New isotopic evidence of lead contamination in wheat grain from atmospheric fallout

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Abstract Crops could accumulate trace metals by soil-root transfer and foliar uptake from atmospheric fallout, and an accurate assessment of pollution sources is a prerequisite for preventing heavy metal pollution in agricultural products. In this study, we examined Pb isotope rates to trace the sources of Pb in wheat grain grown in suburbs. Results showed that, even in zones with scarcely any air pollution spots, atmospheric fallout was still a considerable source of Pb accumulation in wheat. The concentration of Pb in wheat grain has poor correlation with that in farm soil. The Pb concentration in wheat grains with dust in bran coat was significantly higher than that in wheat grains, which indicates that Pb may accumulate by foliar uptake. The Pb isotope rate has obvious differences between the soil and atmospheric fallout, and scatter ratio is significantly closer between the wheat grain and atmospheric fallout. Atmospheric fallout is a more significant source of Pb concentration in wheat grains than in soil. As far as we know, this is the first study on the main sources of lead in grain crop (wheat) samples with isotope. This study aims to improve our

understanding of the translocation of foliar-absorbed metals to nonexposed parts of plants.

Keywords Pb isotopic · Pb contamination · Wheat grain · Atmospheric · Fallout · Soil

Introduction

Lead (Pb) contamination and its health effects are widespread around the world (Duzgoren-Aydin et al. 2004; Rankin et al. 2005). The unhealthy effects of Pb include neurological impairment and deficits in the function of the central nervous system (Huang et al. 2011; Ortega et al. 2013; Szkup-Jablonska et al. 2012). The Pb accumulation in crops represents an important route of toxins into human food chains (Henson and Reardon 2005; Luo et al. 2012; McHugh and Avena-Bustillos 2011). Wheat (*Triticum aestivum* L.) is an important crop worldwide as a basic food commodity (Food and Agriculture Organization 2013). Various studies have revealed the link between the different degrees of Pb pollution in wheat grains and the threat they pose to human beings (Bermudez et al. 2011, 2012; Boussen et al. 2013; Douay et al. 2008; Nan et al. 2002). Preventing Pb pollution in wheat grains is necessary for food safety, which requires accurate verification of pollution sources.

The existing methods of heavy metal source tracing in plants include field test, mathematical analysis, and isotopic tracing. Douay et al. (2008) compared the Pb contents in wheat grains before and after closure of a local Pb and Zn smelter and found that, after the shutdown, the Pb contents in wheat grains and straw decreased by 82 and 91 %, respectively. These findings indicated that the atmospheric fallout from the smelter was responsible for the Pb pollution in wheat.

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Harrison and Chirgawi (1989) used growth cabinets with filtered air to demonstrate the effect of airborne metals on several plant species; the atmospheric contribution of Pb to the contamination of spinach was up to 85 %. Bermudez et al. (2012) set up a linear regression model based on the Pb contents in atmospheric fallout, root soil, and wheat grains to assess the contributions of various sources. However, mathematical analysis can hardly be conducted on the limited data acquired from field tests, and setting a control group presents a problem.

Fractionation because of microbial, chemical, and physical processes has no influence on the isotopic ratio of Pb in the environment; thus, Pb isotope has been employed to track heavy metal assimilation paths in plants. Hu et al. (2011) used ^{210}Pb to estimate quantitatively the amount of lead for *Aster subulatus*, and the results showed that the approximate contribution of airborne Pb in plant leaves was 72.2 %. Dollard (1986) used ^{210}Pb to examine the foliar uptake and redistribution of lead in several plant species and found that Pb foliar absorption accounts for approximately 35 % of the internal lead burden of radish root tissues, but only for 3 % of the Pb in carrots. $^{206}\text{Pb}/^{207}\text{Pb}$ ratios indicated that the Pb in the needles of pine trees in Sweden was dominated by direct Pb accumulation from the atmosphere (Uzu et al. 2010). Corn plants around an industrial air pollutant (smelter) assimilated the atmospheric fallout through their leaves and transferred them to the grains, as revealed by the isotopic tracing test conducted by Bi et al. (2009). However, most of the available data focus only on the effect of atmospheric dust on plants, and research on the effects on cereal grain is limited.

The objective of the present study is to trace the contributions of the suburban atmospheric fallout to the lead content in grain crops (wheat) with Pb isotope. The results are intended to improve our understanding of the translocation of foliar-absorbed metals to nonexposed parts of plants. As far as we know, this is the first study on the main sources of lead on grain crop (wheat) samples with isotopes collected from a suburban area.

Materials and methods

Study area

The experiment was conducted in the east-south suburb of Beijing in North China Plain, the main wheat-producing area in China. The industrial–structural adjustment resulted in the relocation of the smelting industry to the surrounding cities (e.g., Shougang Group moved to Tangshan, Hebei province). Thus, the study area is clear of air pollution. The dominant crops in the study area are corn and wheat. The annual precipitation is 620 mm. The main soil types are fluvo-aquic

(calcaric cambisol) and cinnamon (anthrosol). The soil pH of the study area is 7.8–8.6.

Sampling

Three sampling points were distributed in the study area as shown in Fig. 1. Samplers were installed on a telegraph pole in the southeast suburb of Beijing, over 5 m above ground, to avoid disturbance from surrounding buildings and secondary blowing dust. Fifty milliliters of hexanediol was added to the samplers in advance to keep the sampler moist while inhibiting microbes and algae. After the leaves and insects were removed, distilled water was added to wash out the retained dust. The mixture was kept in a refrigerator before analysis.

Atmospheric fallout was sampled monthly from February to July in 2008. A total of 14 samples were collected. A total of 21 wheat grain samples and corresponding root-soil samples were also collected. Sampling spots were distributed as shown in Fig. 1.

Chemical analyses

Natural dried soil samples were ground to 100 mesh, and approximately 300 mg of sample was digested with 6 ml of HCl (30 %, v/v), 2 ml of HNO_3 (65 %, v/v), and 2 ml of HF (40 %, v/v) in a microwave digestion system (MARS 5, CEM) for 30 min (Bi et al. 2009). The digested solution was diluted with 25 ml of Milli-Q water. Atmospheric fallout samples (200 mg) were digested with 6 ml of HCl (30 %, v/v), 2 ml of HNO_3 (65 %, v/v), and 2 ml of HF (40 %, v/v) in a microwave digestion system (MARS 5, CEM) for 30 min after the digested mixture was dissolved to 25 ml (Bi et al. 2009). The Pb concentrations of the solutions were determined via flame atomic absorption spectrometry (AAS 5100, Perkin-Elmer Inc.).

To compare the difference in Pb concentrations between wheat grains and wheat grains with dust in bran coat, we divided wheat seed samples into two groups; one group was washed with deionized water and dried to a constant weight, and the other group was dried to a constant weight without washing. The dried wheat seeds were then ground, and the samples (500 mg) were digested with 6 ml of HNO_3 (65 %, v/v) and 2 ml of H_2O_2 (30 %, v/v) in a microwave digestion system for 30 min (Bi et al. 2009). The digested solution was diluted with 25 ml of Milli-Q water. The Pb contents of the solutions were determined with graphite furnace atomic absorption spectrometry (AAS 5100, Perkin-Elmer Inc.).

Nationally certified reference materials of soil (GSS-1, GSS-2) and plant (GSV-1, GSV-3) were followed for quality control. The results showed that the recovery rate of heavy metals in the soil and plant samples were within the allowable/acceptable range of the nationally certified reference

Fig. 1 Sketch map showing the sampling sites in the southeastern suburbs of Beijing, China

materials. The recovery rate of Pb in the standard plant samples was 90.6 ± 5.1 % (84.5–115 % as required by the national standard), and the recovery rate of Pb in the standard soil samples was 95.6 ± 4.1 % (85.0–115 % as required by the national standard).

Isotope analysis

The Pb isotopic composition was analyzed for atmospheric fallout, soil, and wheat grain samples by ICP-MS (Perkin-Elmer Elan 6100 DRC^{plus}) at the Beijing Research Institute of Uranium Geology. The details of the procedure were based on the determinations for isotopes of lead, strontium, and neodymium in rock samples (GB/T 17,672–1999). Standard material was used for the isotopic ratio of Pb (SRM981, common lead isotopic material, National Institute of Standards and Technology, USA). The samples were analyzed four times. The instruments were calibrated with a standard material for Pb (SRM981) for every three samples (Bi et al. 2009; Hu et al. 2011). The analysis was repeated when the difference between the measured and certified values of the standard reference

material exceeded 0.5 %. The average Pb ratios of $^{206}\text{Pb}/^{207}\text{Pb}$ and $^{208}\text{Pb}/^{207}\text{Pb}$ (1.0909 ± 0.0011 and 2.3625 ± 0.0021) were in good agreement with the standard reference values (1.0933 and 2.3704, respectively).

Result and discussion

Pb concentration in soil and atmospheric fallout

No significant Pb pollution was found in the study area. The Pb content in the soil in the study area (12.66 ± 1.13 mg kg⁻¹) was lower than the background content in the urban Beijing district (content 24.6 ± 5.8 mg kg⁻¹; range 11.5–38.2 mg kg⁻¹) and was close to the rate reported by Zheng et al. (2008).

The Pb content in the atmospheric fallout was 120.3 ± 6.08 mg kg⁻¹ (77.1–194.5 mg kg⁻¹) in the study area. The Pb concentrations in the atmospheric fallout sampled from three sites were 117.2 ± 43.0 mg kg⁻¹, 116.4 ± 8.16 mg kg⁻¹, and 127.3 ± 43.0 mg kg⁻¹ (Fig. 2), close to the Pb content in the total suspended particulates (TSP) emitted by thermo-

Fig. 2 Pb concentration of atmospheric fallout in the southeastern suburbs of Beijing, China

electric plants in Beijing as reported by Widory et al. (2010), higher than the Pb content in the TSP emitted by cement plants and surrounding soil around Beijing ($33.0 \pm 8.00 \text{ mg kg}^{-1}$) (Widory et al. 2010), and significantly lower than the Pb content in the TSP emitted by smelters in Hebei province ($16,100\text{--}44,400 \text{ mg kg}^{-1}$) (Liu et al. 2011).

The ratio range for $^{206}\text{Pb}/^{207}\text{Pb}$ in the atmospheric fallout in this study was 1.166–1.179 (Fig. 3), which was evidently higher than the content in petrol (1.06–1.09) and the TSP near lead-refining plants (1.137–1.156), partially overlapping with the industrial coal dust in Beijing (1.157–1.176) and the soil of Inner Mongolia (1.173–1.181).

Although no perceptible industrial air pollution point is around the study area, the diffusion of atmospheric fallout from a long distance leads to a mixture of Pb from multiple sources in this area (Gouin et al. 2004; Inoue et al. 2014). Despite the strong decrease in industrial and vehicle lead emissions at a global scale in recent decades (Glorennec

Fig. 4 Comparison of Pb concentration in wheat grains and wheat grains with dust in bran coat

et al. 2007), lead-enriched particles are still emitted to the environment, especially by coal combustion (Diaz-Somoano et al. 2009). Coal burning and biowaste burning account for 27 % of the $\text{PM}_{2.5}$ (aerosol particles with aerodynamic diameter less than $2.5 \mu\text{m}$) pollution in Beijing in 2009. Apart from coal burning, the long distance transfer of dust from Inner Mongolia also contributes to the Pb in the atmospheric fallout in Beijing. It accounts for 23 % of the $\text{PM}_{2.5}$ pollution in Beijing during the Spring Festival.

Pb concentration in wheat grain

The Pb concentration in wheat grains ranged from 0.036 to 0.273 mg kg^{-1} , and the average value was $0.140 \pm 0.062 \text{ mg kg}^{-1}$ (Fig. 4). Four grain samples (accounting for 20 % of the total samples) in this study had higher concentrations of Pb than the national guidance limit for various types of food in China ($0.2 \text{ mg Pb kg}^{-1}$) (National Health and

Fig. 3 $^{206}\text{Pb}/^{207}\text{Pb}$ ratio in different mediums

Fig. 5 $^{206}\text{Pb}/^{207}\text{Pb}$ - $^{208}\text{Pb}/^{206}\text{Pb}$ ratio scatter diagram of different mediums

Family Planning Commission of China 2012). Compared with the findings of previous studies, the wheat grains in the study area were slightly polluted, with a Pb content of $0.140 \pm 0.062 \text{ mg kg}^{-1}$, which was lower than the level (1.31 and 0.65 mg kg^{-1}) in the zones polluted by smelters (Douay et al. 2008; Pruvot et al. 2006) and that in Baiyin, Gansu province, China, where plants were irrigated by polluted water (0.48 mg kg^{-1}) (Nan et al. 2002). The pollution level in the study area was also slightly higher than the 0.088 mg kg^{-1} rate in the agricultural zone in Córdoba, Argentina (Bermudez et al. 2011).

The Pb concentration in wheat grains with dust in bran coat ranged from 0.059 to 0.915 mg kg^{-1} , and the average value was $0.343 \pm 0.255 \text{ mg kg}^{-1}$. The Pb concentration in wheat grains with dust in bran coat was 2.04 times that of the

concentration in wheat grains. The difference in the test of statistical significance indicated that the Pb concentration in wheat grains with dust in bran coat was significantly higher than that in wheat grains ($p=0.008 < 0.05$).

Contribution of soil lead to wheat-seed lead content

In the present study, correlation analyses between the Pb concentrations in soils and in the wheat grains showed that soil Pb concentrations were not significantly correlated to the Pb concentrations in the wheat grains ($p=0.618 > 0.05$). This result indicated that the Pb content in soil was not a determinant of the Pb content in wheat grains. According to Douay et al. (2008), the influence of soil Pb on wheat grain Pb content was significantly less than that of atmospheric fallout, even with a soil Pb concentration as high as 290 mg kg^{-1} . Another study (Bi et al. 2009) showed that with a soil Pb concentration of 2300 mg kg^{-1} , Pb in corn mainly occurred in roots and scarcely in corn grains.

Accumulating Pb from soil is difficult for wheat grains because of the low bioavailability of Pb in the soil and the combination form of Pb in the roots. The contents of DTPA-extractable Pb of soils in the study area accounted for 10.7 % of the total amount of soil Pb and were significantly less than the contents of the DTPA-extractable Cu and Cd (Yang et al. 2005). A lower Pb bioavailability would inhibit the assimilation by plants. In the aspect of binding characteristics, Pb in the soil is nearly always tightly bound to organic or colloidal material or in a precipitated form, all of which serve to reduce the uptake of Pb by the plant roots. At the root surface, Pb binds to carboxyl groups of mucilage uronic acids. Mucilage binding restricts metal uptake into the root and establishes an

Table 1 Isotope ratios and elemental content of different mediums

Atmospheric fallout			Soil in Beijing			Wheat grain		
Pb (mg/kg)	$^{208}\text{Pb}/^{206}\text{Pb}$	$^{206}\text{Pb}/^{207}\text{Pb}$	Pb (mg/kg)	$^{208}\text{Pb}/^{206}\text{Pb}$	$^{206}\text{Pb}/^{207}\text{Pb}$	Pb (mg/kg)	$^{208}\text{Pb}/^{206}\text{Pb}$	$^{206}\text{Pb}/^{207}\text{Pb}$
119.4	2.0806	1.1788	13.3	2.1019	1.1716	0.110	2.0798	1.176
104.7	2.0749	1.1762	12.6	2.1047	1.1699	0.182	2.0748	1.1779
122.8	2.0822	1.1713	10.6	2.0980	1.1735	0.139	2.0695	1.1817
111.3	2.0805	1.1737	12.6	2.0991	1.1720	0.273	2.0732	1.1830
123.7	2.0831	1.1706	11.9	2.1125	1.1623	0.103	2.0801	1.1781
77.1	2.0856	1.1683	13.8	2.1053	1.1686	0.122	2.0788	1.1801
102.7	2.0908	1.1667	12.1	2.1041	1.1698	0.13	2.0776	1.1787
189.1	2.0869	1.1731	12.2	2.1103	1.1630	0.123	2.0771	1.1803
96.9	2.0872	1.1682				0.141	2.0741	1.1792
120.0	2.0962	1.1677				0.095	2.0732	1.1784
146.6	2.0804	1.1746				0.203	2.0961	1.1633
194.5	2.0815	1.1686						
94.7	2.0937	1.1686						
97.5	2.0944	1.1663						

important barrier that protects the root system (Sharma and Dubey 2005). In the transfer paths in plants, Pb retention in the roots is based on the binding of Pb to ion exchangeable sites on the cell wall and on extracellular precipitation mainly in the form of Pb-carbonated deposits in the cell wall, which restrict Pb transportation from the roots to the aboveground parts of plants (Krzeslowska 2011; Sharma and Dubey 2005). Therefore, the Pb concentrations transported from soil in the wheat grain are low and mainly accumulated in the roots, indicating that atmospheric fallout may be responsible for the presence of Pb in wheat grains.

Contribution of atmospheric fallout to lead content in wheat seed

The scatter plot in Fig. 5 shows that the ranges of Pb isotope ratios in wheat grain overlapped partially with those in the atmospheric fallout and were far from those in the soils. The wheat grains and atmospheric fallout were characterized by relatively high $^{206}\text{Pb}/^{207}\text{Pb}$ (1.163–1.190, 1.166–1.179) and low $^{208}\text{Pb}/^{206}\text{Pb}$ (2.059–2.096, 2.075–2.096). By contrast, soils exhibited a rather low $^{206}\text{Pb}/^{207}\text{Pb}$ (1.162–1.172) and high $^{208}\text{Pb}/^{206}\text{Pb}$ (2.098–2.113) (Table 1). A significant difference was observed between the Pb isotope compositions in the atmospheric fallout and in the soil.

As a result of different inputs, pathways may contain Pb with characteristic ratios. Tracing various Pb sources in a plant based on Pb isotope composition analyses is possible (Gwiazda and Smith 2000; Haack et al. 2004; Monna et al. 1997; Siu et al. 2005). In our study, the Pb content in the wheat grains was mainly derived from atmospheric fallout as indicated by similar isotope ratios. This finding is consistent with reported results that plant leaves can uptake Pb directly from atmospheric fallout (Bi et al. 2009; Dollard 1986; Hu et al. 2011; Uzu et al. 2010).

Lead may penetrate the leaf by two pathways. First, the lead-containing nanoparticles observed in the stomata may penetrate the apoplast as solid compounds. Second, the lead resulting from the dissolution of source particles may diffuse through the aqueous pores of the cuticle and the stomata, following the hydrophilic pathway and inducing the formation of necroses enriched with lead (Uzu et al. 2010). According to Schreck et al. (2012), the original particles contained PbS and PbS transformed to lead secondary species (PbCO_3 and organic Pb) on leaf surfaces, and some compounds were internalized in their primary form (PbSO_4) underneath an organic layer (Choël et al. 2006; Huang et al. 1994). However, for grains wrapped by coating, the accumulated Pb in wheat grain is possibly transported from the wheat bran bread via phloem. Some studies proved that foliar Pb could be translocated to actively growing regions. With the help of isotopic tracing, Bi et al. (2009) found that the Pb that settled on the leaves was assimilated by the leaf tissue and then transferred to the core

grains. However, the manner by which Pb is transported to wheat grain should be explored in depth.

According to the results of our study, atmospheric fallout may be responsible for the Pb pollution in wheat grains even in farmlands away from industrial pollution sites. This study demonstrated a direct link between air pollution and the threat it poses to food safety. Air pollution is a serious problem in China partly because it threatens food safety. This study also represents a new exploration of the mechanism of Pb transfer between atmospheric fallout, grain plants (wheat), and soil system.

Conclusion

The Pb contents and their isotopic ratios in wheat grains, root soil, and atmospheric fallout collected during the wheat-growing season were analyzed to identify the main source of Pb in wheat grains in the suburban study area. The results showed that the Pb content in root soil was weakly linked to the Pb content in wheat grains. Atmospheric fallout adheres to the theory that wheat grains may be responsible for the Pb assimilated by plants. The Pb isotope rates of the soil and atmospheric fallout had an obvious difference, and the scatter ratio was significantly closer between the wheat grain and atmospheric fallout. Atmospheric fallout was a more important source of Pb concentration in wheat grains than in soil. The assimilation of Pb from atmospheric fallout was the main transfer path of Pb in wheat grains. These findings provide new evidence to explore the Pb transfer mechanism in a multimedium system such as atmospheric fallout, grain plant (wheat), and soil. Such knowledge may be relevant from the perspective of risk assessment of atmospheric emissions in urban environments.

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