Occurrence of 8 trace elements in *Rhizoma Cibotii* from China and exposure assessment

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Received: 8 August 2023 / Accepted: 16 October 2023 / Published online: 28 October 2023
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Abstract
The contamination of trace elements in Chinese edible herbs has attracted worldwide concern over the world. The objective of the present study was to investigate the occurrence and exposure assessment of eight trace elements in *Rhizoma Cibotii* from China. For this purpose, the method of inductively coupled plasma mass spectrometry was employed to detect the contamination levels of target trace elements in 58 *Rhizoma Cibotii* samples. The results demonstrated that the trace elements of Cr, Ni, Cu, Zn, and Pb were detected in all analyzed samples; the occurrence frequencies of As, Se, and Cd were 98.3%, 96.6%, and 98.3%, respectively. The highest mean levels were found in Zn (17.32 mg/kg), followed by Pb (8.50 mg/kg) and Cu (3.51 mg/kg). For a further step, one-way ANOVA was used to compare the difference of eight elements levels among groups, and Pearson’s correlation analysis was used to explore the correlation between elements in *Rhizoma Cibotii*. A strong positive correlation between Zn and Cd was observed by Pearson’s correlation analysis, which indicated that the possible presence of Cd contamination in *Rhizoma Cibotii*. Based on the contamination levels, the mean exposure of individual element and the health risks of eight trace elements in *Rhizoma Cibotii* were estimated by health risk assessment models. The calculated HQ values were less than 1, indicating that the contamination of trace elements in *Rhizoma Cibotii* did not pose significant health risks to human. In conclusion, the study provided baseline information on the contamination levels of trace elements in *Rhizoma Cibotii*. Moreover, it is necessary to monitor the trend of trace elements levels in *Rhizoma Cibotii*, which will be useful for ingredient control and human health protection.

Keywords Trace elements · Occurrence · Dietary exposure · Health risk · Food safety · *Rhizoma Cibotii*

Introduction
Trace elements are essential for the normal development, growth, and physiology of an organism (Zhang 2017). Humans are exposed to trace elements in daily life from a variety of sources, such as water, air, food, and medicines (Gaonkar et al. 2020; Sadeghi et al. 2019; Wang et al. 2022; Zhao et al. 2023). At present, various trace elements have been studied and detected in different fields, especially the investigation of trace elements in food has attracted more and more attention (Gupta et al. 2019; Jiang et al. 2022; Latifi and Jalali 2018). Among these trace elements, chromium (Cr), nickel (Ni), copper (Cu), zinc (Zn), arsenic (As), selenium (Se), cadmium (Cd), and lead (Pb) have been widely studied in various foods (Haseeb-Ur-Rehman et al. 2023; Jiang et al. 2016; Jiao et al. 2018; Raeeszadeh et al. 2022). Exposure to As, Cd, and Pb is known to be very dangerous and may cause adverse effects in animals and humans.
(Pierzan et al. 2022). On the other hand, Cr, Cu, Ni, Zn, and Se are all essential elements for the human body, which play important roles in the maintenance of numerous physiological processes. However, they can also have toxic effects at high concentrations (Jomova et al. 2022; Zhou et al. 2022). Thus, many organizations and countries have regulated maximum levels (MLs) of trace elements in foods with the aim of maintaining consumer confidence and human health (Li et al. 2018; Potorti et al. 2020; Sun et al. 2022).

Chinese edible herbs are closely associated with a rich history, civilization, and accumulation of knowledge and practice over the last thousand years (Wang et al. 2018; Yang et al. 2018). They are broadly used for alleviating and treating various human diseases owing to their benefits to human health worldwide (Wang et al. 2021). However, Chinese edible herbs may suffer from a few safety issues, such as the contamination of trace elements (Li et al. 2018; Yang et al. 2018). The pollution of soil with trace elements is the main source for the contamination of these herbs, which is caused by polluted irrigation, atmospheric dusts, industrial activity, pesticides, and fertilizer residue (Tokaloğlu 2012; Witkowska et al. 2021). The absorption of heavy metals by Chinese edible herbs poses a potential health threat to animal and human health, which can cause functional necrosis of liver and kidney and damage the basic function and metabolism of cells (Chen et al. 2023; Luo et al. 2020). Therefore, Chinese edible herbs may contain high concentrations of various trace elements that can adversely affect human physiological health. Especially, the quantitative analysis of trace element concentrations plays an important role in understanding the effectiveness of Chinese edible herbs in treating different diseases and exploring their pharmacological action. So, it is necessary to investigate the contamination concentrations of multiple trace elements in Chinese edible herbs and then estimate the risk assessment of the consumers.

From this perspective, the methods with high sensitivity and selectivity are needed for multi-component detection in Chinese edible herbs. Atomic absorption spectrometry (AAS) and atomic fluorescence spectrometry (AFS) are widely used in food safety, biological samples, and water quality analysis, and have become indispensable routine analysis techniques for trace elements analysis (Daşbaşı et al. 2016; Gemeda et al. 2021). Due to the interference of the complex matrix of traditional Chinese medicine, it is necessary to develop new methods to meet the multi-element trace detection. Inductively coupled plasma mass spectrometry (ICP-MS), emerging as an element-specific detector, shows the advantages of wide linear range, high sensitivity, and multiple element detection capability. It has the ability to identify both major and trace elements in a sample at a single injection (Jin et al. 2020). Therefore, ICP-MS has been employed in the simultaneous detection of multiple trace elements in a wide range of foods (Milani et al. 2019; Potorti et al. 2020).

*Rhizoma Cibotii*, known as one of the Chinese edible herbs, is getting broad interest all over the world. It is the dried rhizome of *Cibotium barometz* (Linn.) J. Sm., which has a variety of therapeutic effects for human health. However, there is a lack of research on trace elements in *Rhizoma Cibotii*. Therefore, the aim of the recent study was first to determine the occurrence and contamination levels of eight race elements, namely, Cr, Ni, Cu, Zn, As, Se, Cd, and Pb in *Rhizoma Cibotii* by ICP-MS. Then, the estimated daily intake and potential health risks were performed by a combination of the contamination levels with the consumption data of *Rhizoma Cibotii* products.

### Materials and methods

#### Reagents and chemicals

A multi-element standard solution, with concentrations of 20.00 μg/mL Cr, Ni, and Cu; 50.00 μg/mL for Zn; 10.00 μg/mL for As and Pb; and 5.00 μg/mL for Se and Cd, was purchased from Inorganic Ventures (Virginia, USA). A mixed internal standard solution (Sc, Rh, In, Bi) with an individual concentration of 1000 μg/mL was provided by the National Center for Analysis and Testing of Non-ferrous Metals and Electronic Materials (Beijing, China). In this study, the reagents used in this method are of superior purity unless otherwise specified. Nitric acid was obtained from Merc (Darmstadt, Germany). Ultrapure water (18.2 MΩ * cm, 25 °C) was produced by a Milli-Q purification water apparatus (Millipore Co., USA). ICP-MS tuning solution was purchased from Thermo Fisher Scientific (Bremen, Germany).

#### Sample preparation and analysis

In this study, 58 samples were collected from 11 sites (Provinces of Fujian, Guangxi, Jiangxi, Sichuan, Yunnan, Chongqing, Hunan, Hubei, Guangdong, Guizhou, and Anhui) from June to October 2018. The quantitative analysis of trace elements was performed based on the previously reported method (Jiao et al. 2018). Briefly, the samples were digested using nitric acid in Teflon bombs. The obtained test solutions were determined by ICP-MS (Thermo iCAPQ, Bremen, Germany). The working standard solutions, with concentrations ranging from 50 to 4000 ng/mL for Pb and Zn; from 1 to 400 ng/mL for Cr, Cd, Ni, and Cu; and from 0.1 to 50 ng/mL for As and Se, were prepared from suitable dilutions of the stock solutions, which were analyzed in triplicate and the average was used.
Method validation

The data acquisition and analysis were processed with the Otegra™ Intelligent Scientific Data Solution™ software. The internal standard method was used for quantification. The method was validated by linearity, the limit of detection (LOD, S/N > 3) and quantification (LOQ, S/N > 10), accuracy, and precision. Accuracy and precision were evaluated by recovery experiments at two different spiking levels (100 and 1000 ng/mL for Pb and Zn. The inter-day precision was accomplished by repeating experiments consecutively for three working days. In addition, the instrument method validation was tested by the Component Analysis Reference Materials of Ginseng (GBW10027, GSB-18).

Risk assessment for human health

The dietary exposure assessment to trace elements from Rhizoma Cibotii consumption was investigated by the estimated daily intake (EDI), which was calculated using the following Equation:

\[ EDI = \frac{(C \times IR)}{BW} \]  
(1)

where \( C \) represents the mean element concentration in the Rhizoma Cibotii samples (mg/kg); \( IR \) is the daily ingestion amount of Rhizoma Cibotii for adults. \( BW \) is the mean human body weight (60 kg for adults) (Meng et al. 2022).

The human risk assessment associated with Rhizoma Cibotii consumption was characterized by the hazard quotient (HQ), which was calculated according to the equation:

\[ HQ = \frac{EDI}{RfD} \]  
(2)

where RfD is the reference dose (mg/kg/day) set by the US Environmental Protection Agency. The RfD values for Cr, Ni, Cu, Zn, As, Se, Cd, and Pb elements were 0.003, 0.020, 0.040, 0.300, 0.0003, 0.005, 0.001, and 0.004 mg/kg bw/day, respectively. The hazard risk can be divided into five levels according to the HQ value: not significant (HQ ≤ 1), low (1 < HQ < 9.9), moderate (10 < HQ < 19.9), high (20 < HQ < 99), and serious (≥100) (Jiao et al. 2018).

Statistical analysis

The data were statistically analyzed by Excel and SPSS 16.0. One-way ANOVA and Pearson’s correlation coefficient were used for analyzing the detection results, and \( P < 0.05 \) was considered a significant difference.

Results and discussion

Method validation

The validation parameter of the ICP-MS method has been listed in Table 1. Good linearity was obtained for each trace element with correlation coefficients \( (r^2) \) higher than 0.999. The values of LODs and LOQs were 0.003–0.006 mg/kg and 0.010–0.020 mg/kg, respectively. Accuracy was expressed as the recoveries, which ranged from 88.7 to 103%. The intra- and inter-day precision were expressed as the relative standard deviations (RSDs), which ranged from 2.75 to 6.21%, and ranged from 3.29 to 7.95%, respectively. The method validation parameters were similar to the study about multi-elements determination in Alpinia oxyphylla and Morinda officinalis by ICP-MS (Zhao et al. 2016). Obviously, the obtained results demonstrated that the proposed method was suitable for the determination of trace elements in Rhizoma Cibotii products.

Concentrations of trace elements in Rhizoma Cibotii

The concentrations of eight trace elements in the 58 Rhizoma Cibotii samples were shown in Table 2. Briefly, Cr,

| Table 1 | Validation parameters of ICP-MS method for the detection of multi-elements |
|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|
| Element | \( r^2 \) | LOD (mg/kg) | LOQ (mg/kg) | Level 1a |
|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|
| Cr | 0.9996 | 0.005 | 0.015 | Recovery (%) | Intra-RSD (%) | Inter-RSD (%) |
| Ni | 0.9997 | 0.003 | 0.010 | 102 | 4.22 | 5.37 |
| Cu | 0.9998 | 0.005 | 0.015 | 96.7 | 5.38 | 5.72 |
| Zn | 0.9991 | 0.006 | 0.020 | 95.8 | 4.33 | 4.98 |
| As | 0.9996 | 0.003 | 0.010 | 88.7 | 5.48 | 4.96 |
| Se | 0.9992 | 0.006 | 0.020 | 95.7 | 3.19 | 4.36 |
| Cd | 0.9994 | 0.003 | 0.010 | 89.2 | 2.75 | 3.59 |
| Pb | 0.9993 | 0.003 | 0.010 | 96.6 | 4.89 | 5.23 |

Level 1a: 100 ng/mL for Pb and Zn; 10 ng/mL for Cr, Cd, Ni and Cu; 1 ng/mL for As and Se; Level 2b: 1000 ng/mL for Pb and Zn; 100 ng/mL for Cr, Cd, Ni and Cu; 10 ng/mL for As and Se

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\( (1) \) EDI = \( (C \times IR)/BW \) 

\( (2) \) HQ = \( EDI/RfD \) 

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Table 3 Mean concentrations of trace elements in different kinds of Rhizoma Cibotii products

| Mean concentrations (mg/kg) | Elements |
|-----------------------------|---------|
|                             | Cr      | Ni      | Cu      | Zn      | As      | Se      | Cd      | Pb      |
| Sand-scorch Rhizoma Cibotii (n = 43) | 2.03    | 1.85    | 3.55    | 18.85   | 0.06    | 0.13    | 0.10    | 8.33    |
| Raw Rhizoma Cibotii (n = 15)    | 1.62    | 1.46    | 3.40    | 12.95   | 0.04    | 0.33    | 0.07    | 8.99    |
| P-value                      | 1.92    | 1.75    | 3.51    | 17.32   | 0.05    | 0.18    | 0.09    | 8.50    |
medicinal herb samples from Turkey was 2.59 mg/kg, which was similar to the value in the present study (Tokalıoğlu 2012). Moreover, the Pharmacopoeia of China has set the permissible limit of 2.0 mg/kg for Cr in herbal medicine. Therefore, the concentrations in 23 *Rhizoma Cibotii* products (39.7%) exceeded the limit, indicating more attention should be given to control the contamination of Cr.

Ni is considered as an essential trace element. However, at higher concentrations, it can propose as a carcinogen affecting the lung and nasal cavities (Alhusban et al. 2019; Kohzadi et al. 2019). In this study, the concentrations of Ni were in the range of 0.37-3.92 mg/kg and the mean level was 1.75 mg/kg. Potortì et al. (2020) reported that the concentrations of Ni in different types of spices and herbs collected from Italy and Tunisia were varied from 0.165 mg/kg (detected in Laurel) to 0.834 mg/kg (detected in Thyme), which was lower than the results here.

The concentrations of Se were in the range of <0.02–4.25 mg/kg with the mean level of 0.18 mg/kg. Except that the concentrations in the two products were 4.12 and 4.25 mg/kg, the results in the other 56 products were in the range of <0.02–0.08 mg/kg. These values were largely in agreement with the Se results in the medicinal and aromatic plants studied by Ozkutlu et al. (2011), which ranged between 0.01 and 1.13 mg/kg in the analyzed plants. Sheded et al. (2006) found the Se concentrations ranged from 0.058 to 0.600 mg/kg in seven medicinal plants in Egypt. Se is an essential trace element. However, excessive intake of Se can also cause poisoning, which indicated that Se is essential only in a narrow concentration range (Ju et al. 2018).

The range of Cd concentrations in 58 *Rhizoma Cibotii* products was <0.01–2.65 mg/kg, and the mean concentration was 0.09 mg/kg. Cd has been classified as a human carcinogen by the International Agency for Research on Cancer (IARC) (Jiao et al. 2018). More pieces of evidence have indicated that Cd poisoning would lead to many diseases including osteopenia, anemia, and kidney failure (a disease called Itai-itai) (Kohzadi et al. 2019). The permissible limit for Cd in medicinal herb set by China, the WHO, and the USA National Sanitation Foundation International (NSFI) were 0.3 mg/kg (Li et al. 2018). Here, two samples exceeded the permissible limit, which should arouse more attention to the health of people.

The range of As levels among the total 58 *Rhizoma Cibotii* products was <0.01–0.19 mg/kg, and the mean concentration was 0.06 mg/kg. Compared with the concentrations of As in Epimedium sample (ranged from 0.16 to 2.34 mg/kg), the data in this work was much lower (Yang et al. 2017). Besides, the results were also lower than those in Mediterranean edible herbs (ranged from 1.35 to 3.25 mg/kg) (Volpe et al. 2015). As was a toxicity element, which has been classified as carcinogenic to humans by IARC (Alhusban et al. 2019). According to the permissible limit of 5 mg/kg set by WHO, the concentrations in all analyzed samples were below the limit (Alhusban et al. 2019).

### Correlation analysis of trace elements in *Rhizoma Cibotii*

In this study, Pearson’s correlation analysis was used to explore the relationship between 8 trace elements in *Rhizoma Cibotii*. As results showed in Table 4, there were significant positive correlations in Cr-As (*r* = 0.278, *P* < 0.05), Cu-Ni (*r* = 0.329, *P* < 0.05), Se-Cu (*r* = 0.265, *P* < 0.05), Zn-Cd (*r* = 0.997, *P* < 0.01), and As-Cd (*r* = 0.272, *P* < 0.05). Trace elements are active center components of enzymes and play an important role in catalytic biochemical reactions. Combined with the contamination levels and correlativity, this study focused on the analysis of zinc and cadmium.

The content of Zn was highest in the study, and was significant correlated with Cd. Zn is an important dietary trace element which involved in regulating metal ion homeostasis in different cells (Kim and Lee 2021).

Due to interactions between the elements, excessive Zn intake can affect the absorption of other elements. Increased Zn intake could upregulate the metallothionein synthesis to prevent further zinc absorption (Baltaci et al. 2018).

| Elements | Cr | Ni | Cu | Zn | As | Se | Cd | Pb |
|----------|----|----|----|----|----|----|----|----|
| Cr       | 1  |    |    |    |    |    |    |    |
| Ni       | 0.204 | 1  |    |    |    |    |    |    |
| Cu       | 0.025 | 0.329*| 1  |    |    |    |    |    |
| Zn       | −0.065 | −0.019 | 0.190 | 1  |    |    |    |    |
| As       | 0.278*| 0.231 | −0.052 | 0.252 | 1  |    |    |    |
| Se       | 0.003 | 0.120 | 0.265* | −0.019 | 0.096 | 1  |    |    |
| Cd       | −0.067 | −0.045 | 0.165 | 0.997**| 0.272*| 0.003 | 1  |    |
| Pb       | −0.024 | 0.071 | −0.028 | 0.289*| 0.135| −0.088 | 0.278 | 1  |

**Significant correlation at the 0.01 level (two-sided test)
*Significant correlation at the 0.05 level (two-sided test)
Table 5 Exposure assessment of heavy metals in Rhizoma Cibotii products

| Values      | Elements | Cr  | Ni  | Cu  | Zn  | As  | Se  | Cd  | Pb  |
|-------------|----------|-----|-----|-----|-----|-----|-----|-----|-----|
| EDI (μg/kg/d) |          | 0.38| 0.35| 0.70| 3.46| 0.012| 0.036| 0.018| 1.70 |
| HQ          |          | 0.13| 0.018| 0.018| 0.012| 0.040| 0.0072| 0.018| 0.43 |

Also, high Zn intake could induce systemic effects of the gut microbiota, including systemic inflammation, autism spectrum disorder, and obesity (Skalny et al. 2021). Cd is one of the heavy metals with strong toxicity, which is also a serious environmental pollutant. Cd and Zn ions have similar electronic configurations and can bind to the same proteins. Dietary Zn can inhibit the accumulation of Cd and reduce the toxicity of cadmium (Yu et al. 2021). Specific transporters of Cd are limited in plants, and it could enter plants only with the aid of general ion transporters or channel proteins when the abundance of valence cations is low (Zhou et al. 2020). However, our study still found a strong relationship between Zn and Cd. Considering the long cultivation period of Chinese medicinal herbs, this indicates that Rhizoma Cibotii have been contaminated by Cd and there is a trend of continuous contamination. And excessive Zn and Cd in Rhizoma Cibotii could be transferred to human body with the intake of medicine, causing potential health risks. It is necessary to continuously monitor the level of trace elements in traditional Chinese medicinal materials and ensure the quality and safety of traditional Chinese medicinal products.

Risk assessment of trace elements for human health

This study estimated the intake of trace elements through the ingestion of Rhizoma Cibotii. Use the maximum consumption value of Rhizoma Cibotii based on the Chinese Pharmacopoeia to provide the “worst-case” (12 g/day) to estimate daily intake (Liu et al. 2018). As shown in Table 5, the highest estimated daily heavy metal intake was Zn (3.46 μg/kg bw/day), followed by Pb (1.70 μg/kg bw/day), Cu (0.70 μg/kg bw/day), Cr (0.38 μg/kg bw/day), Ni (0.35 μg/kg bw/day), Se (0.036 μg/kg bw/day), Cd (0.018 μg/kg bw/day), and As (0.012 μg/kg bw/day). HQs of all trace elements were below 1, indicating that in this study, Rhizoma Cibotii may not pose a health risk to the human body due to consumption. The results were similar to those of previous study (Shen et al. 2017), which reported that EDI of Cu, Zn, Pb, and Cd was within the tolerable range during Cortex Moutan (Chinese edible herbs) consumption. Nan et al. (2020) reported that the mean daily intake level and target hazard quotient calculated by the total concentration of Pb in Paeoniae Radix Rubra and Astragali Radixwas over safety standards, suggesting taking these Chinese edible herbs may pose potential health risks.

Conclusions

In conclusion, the occurrence and quantitative analysis of eight trace elements was investigated in 58 Rhizoma Cibotii samples. Based on determined results, dietary exposure was evaluated by EDI and potential health risks were assessed by HQ. The results showed that the contamination of trace elements in Rhizoma Cibotii caused low health risks, but additional and/or interactive effects might occur due to exposure to multiple contaminants. Notably, Zn was the most abundant trace element in the Rhizoma Cibotii, but its mean level was lower than permissible limit. However, Correlation analysis results showed a positive correlation between Zn and Cd, which means Zn may promote the absorption of Cd through synergistic action in plants. Therefore, the analysis of eight elements provides a new perspective for the health risk assessments and monitor during the Chinese edible herbs planting period and application period. Nevertheless, since the study found the relationship among elements and accessed the health risks, a more accurate analysis of the element pollution in Chinese edible herbs needs to take into account the absorption and metabolism of plants. In future, it is necessary to monitor the trend of trace elements in Rhizoma Cibotii, which will be useful for ingredient control and human health protection.

Supplementary Information  The online version contains supplementary material available at https://doi.org/10.1007/s11356-023-30576-8.

Author contribution Zhijie Chu: experiment, data analysis, writing-original draft. Nannan Zhu: data analysis, writing-revised manuscript. Lijun Shao: collected samples, validation, data analysis. Hongxia Xu: collected samples, experiment, writing-original draft. Jin Li: collected samples, data analysis. Xiaolin Wang: experiment, data analysis. Yanni Jiao: software, writing-original draft. Dafeng Jiang: funding acquisition, ethology, writing-reviewing, and editing. Peimin Yang: methodology, writing-reviewing, and editing.

Funding This work was supported by the Science and the Technology Development Program of Traditional Chinese Medicine of Shandong Province (Grant number 2017-331 182) and the Science and Technology Development Program of the Health Commission of Shandong Province (Grant number: 202112061045).

Data availability Not applicable.

Declarations

Ethics approval Not applicable.

Consent to participate Not applicable.
Consent for publication The authors confirm that the manuscript described has not been published before, it is not under consideration for publication elsewhere, and the publication has been approved by all co-author and the responsible authorities at the institution where the work was carried out.

Conflict of interest The authors declare no competing interests.

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