Analysis and evaluation of heavy metal pollution in agricultural soils in six cities of Hunan Province, China

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Abstract. Environmental problems caused by the exploitation of mineral resources are worsening, especially the heavy metal pollution in soil. Food safety caused by heavy metal pollution in soil has become the focus of increasing attention. Sampling points were selected in Hunan Province. By sampling the soil and rice point by point, the total concentration and the concentration in rice were monitored. The results showed that the mean concentration of Cd, Cu, Hg, Pb and Zn were 1.31 mg/kg, 45.3 mg/kg, 0.61 mg/kg, 49.15 mg/kg and 133.3 mg/kg. The soils were contaminated in some parts of the 85 investigated sites in Hunan Province. Also, the analysis results showed that the average of PI (pollution index) descended in the order of Cd (2.58) > Hg (1.05) > Cu (0.52) > Zn (0.51) > Pb (0.41). The NIPI values of the research area were 28.59, belong to the high-level pollution, which mainly caused by Cd. The pollution ratio of soil samples were 100.0% in Zhuzhou-Xiangtan and Changning, 94.1%, 90.9% and 88.2% in Changde, Yiyang and Yongzhou. According to the potential ecological risk evaluation result, there was a medium level risk in the investigation area of Hunan and the potential ecological risk mainly caused by Cd.

1. Introduction

In recent years, with the continuous development of mining and smelting industry, the pollution of heavy metals in the ecological environment around the mining area, especially in the soil compartment, has become a prominent problem of environmental pollution [1,2]. Tailings, waste-water, smoke and dust from heavy metal smelting have a serious impact on the surrounding soil. Heavy metal pollution in soil has potential ecological hazards and has become an important factor affecting the safety of the ecosystem [3].

Ecological risk assessment (ERA) is a process of collecting, organizing, and analyzing environmental data to estimate the risk or probability of undesired effects on organisms, populations, or ecosystems caused by various stressors associated with human activities. The basic principles of ecological risk assessment are described in numerous papers [4]. There are many kinds of research methods and evaluation models proposed, among which Nemerow index method, potential ecological risk index method, fuzzy comprehensive evaluation method, enrichment factor method, geoaccumulation index method and other evaluation methods are most used [5]. These evaluation
methods have their own advantages and disadvantages, and the scope of application is also different [6]. The single factor index method and Nemerow comprehensive pollution index method are widely used [7] and are the basis for other environmental quality indexes, environmental quality classification and comprehensive evaluation [8]. The Potential Ecological Risk Index (PERI) proposed by Hakanson is one of the most widely used and internationally influential methods in evaluating the environmental impact of soil (sediment) pollutants [9].

There are 20 million hectares of agricultural land polluted by heavy metals in our country, and the content of heavy metals exceeds the standard in 12 million tons of grain every year [10]. Hunan is one of China's most important rice-producing areas and has been reported to have various contaminations across the province [11]. Hunan is located in the middle reaches of the Yangtze River, and were run through by Xiangjiang river from north to south which was seriously polluted. It is particularly significant to evaluate the potential ecological risk and predict the pollution trend of heavy metal in soil around this area in order to carry out a targeted control or prevention measures. Although a number of articles have been published on soil contamination in Hunan province, which were mainly based on risk assessment in specific regions, and there were few reports on large-scale watersheds, few studies could be found in literature about comparing pollution levels between different regions.

This paper mainly take research in 6 cities which located through Hunan Province from north to south along the Xiangjiang river as the research object, investigates and studies the characteristics of heavy metals in the soil and rice in the modified region and evaluates their pollution status, So as to provide scientific reference basis for the safe use and ecological restoration on the soil in Hunan Province and its future development and utilization.

2. Materials and methods

2.1. Sampling sites

![Figure 1. Sketch map showing sample location.](image)
85 sampling sites were selected in Hunan province through Hunan Province from north to south along
the Xiangjiang river (Figure 1), where the heavy metal contamination have been identified by a
historical study conducted during 1978-1986[12]. The results obtained from the historical study were
also reported in this study for comparison purpose.

2.1.1. Sampling. Investigation were carried out in 6 cities of Hunan Province, they were Yongzhou,
Changning, Changde, Yiyang, Zhuzhou and Xiangtan. All samples were collected from the upper
horizon (0-20 cm). A 50×50 m sampling area was selected at each sample site. All soil samples were
spread out on a piece of Kraft paper (80×110 cm) in an air-drying room, in a layer with a thickness of
2 cm. After removing plant leaves, crushed stone and so on, the samples were dried naturally.
Following grinding and passing through a 0.15 mm screen, the samples were prepared for the total and
the available content of heavy metal elements, and pH analysis.

2.1.2. Rice sampling. Rice plants were harvested from each soil sample position and were manually
threshed to separate grains. Then air dried to constant weights and processed according to “The
Testing Methods of Rice Qualities” published by the Chinese Ministry of Agriculture (NY147-88)
[13]. Husk from the rice grains was removed by using a laboratory de-husker (OHYA-25, Japan), and
the brown rice was polished with a rice polishing machine (CPC 96-3, China) until the cortex was
removed from the brown rice. The polished rice samples were ground to make powder in a ball mill
(JXFM110, China)

2.2. Sample analysis

2.2.1. Analysis of the total content of heavy metal in soil. For determination of total Cd, Cu, Hg, Pb
and Zn in soil, portions of each 0.20 g of soil were digested with a mix of 5 mL of HNO₃, 1 mL of
HClO₄, 1 mL of HF, and processed according to “Soil-quality-Determination of lead, cadmium-
Graphite furnace atomic absorption spectrophotometry” (GB/T 17141-1997) [14]. The concentrations
of Cd, Cu, Hg, Pb and Zn were determined using inductively coupled plasma-mass spectrometry (ICP-
MS, Agilent, 7700x) following a standard procedure. Total Pb in soil was analyzed using a X-ray
fluorescence spectrometry [11].

2.2.2. Analysis of the available content of heavy metal in soil. The air-dried soil samples of 5 g passed
through 2 mm pore size sieve were placed in a 100 mL cone bottle, added 25 mL DTPA extractor with
a pipette. The suspensions were shaken for 2 h (200 rpm) at 25 °C and then centrifuged at 8000 rpm
for 10 min and then filtered through 0.45 µm filter paper.

2.2.3. Analysis of the heavy metal content in rice. For rice samples, 0.20 g was digested with 7 mL of
HNO₃. After cooling, resultant solutions were diluted to 50 mL using distilled water to 50 mL. The
concentrations of Cd, Cu, Hg, Pb and Zn in the filtrate were determined using ICP-MS (Agilent, 7500a)
following the standard procedure.

2.2.4. Analysis of soil pH. The air-dried soil sample was filtered through a 0.15 mm sieve, put 10 g
sample into a 25 mL beaker, 10 mL of distilled water was added, and the mixture was allowed to stand
for 30 min. The pH value of the suspension was determined with a corrected pH meter (METTLER-
TOLEDO, S220).

2.3. Data analysis
SPSS software was used for one-tailed chi-square tests and correlation analyses. The least significant
difference in the data was computed at a significance level of 95%.
2.4. Quality assurance and quality control
The same procedure without samples was used as control and three replications were conducted for each sample. Quality assurance and quality control (QA/QC) for Cd in soil and rice grain was estimated by determining Cd contents in the standard reference materials, soil GBW07405 (GSS-5, GSS-16 and GSS-28) and rice GBW(E)100377 respectively, approved by General Administration of Quality Supervision, Inspection and Quarantine of the People’s Republic of China (AQSIQ) , with a recovery rate in line with the requirements.

2.5. Methods of heavy-metal pollution assessment
To assess the degree of heavy-metal contamination, pollution index (PI) for each metal and Nemerow integrated pollution index (NIPI) [15] for the five heavy metals were calculated for each sample. The PI was defined as follows:

\[ PI = \frac{C_i}{S_i} \]  

where \( C_i \) is the measured concentration of each metal (Cd, Cu, Hg, Pb and Zn) in this study, \( S_i \) is the screening value of pollution risk of heavy metal i in soil. In this study, the heavy metal pollution status of the paddy soil was evaluated by using the screening value of agricultural land soil pollution risk specified in "Soil Environmental Quality Agricultural Land Pollution Risk Control Standard" (GB 15618-2018) [16] (Table 1). The PI of each metal is classified as non-pollution (PI < 1), low level of pollution (1 ≤ PI < 2), moderate level of pollution (2 ≤ PI < 3), high level of pollution (3 ≤ PI < 5) and very strong level of pollution (PI>5). The NIPI of the five metals for each sample was defined as follows:

\[ NIPI = \sqrt{\frac{PI_{ave}^2 + PI_{max}^2}{2}} \]  

where PI_{max} is the maximum PI value of each heavy metal and PI_{ave} is the mean PI value of each heavy metal. The NIPI is classified as non-pollution (NIPI ≤ 0.7), warning line of pollution (0.7 < NIPI ≤ 1), low level of pollution (1 < NIPI ≤ 2), moderate level of pollution (2 < NIPI ≤ 3) and high level of pollution (NIPI > 3) [15].

The Potential Ecological Risk Index (PERI) method was proposed by Hakanson (1980) to evaluate the potential ecological risk of heavy metals. This method comprehensively considers the synergy, toxic level, concentration of the heavy metals and ecological sensitivity of heavy metals [17]. PERI is formed by three basic modules: the degree of contamination (\( C_D \)), toxic-response factor (\( T_R \)) and potential ecological risk factor (\( E_R \)). According to this method, the potential ecological risk index of a single element (\( E_R^i \)) and comprehensive potential ecological risk index (RI) can be calculated via the following equations:

\[ C_D^i = \frac{C_D^i}{C_T^i} \]  

\[ E_R^i = T_R^i \times C_D^i \]  

\[ RI = \sum_{i=1}^{m} E_R^i \]  

Where \( C_D^i \) is the measured concentration of heavy metal in each sampling point; \( C_R^i \) is the reference value for which the screening value of each heavy metal in soil (GB15618-2018) is used; \( C_D^i \) is the pollution of a single element factor; \( E_R^i \) is the potential ecological risk index of a single element; RI is a comprehensive potential ecological risk index; and \( T_R^i \) is the biological toxic factor of a single element, which is determined as Zn=1, Cu=Pb=5, Cd=30, Hg=40 [18,19].

Table 1. Classification standard of potential ecological risk of heavy metals in soils.

| Pollution degree | Slight | Medium | High | Very high | Extremely high |
|------------------|--------|--------|------|-----------|----------------|
| \( E_R^i \)      | \( E_R^i < 40 \) | \( 40 \leq E_R^i < 80 \) | \( 80 \leq E_R^i < 160 \) | \( 160 \leq E_R^i < 320 \) | \( E_R^i > 320 \) |
| RI               | \( EI < 50 \) | \( 50 \leq EI < 300 \) | \( 200 \leq EI < 600 \) | \( 600 \leq EI < 1200 \) | \( EI > 1200 \) |
Table 2. Soil environmental quality risk standard for soil contamination of agricultural land (mg/kg).

| pH    | Screening value | Control value |
|-------|-----------------|---------------|
|       | ≤5.5 | 5.5~6.5 | 6.5~7.5 | >7.5 | ≤5.5 | 5.5~6.5 | 6.5~7.5 | >7.5 |
| Cd    | 0.30  | 0.40   | 0.60   | 0.80  | 1.5  | 2.0     | 3.0     | 4.0  |
| Pb    | 80    | 100    | 140    | 240   | 400  | 500     | 700     | 1000 |
| Zn    | 200   | 200    | 250    | 300   | -    | -       | -       | -    |
| Hg    | 0.50  | 0.50   | 0.60   | 1.0   | 2.0  | 2.5     | 4.0     | 6.0  |
| Cu    | 50    | 50     | 100    | 100   | -    | -       | -       | -    |

3. Results and discussion

3.1. Analysis of soil background value and heavy metal content

The pH value was between 4.63 and 7.86; the average value of pH in the study area was 6.3. The mean concentration of Cd, Cu, Hg, Pb and Zn were 1.31 mg/kg, 45.3 mg/kg, 0.61 mg/kg, 49.15 mg/kg and 133.3 mg/kg; respectively. Compared with the “Environmental Quality Standard for Soils in China published by National Environmental Protection Agency of China (GB15618-2018)”, The mean concentration of Cu, Zn and Pb in this area were lower than the screening value (Table 2), and the mean concentrations of Cd and Hg were higher than the screening value but lower than the control value. However, Cu, Zn, Pb, Cd and Hg exceeded the standard in some samples, and all higher than background values in the study area, which indicated that the soil was contaminated in some parts of the 85 investigated sites in Hunan Province (Table 3).

Table 3. Contents and historical background values of heavy metal elements in soil (mg/kg)

| Element | N | Min | Max  | Median | Mean content | SD | 95% confidence interval | Background value |
|---------|---|-----|------|--------|--------------|----|------------------------|-----------------|
|         |   |     |      |        |              |    | Upper limit            | Lower limit      |
| Cu      | 85| 15.9| 993.4| 30.3   | 45.3         | 104.9 | 39.3                  | 32.0            | 27.3            |
| Zn      | 85| 47.8| 1680.8| 104.8  | 133.3        | 176.9 | 174.9                 | 107.9           | 94.4            |
| Pb      | 84| 0.0 | 288.5| 39.3   | 49.15        | 35.12 | 57.3                  | 42.2            | 29.7            |
| Cd      | 85| 0.19| 24.25| 0.64   | 1.31         | 2.77  | 2.01                  | 0.85            | 0.13            |
| Hg      | 59| 0.01| 5.04 | 0.28   | 0.61         | 0.20  | 0.83                  | 0.42            | 0.12            |

3.2. Heavy-metal pollution assessment

Based on the monitoring data of soil quality in the study area, a quantitative analysis of heavy metal pollution in soil was conducted using the method of PI and NIPI. The PI values of Pb, Zn and Cu in soil ranged from 0.11 to 1.07, 0.24 to 1.40 and 0.20 to 1.55; respectively, indicating that the soil was non-pollution or low-level pollution by Pb, Zn and Cu. The PI of Cd and Hg ranged from 0.48 to 40.42 and 0.01 to 5.04; respectively, indicating that the contamination level of soil was between non-pollution and very strong pollution level. The analysis results showed that the average of PI descended in the order of Cd (2.58) > Hg (1.05) > Cu (0.52) > Zn (0.51) > Pb (0.41) (Table 4). The NIPI values in soil was 28.59, the soil was high-level pollution. The concentration of Cd in one site was very high, which was 24.25mg/kg, but the concentration of the other 84 sites were all lower than 7.23mg/kg. If the maximum value is eliminated, The soil was still high-level pollution but NIPI was obviously reduced. This result was in agreement with the findings of water pollution of this area, Wu Jia got the research that in this area the water environment has reached a lower risk level in some parts of Hunan province [20].
Table 4. Results of pollution index (PI), Nemerow integrated pollution index (NIPI).

|        | Cd     | Pb     | Zn     | Hg     | Cu     |
|--------|--------|--------|--------|--------|--------|
| Max    | 40.42  | 1.07   | 1.40   | 5.04   | 1.55   |
| Min    | 0.48   | 0.11   | 0.24   | 0.01   | 0.20   |
| Mean   | 2.58   | 0.41   | 0.51   | 1.05   | 0.52   |
| NIPI   |        |        |        |        | 28.59  |

The results of soil pollution index are shown in Table 5. Pb, Zn and Cu in the soil of the investigation area only exceeded the standard at some points and the concentration was relatively low. The single-factor pollution indexes in the areas exceeding the standard all belong to the low level of pollution, indicating that there was no obvious pollution of Pb, Zn and Cu in the investigation area. The concentrations of Cd and Hg were relatively high and there was obvious pollution in the investigation area. There was a high pollution rate of Cd and Hg which was 70.59% and 36.21%; respectively (Table 5). Many studies show that the content of heavy metals in plants is more related to the soil environment and forms of heavy metals [21-24]. At present, researchers have also studied the speciation and bioavailability of heavy metals in soil, showing that Pb, Zn and Cd are easier to migrate than other heavy metals, utilized by plants, and have greater potential ecological risks [12]. The PI value for Cd was the highest, which was probably related to the presence of these heavy metals in the wastewater used for irrigation. Other possible heavy metal sources of vegetable soils include the application of solid waste, chemical fertilizer [25].

Table 5. Results of single factor pollution index evaluation of heavy metals in soil samples.

| Element | Distribution proportion of different pollution levels | Pollution rate (%) |
|---------|-------------------------------------------------------|--------------------|
|         | PI≤1        | 1< PI≤2    | 2<PI≤3    | 3<PI≤5    | PI>5    |                  |
| Cd      | 29.41       | 41.18      | 10.59     | 8.24      | 10.59   | 70.59            |
| Pb      | 97.65       | 2.35       | 0.00      | 0.00      | 0.00    | 2.35             |
| Zn      | 93.98       | 6.02       | 0.00      | 0.00      | 0.00    | 6.02             |
| Hg      | 63.79       | 18.97      | 8.62      | 6.90      | 1.72    | 36.21            |
| Cu      | 96.39       | 3.61       | 0.00      | 0.00      | 0.00    | 3.61             |

3.3. Risk analysis of soil pollution in different sampling areas

The farmland soil samples from different counties and cities in the study area were analyzed, and evaluated by the Nemerow index method. The results are shown in Table 6.

Table 6. Evaluation of heavy metals in soil samples by Nemerow index

| City              | Different NIPI levels | Pollution rate (%) |
|-------------------|-----------------------|--------------------|
|                   | P_I≤0.7               | 0.7<P_I≤1          | 1<P_I≤2           | 2<P_I≤3           | P_I>3          |                  |
| Xiangtan - Zhuzhou| 0.0                   | 37.5               | 43.8               | 6.3               | 12.5           | 100.0            |
| Changde           | 5.9                   | 5.9                | 23.5               | 29.4              | 35.3           | 94.1             |
| Yiyang            | 9.1                   | 9.1                | 63.6               | 9.1               | 9.1            | 90.9             |
| Yongzhou          | 11.8                  | 47.1               | 35.3               | 0.0               | 5.9            | 88.2             |
| Changning         | 0.0                   | 20.0               | 33.3               | 20.0              | 26.7           | 100.0            |

*The area of Xiangtan and Zhuzhou is small, These two counties were treated as a region in this paper
According to the survey results in Table 6, the risk ratio of soil samples collected in Xiangtan-Zhuzhou and Changning was 100.0%. Contaminated soil samples in Changde, Yiyang and Yongzhou accounted for 94.1%, 90.9% and 88.2% of the total samples. This result was in agreement with Xiong yuebin’s opinion that the Changsha-Zhuzhou-Xiangtan region is located in the downstream of the Yangtze River, Agricultural land is extensively polluted by heavy metals [25]. The proportion of moderate and high level pollution in each city were different, from high to low was Changde (64.7%), Changning (46.7%), Xiangtan-Zhuzhou (18.8%), Yiyang (18.2%) and Yongzhou (5.9%). It can be seen that all investigation areas were polluted by heavy metals, Xiangtan-Zhuzhou and Changning were the most polluted cities (Table 6).

3.4. Assessment of potential ecological risk

The coefficient of variation for each element sample was between 6.10% and 27.46%. The variation coefficient of Cd was the highest in five heavy metals, which indicates that the distribution of Cd in the soil was not uniform, and it may be significantly disturbed by human activities [24]. The potential ecological risk index value of Pb, Zn, Cu and Hg were less than 40, indicating that there was a low potential ecological risk for these 4 heavy metals. Cd was less than 80, belonging to medium potential ecological risk (Table 7). It indicates that there was a medium ecological risk in the investigation area and the potential ecological risk mainly caused by Cd. From the evaluation result of PI and PERI, the same conclusion is that Cd is the major local pollution, the pollution level is moderate, the risk level is medium. There are different concerns based on the two evaluation methods, but the overall trend was similar.

Table 7. Evaluation results of the potential ecological risk of heavy metals.

| Potential ecological risk index $E^l_R$ | Cd | Pb | Zn | Hg | Cu |
|----------------------------------------|----|----|----|----|----|
| Min(mg/kg)                             | 14.3 | 0.55 | 0.24 | 0.46 | 0.98 |
| Max(mg/kg)                             | 1212.5 | 5.37 | 1.38 | 165.38 | 5.86 |
| Eve(mg/kg)                             | 77.2 | 2.01 | 0.49 | 39.23 | 2.46 |
| Sd(mg/kg)                              | 21.2 | 0.14 | 0.03 | 5.79 | 0.15 |
| CV(%)                                  | 27.46 | 6.97 | 6.12 | 14.76 | 6.10 |
| Risk level                             | Medium | Low | Low | Low | Medium |

4. Conclusions

(1) According to the concentration of Cu, Zn, Pb, Cd and Hg, some samples were higher than the background values in the study area, which indicated that the soil was contaminated in some parts of the 85 investigated sites in 6 cities of Hunan Province.

(2) The results of PI indicated that the soil was moderate polluted by Cd, low level polluted by Hg, non-polluted by Pb, Zn and Cu. The pollution index descended in the order of Cd (2.58) > Hg (1.05) > Cu (0.52) > Zn (0.51) > Pb (0.41). The soil in 6 cities of Hunan province was high level polluted base on NIPI, mainly caused by Cd.

(3) The results of potential ecological risk assessment indicated that the potential ecological risk was in the order of ER (Cd) > ER (Hg) > ER (Cu) > ER (Pb) > ER (Zn). Cd was the key heavy metal causing the risk in the 6 cities of Hunan Province. The potential ecological risk level is medium.

(4) Heavy metals concentration exceeded the national standard in the soils of 6 investigation areas in Hunan Province. The risk ratio in the order of Xiangtan-Zhuzhou = Changning > Changde > Yiyang > Yongzhou in term of percentage of area. As a grain producing area, more attention should be paid to the situation of heavy metals in soil and rice in the Xiangjiang river area to ensure the safe use of rice.
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