organic/sulphide phase (extracted by H_2O_2 and HNO_3) and F5: residual fraction (digested by HCl and HNO_3).

The bioaccessibility of Pb was determined with a fast *in vitro* method described by Mercier et al. (2002):

- Add 1.0 g dust sample to polyethylene centrifugal tube, then add 20 ml extracting solution which mixed with 6 ml glacial acetic acid and 8L distilled water and seal up the tube.
- Heat it at 37 °C in water-bath pot for 20 min, adjust pH = 6 with HCl; agitate for 20 min.
- Heat it at 37 °C in water-bath pot for 20 min, adjust pH = 4 with HCl; agitate for 20 min.
- Heat it at 37 °C in water-bath pot for 20 min, adjust pH = 2.5 with HCl; agitate for 20 min.
- Heat it at 37 °C in water-bath pot for 20 min, adjust pH = 2 with HCl; agitate for 20 min.

At the end of the extraction, the samples were left to settle for 5 min and then centrifuged at 3500 rpm for 10 min. The supernatant was subsequently filtered and acidified to a pH of less than 1 with concentrated HNO_3. Atomic absorption spectrophotometry (AAS) was employed to determine Pb concentration. The quality of the tests was controlled by national standard reference materials (GSS-1) and repeated samples. The accuracy of the sequential extraction procedure was verified by comparison of the difference between the sum of each phase concentration and the total digested concentration. The recovery ((species sum/total Pb) × 100%) was 63 to 135%.

2.3. Risk assessment model

In this study, models developed by the US Environmental Protection Agency (USEPA, 1996) and the Dutch National Institute of Public Health and Environmental Protection (Van den Berg, 1995) was used to calculate the children exposure to Pb in household dusts.

Children exposure of Pb to dust particles occurs via three main pathways including ingestion (D_{ing}), inhalation (D_{inh}), and dermal contact (D_{dermal}). The dose received through each of the three pathways was calculated with Eqs. (1)–(3) adopted from the USEPA (USEPA, 1989, 1996) and Guney et al. (2010).

$$D_{ing} = \left[C \times \frac{\mathrm{IngR} \times \mathrm{EF} \times \mathrm{ED}}{\mathrm{BW} \times \mathrm{AT}} \times 10^{-6}\right] \times B \qquad (1)$$

$$D_{inh} = C \times \frac{\mathrm{InhR} \times \mathrm{EF} \times \mathrm{ED}}{\mathrm{PEF} \times \mathrm{BW} \times \mathrm{AT}} \qquad (2)$$

$$D_{dermal} = C \times \frac{\mathrm{SL} \times \mathrm{SA} \times \mathrm{ABS} \times \mathrm{EF} \times \mathrm{ED}}{\mathrm{BW} \times \mathrm{AT}} \times 10^{-6} \qquad (3)$$

It assumed that every child was likely to stay 8 h per day on the floor in Chinese rural areas, so that we used 2920 h year^{-1} as the average exposure frequency (EF) for each child resident. Other exposure parameters are listed as follows:

IngR is the ingestion rate, in this study, 20 mg h^{-1} (USEPA, 1997);
InhR is the inhalation rate, in this study, 1.2 m^3 h^{-1} (USEPA, 2002);
EF is the exposure frequency, in this study, 2920 h year^{-1} for household dust exposure;
ED is the exposure duration, in this study, 6 years (De Miguel et al., 2007);
SA is the exposed skin area, in this study, 2800 cm^2 (USEPA, 2001a,b);
SL is the skin adherence factor, in this study, 0.07 mg cm^{-2} h $^{-1}$ (USEPA, 2002);
ABS is dermal absorption factor (unitless), in this study, 0.001 for Pb;
PEF is particle emission factor, in this study, 6.8 × 10^8 m^3 kg^{-1} (adopted for absence of vegetative cover from USEPA, 2001a,b);
BW is the average bodyweight, in this study, 15 kg (USEPA, 1989);
AT is the averaging time, in this study, ED ×365 for Pb.
C is the total concentration (mg kg^{-1}) of Pb in the dust and B is the bioaccessibility (%) of the dust Pb. An average bioaccessibility value of 53% was used to calculate the D_{ing} for Shandong and Qinghai provinces since bioaccessibe Pb in dust from these two provinces had not been determined due to the insufficiency of sample mass.

The C value in Eqs. (1)–(3), combine with the values for the exposure factors shown above yielded an estimate of the “reasonable maximum exposure” (USEPA, 1989), which is the upper limit of the 95% confidence interval for the mean (95% UCL). In this study, the concentration of Pb in the samples follows a log-normal distribution and consequently 95% UCL was calculated by Eq. (4) (USEPA, 1996). $\overline{X}$ is the arithmetic mean of the log-transformed data; s is the standard deviation of the log-transformed data; H is the H-statistics (Gilbert, 1987); n is the number of samples.

$$C_{95\%UCL} = \exp\left\{\overline{X} + 0.5 \times s^2 + \frac{s \times H}{\sqrt{n-1}}\right\} \qquad (4)$$

The doses calculated with Eqs. (1)–(3) for each exposure pathways were subsequently divided by the corresponding reference dose (RfD) to yield a hazard quotient (HQ). The HQ assumes that there is a level of exposure (RfD), below which it is unlikely for sensitive

Table 1
Total Pb concentrations in household dusts from Chinese rural areas (mg kg^{-1}).

Region	N[a]	Min	Max	Mean	SD
Shandong	9	80	534	207	139
Hubei	32	60	1036	238	191
Hunan	15	74	2510	538	739
Fujian	12	40	140	80	35
Shaanxi	17	29	243	96	75
Yunnan	4	42	941	280	441
Inner Mongolia	21	18	695	116	177
Qinghai	12	29	670	135	182
Total	122	18	2510	208	328

[a] Sample number.

populations to experience adverse health effects. If the exposure level (E) exceeds this threshold (i.e., if E/RfD > 1), potential adverse health effects may occur. As a rule of thumb, the greater the value of HQ, the greater the level of concern (USEPA, 1989).

Hazard index (HI) is equal to the sum of the HQ. The approach assumes that simultaneous sub-threshold exposures to several chemicals could result in adverse health effects. It also assumes that the magnitude of the adverse effect will be proportional to the sum of the ratios of the sub-threshold exposures to acceptable exposures (USEPA, 1989). In this study, Hazard index methods were used to assess health risk of Pb exposure to household dusts for children in Chinese rural areas.

3. Results and discussion

3.1. Lead concentrations in household dust

Pb concentrations in the household dusts from Chinese rural areas have an approximately log-normal distribution. The dust Pb exhibited a wide range from 18 mg kg^{-1} (Inner Mongolia) to 2510 mg kg^{-1} (Hunan) with a mean value of 208 mg kg^{-1} (Table 1). The spatial variations among different provinces may reflect the geological backgrounds and potential pollution sources. The high dust Pb concentrations found in Hunan and Yunnan provinces match well with the location of major lead–zinc mineralization belts in China (Chen and Peng, 2008). Consequently, the elevated levels of Pb in dust may be attributed to the high natural background (CNEMC, 1990) or/and the impact of the mining activities

Table 2
Comparison of dust Pb concentrations from different regions.

Region	Samples type	N[a]	Pb (mg kg^{-1})	References
Chinese rural areas	Household dust	122	208 (18–2510)[b]	This study
Shanghai	Street dust	273	295 (28–4443)	Shi et al., 2008
Beijing	Street dust	12	70 (27–135)	Xiang et al., 2010
Xi'an, Shaanxi	Street dust	65	231	Han et al., 2008
Shenyang, Liaoning	Street dust	61	106 (63–509)	Li et al., 2008
Huludao, Liaoning	Street dust	30	533 (97–3903)	Zheng et al., 2010
Hong Kong	Street dust	45	181	Li et al., 2001
U.K. (national survey)	Household dust	32	181 (54–358)	Turner and Simmonds, 2006
Sydney, Australia	Household dust	82	85 (18–16,600)	Chattopadhyay et al., 2003
Dhaka, Bangladesh	Street dust	64	54 (19–205)	Ahmed and Ishiga, 2006
Ottawa, Canada	Street dust	48	406 (50–3226)	Rasmussen et al., 2001
Kavala, Greece	Street dust	96	301 (8–2500)	Christoforidis and Stamatis, 2009

[a] Sample number.
[b] Mean (range).

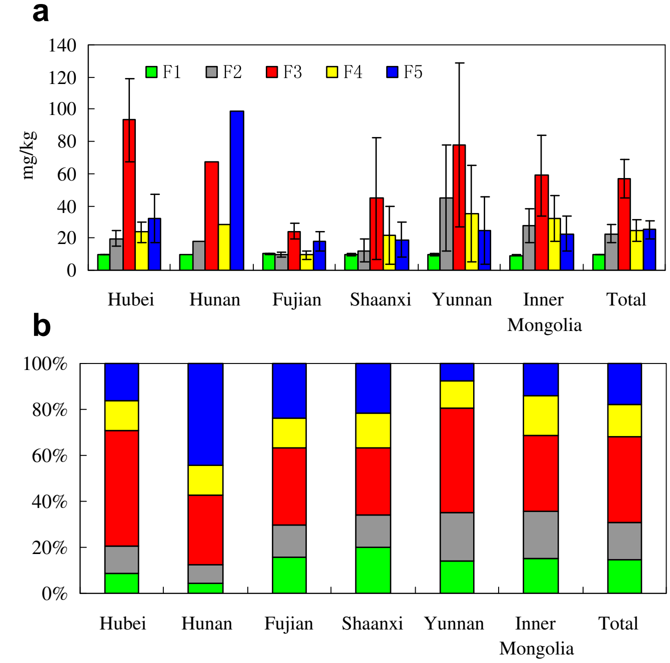

Fig. 1. Speciation of Pb in household dusts from Chinese rural areas. a: total concentration; b: percentage (N = 33).

(Gu and Zhao, 2010; Long et al., 2010; Williams et al., 2009). The lowest Pb concentrations were found in Shaanxi and Fujian provinces. Nevertheless, Pb concentration in dust exhibited a wide range in-between residential buildings within the same province (Table 1), which may be related to a diversity in lifestyles, house decoration and domestic heating (e.g., using of coal and paint).

In comparison with the national soil background value of 24 mg kg^{-1} (CNEMC, 1990), Pb in household dusts of the rural areas was profoundly elevated, indicating that Pb had been enriched through anthropogenic activities, for example via mining activity in Hunan provinces (Williams et al., 2009). About 10% of rural household dust samples exceeded the safety threshold of 400 mg kg^{-1} defined by USEPA (USEPA, 2001a,b). Adhering to the Chinese children's safety benchmark of 282 mg kg^{-1} for soil (Zhang et al., 2009), as much as 17% of the household dust samples surpassed this benchmark level.

Comparison between the Chinese rural areas presented in this study and other urban cities worldwide is presented in Table 2. Results indicated that Pb in dust was basically at a similar level. In urban environment, vehicle emission, coal combustion and industrial activities are the major sources of Pb pollution, along with other anthropogenic sources, such as manufacture of vehicle batteries, glass, radiation shields and soldering (Franco and Mattia,

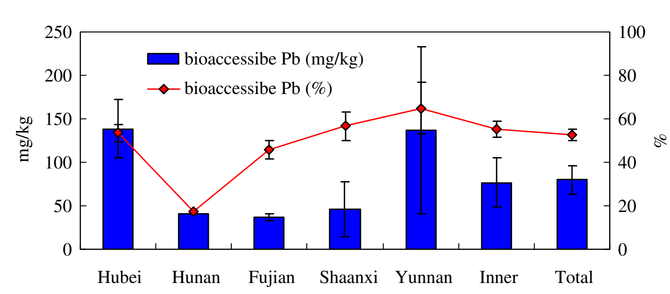

Fig. 2. Bioaccessiblility of Pb in household dusts from Chinese rural areas (N = 33).

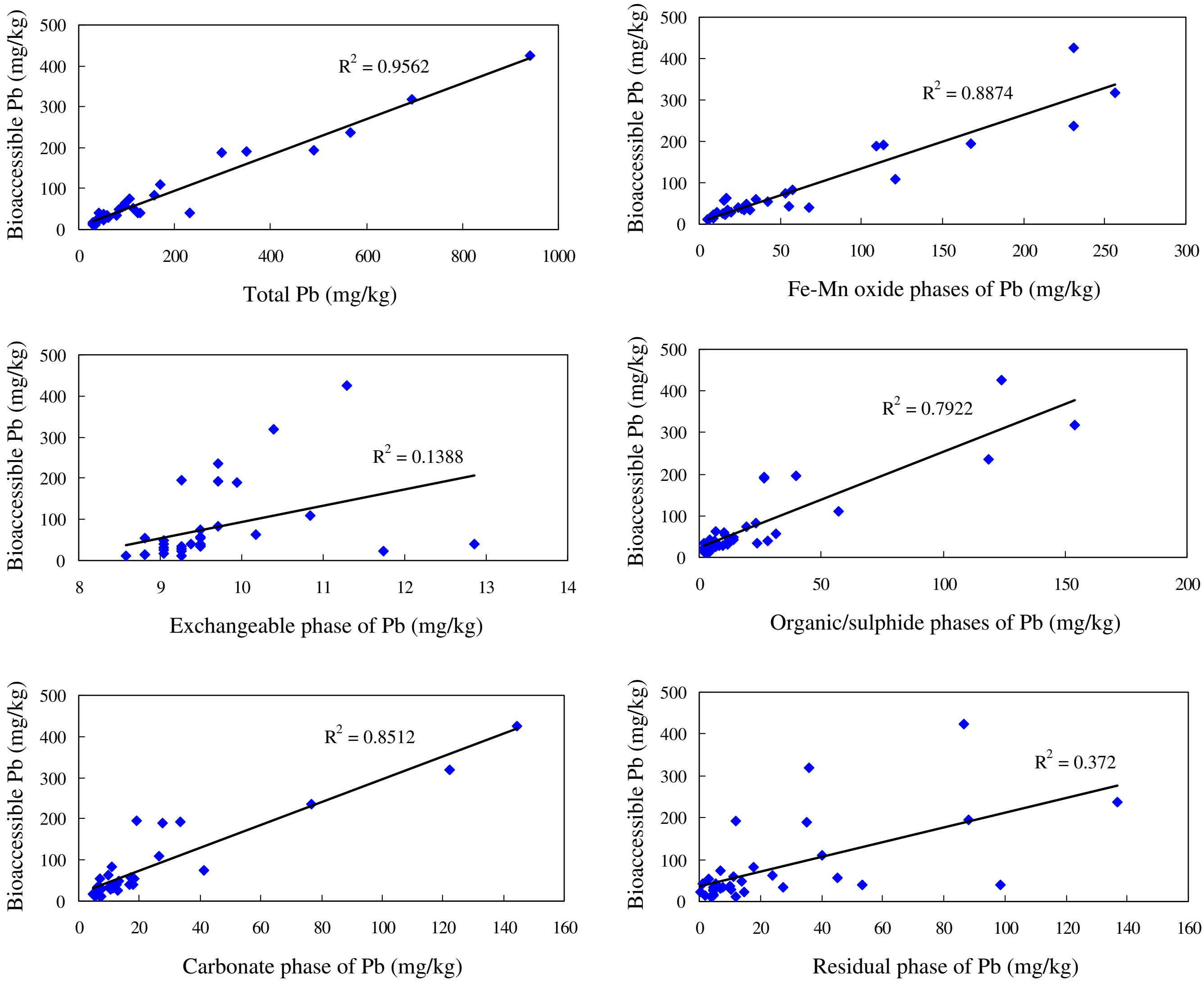

Fig. 3. Correlation between bioaccessible Pb and different phases of Pb in household dusts from Chinese rural areas.

2010; Mielke et al., 2011). However, there was no known anthropogenic pollution source in the rural villages sampled in this study. Accordingly, it is plausible that, besides the possible impacts of the elevated background and mining activity, the household dust Pb to a significant extent was derived from indoor surfaces and indoor air quality (e.g., paints, coal combustion). In addition, Pb concentrations may be affected by the residents' occupational work (e.g. battery factory employees). Enrichment factors ranging from 3 to 26 in the Pb concentration in house dust have been reported for the households including such workers compared with controls (Fergusson et al., 1981; Rice et al., 1977). Thus, further investigations should be carried out to determine what the main sources of lead in dust and soil are in the rural villages.

3.2. Speciation of lead in household dust

A sub-set of the dust samples was analyzed for Pb speciation using sequential extraction procedures. The absolute concentration and relative distribution of Pb in the five phases (F1–5) are shown in Fig. 1. Except for samples from Hunan province having high residual fraction of Pb (45%), that mainly represented the association of Pb with primary or/and secondary minerals, the samples from remaining provinces showed a similar pattern in Pb speciation. In general, Pb in rural household dust was dominated by the form of Fe–Mn oxide phases, which accounting for 37% of the total Pb; followed by residual fraction (18%) and carbonate phase (17%). The percentage of exchangeable phase (14%) and organic/sulphide phase (14%) was slightly lower. The predominance of Pb in Fe-Mn oxide phases observed by many studies (Li et al., 2001; Banerjee, 2003), is owing to the adsorption of Pb cations on the hydrous (amorphic) oxides of Fe and Mn.

The exchangeable phase of trace metals is toxically the most active form. In road dust this fraction often accounts for less than 5% (Fergusson and Kim, 1991; Li et al., 2001) and therefore considered less important in risk assessments. In the present study, the proportion of Pb in the exchangeable phase was significantly higher than those of the previous studies, exceeding 10% (average of 14%) for most samples, indicating that Pb in the investigated dust samples may have a comparatively higher activity and toxicity. A possible cause for elevated Pb levels in the exchangeable phase is acidification. Fergusson and Kim (1991) reported that lower pH value (<5) could notably increase the exchangeable Pb fraction. The comparatively low distribution of Pb in the carbonate phase observed in this study compared to other published data in the range of 30%–40% (Fergusson and Kim, 1991; Li et al., 2001), give further support for this hypothesis given the strong dependence of the carbonate system with pH.

3.3. Lead bioaccessibility in household dust

Bioaccessibility of Pb in dust can be assessed by a *in vitro* method, which aiming to mimic the digestion and absorption environment present in the gastrointestinal system, by adding gastrointestinal enzymes and organic acids to the extracting agent kept thermostated at 37 °C under low-oxygen atmosphere and simulating gastrointerstinal peristalsis. The initial *in vitro* method was established by Ruby et al. (1993), however, it was found impractible to process at a reasonable rate samples due to a great many reagents involved long experimental period, and fussy experimental procedures. Here we have adopted a simple and fast approach described by Mercier et al. (2002). The bioaccessibility of Pb was expressed as the percentage of the extractable Pb with respect to its total concentration in the dust (Fig. 2).

As shown in Fig. 2, the ratios of bioaccessible Pb in the dust ranged from 17% (Hunan) to 65% (Yunnan) with a mean value of 53%. This finding was in good agreement with previous results which varied from 50% to 90% (Mercier et al., 2002; Rasmussen, 2004; Turner and Simmonds, 2006). The low bioaccessibility of Pb in dust from Hunan was in accordance with the sequential extraction result which showed that the residual fraction was dominant. The distribution pattern of bioaccessible Pb in the dusts was similar to the total Pb. Correlation analysis ($R^2 = 0.96$, $p < 0.001$) showed that the bioaccessible Pb was significantly correlated to the total Pb (Fig. 3), indicating that the bioaccessibility of Pb was mainly controlled by total Pb concentrations. This result was in agreement with Rasmussen's study (Rasmussen et al., 2011), in which the bioaccessible Pb was determined after treatment with dilute hydrochloric acid (pH 1.5). Further analysis revealed that the bioaccessible Pb was significantly correlated to the Fe–Mn oxides phases, carbonate phase and organic phase of Pb (Fig. 3), suggesting the important contribution of Fe–Mn oxides phases, the dominant Pb speciation, to the bioaccessible Pb pools. Marschner et al. (2006) found that Pb absorbed by the swine had a close relationship with Fe–Mn oxide phases of Pb, which accordingly was highly soluble in the gastrointestinal anoxic and reducing environment. Consequently, the household dusts with high Fe–Mn oxides fractions of Pb could have high adverse effect to local inhabitants.

3.4. Potential health risk of Pb in household dusts to children

Reference doses for Pb have been derived from the WHO (1993) Guidelines for Drinking Water. Dermal reference doses was 5.25 mg $kg^{-1}day^{-1}$. Inhalation-specific toxicity data was not available for Pb, so the corresponding ingestion reference doses (3.5×10^{-3} mg $kg^{-1}day^{-1}$) was used here, on the assumption that, after inhalation, the absorption of the particle bound toxicants will result in similar health effects as the particles had been ingested (Van den Berg, 1995; Naturvårdsverket, 1996; De Miguel et al., 2007). The results of health risk assessment for children in the rural areas of China are listed in Table 3.

Ingestion of dust particles appears to be the main route of exposure to household dusts and could result in a higher risk, followed by dermal contact. This result was in accordance with previous reports (Ferreira-Baptista and De Miguel, 2005; Zheng et al., 2010). HQ due to inhalation of dust particles is 2–4 orders of magnitude lower than the other two exposure pathways.

Hazard Indexes (HIs) for most samples were lower than 1, indicating that most rural areas were relatively safe for children when they exposure to the household dust. Table 3 shows that Shandong and Hubei provinces had higher HIs, while low HIs were found in Fujian and Inner Mongolia. Although Hunan province had the highest Pb concentration in dust, low bioaccessibility of Pb (17%) here have a moderating effect on the outcome of the risk

Table 3
Hazard quotient and risk for each exposure pathway in household dusts.

Region[a]	C (95%UCL)	HQ_{ing}	HQ_{inh}	HQ_{dermal}	$HI = \sum HQi$
Shandong	2.72E+02	4.39E−01	7.32E−05	5.42E−02	4.94E−01
Hubei	2.40E+02	3.95E−01	6.46E−05	4.78E−02	4.43E−01
Hunan	5.11E+02	2.65E−01	1.37E−04	1.02E−01	3.67E−01
Fujian	9.66E+01	1.35E−01	2.60E−05	1.92E−02	1.54E−01
Shaanxi	1.07E+02	1.87E−01	2.90E−05	2.14E−02	2.08E−01
Inner Mongolia	1.49E+02	1.68E−01	2.70E−05	2.00E−02	1.88E−01
Qinghai	1.33E+02	2.41E−01	4.00E−05	2.96E−02	2.70E−01
Total	2.56E+02	4.13E−01	6.88E−05	5.09E−02	4.64E−01

C: Exposure point concentration (mg kg^{-1}).
[a] Data from Yunnan were not included for statistical reasons due to limited number of samples.

assessment. Overall, 4.1% of the residential buildings investigated had Pb levels in household dust exceeding the criteria level calculated with our model. He et al. (2009) had summarized the BLLs of children from the provinces considered in this study and found that about 7.5–14.7 % of children's BLLs were higher than 10 μg dl^{-1}. The result of the present study suggested that the elevated Pb in household dust may be an important reason for these high BLLs of children; however, its contribution needs to be fully investigated.

4. Conclusion

This research for the first time provided a preliminary database for Pb contamination in household dust from Chinese rural areas. Pb concentrations in this study were similar to many urban cities in the world. Dust Pb in exchangeable phase accounted for more than 10%, showing a higher toxic activity. Pb associated with Fe–Mn oxides phases dominated the speciation in most of the samples and found to be significantly correlated to the bioaccessible Pb fraction. The pathway associated with the highest levels of risk for children exposed to household dust was ingestion, followed by dermal contact and inhalation of resuspended particles. The HIs indicated that most rural areas in this study should be considered relatively safe for children with respect to Pb dust exposure. However, dust Pb in 4.1% of the studied families having HI values higher than 1 may pose adverse health effect to the children.

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