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Heavy Metal Contamination in Soils from a Major Planting Base of Winter Jujube in the Yellow River Delta, China

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Abstract: Surface soils form a major planting base of winter jujube in China were collected and detected for six heavy metals including Co, Ni, Cu, Zn, Cd, and Pb. The concentrations of Co, Ni, Cu, Zn, Cd, and Pb were 27.6 ± 6.0, 57.9 ± 12.8, 67.1 ± 10.3, 102.6 ± 23.4, 0.24 ± 0.07, and 25.1 ±5.9 mg/kg, respectively, showing an order of Zn > Cu > Ni > Co > Pb > Cd. The contents of the investigated metals were frequently observed higher than their related background values, suggesting that extra metal inputs occurred. Levels of all elements were below the associated risk screening values of agricultural soil in China, indicating healthy planting conditions for the winter jujube cultivation. Nemerow comprehensive pollution indexes of the metals in all the sampling stations were lower than 0.7, revealing a non-pollution status of the soils. Geo-accumulation indexes suggested that Zn and Pb caused no pollution, and Co, Ni, Cu, and Cd seemed to result in slight pollution. Co, Ni, Zn, Cd, and Pb had similar sources, which might be related to some natural processes and the use of fertilizers. Extra Cu might be mainly from the use of copper-containing pesticides. Based on our observations, the soils from the planting base of winter jujube in the Yellow River Delta were safe for the cultivation of winter jujube, and the rational utilization of pesticide and fertilizer were proposed to control the new inputs of heavy metals.

Keywords: heavy metals; soil; winter jujube; Yellow River Delta

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1. Introduction

Soil is an important resource for human survival, whose quality determines the quality of agricultural products and directly affects people's health [1]. During the past several decades, the overall situation of the soil environment in China was not optimistic [2]. Soil pollution in China is mainly caused by a high background value of soil environment, industrial and mining activities, and agricultural production [3]. In the long run, considering the current agricultural production mode, heavy metals tend to be the key threats affecting the quality of farmland soils in China [4].

Heavy metals, known as a type of harmful substances, will accumulate in the soil after entering this environmental matrix and will be absorbed by varieties of crops through the root system, thus leading to some human health problems [5–7]. Discharge of industrial wastes and traffic exhaust, unreasonable use of agricultural fertilizers and pesticides, and placing of solid wastes aggravate the heavy metal pollution in soil [8–11]. The monitoring and source analysis of heavy metal pollution in soils, especially in agricultural land, is of great significance for people to master the current situation, assess the potential risks, and carry out the prevention and control of heavy metal pollution. Evaluation and traceability work on heavy metal pollution in soil have been frequently conducted around the world [12–15]. During the tree fruit production, heavy metals might be released into the soil through several processes, such as the application of fertilizer and pesticide [8].

Winter jujube (*Zizyphus jujuba* Mill. cv. Dongzao), a local specialty fruit in China, is delicious and nutritious [16]. The fruit of winter jujube contains about 20 amino acids, such as aspartic acid, threonine, and serine, with a total content of 9.8 mg/kg. The contents of protein, total flavonoids, niacin, dietary fiber, total sugar, riboflavin, carotene, and thiamine in the fruit could reach 1.65%, 0.26%, 8.7 mg/kg, 2.3%, 17.3%, 2.2 mg/kg, 1.1 mg/kg, and 0.1 mg/kg, respectively, and the content of ascorbic acid is 70-times that of apple and 100-times that of pear [17]. Binzhou City in Shandong Province is one of the most important planting bases of winter jujube, with planting areas of about 70,000 hm^2 [18]. Several investigations have reported the heavy metal residues in winter jujube fruits [19–21]. However, information on the heavy metal pollution in the soil planting winter jujube are scarce. Under a background of pursuing ecological protection and high-quality development in the Yellow River Delta region, it is of importance to reveal the pollution status, sources, and potential ecological risks of heavy metals in the soils of the winter jujube planting base.

The objectives of the present study were to (1) determine the levels of heavy metals in the soils from orchards planting winter jujube in Binzhou City; (2) evaluate the pollution status and potential ecological risks of metals in the soil; and (3) analyze the possible sources of these trace elements. Our observations will provide a scientific basis for the safe planting of winter jujube and the prevention and control of soil environmental risk.

2. Materials and Methods

2.1. Study Area and Sampling

The study area is located in Binzhou City, Shandong Province, in China (Figure 1). Geographically, it was in the hinterland of the Yellow River Delta, with a continental monsoon climate. In October 2020, 17 surface soil samples from different orchards were collected using a soil auger. Each sample was mixed by five random sub-samples in a 50 m × 50 m area. The samples were stored in a clean PE package and stored at −20 °C.

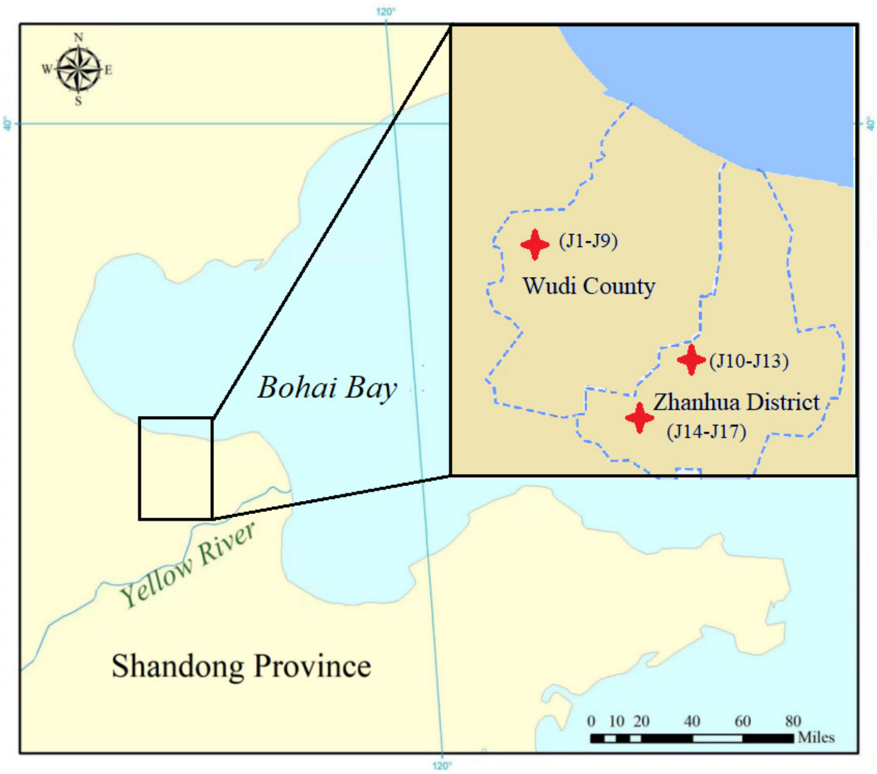

Figure 1. Sampling area in the Yellow River Delta.

2.2. Heavy Metal Determination

In the lab, the soil samples were dried in a vacuum freeze dryer for about 72 h. The dried samples were ground and passed through a 0.5-mm sieve and then microwave digested using an HNO_3-HCl-HF system. After finishing the digestion procedure, samples were heated on an electric hot plate to remove the acid. Afterwards, the sample was diluted by adding 5% HNO_3. Finally, the concentration of heavy metals was measured by an inductively coupled plasma mass spectrometer (ICP-MS). For each batch of experimental samples, 1 program blank and 3 blank samples were set. The recoveries of the reference material (ERM-S-510204) were above 75%.

2.3. Evaluation of the Metal Pollution

The contents of heavy metals were compared with the soil geochemical background values of Binzhou City [22] and risk screening values of agricultural land soil in China (GB 15618-2018) to analyze the pollution status of heavy metals. The pollution degree and potential ecological hazard of heavy metals in the soil were evaluated by the Nemerow comprehensive pollution index [23], geoaccumulation index [24], and potential ecological hazard index [25]. A correlation analysis and principal component analysis of heavy metal content were conducted to analyze the sources of heavy metals in the soil.

2.3.1. Nemerow Comprehensive Pollution Index

The Nemerow comprehensive index method can obtain a comprehensive pollution index of various pollutants through a single factor pollution index (P_i), so as to comprehensively evaluate the pollution degree of all pollutants. P_i could be described as $P_{ij} = C_{ij}/C_{is}$, where P_{ij} is the single pollution index of element i in sampling point j; C_{ij} is the concentration of element i in sampling point j, and C_{is} is the risk screening value of element i for agricultural land soil in China. The formula for the Nemerow comprehensive index is as follows:

$$P_j = \sqrt{(Pjmax^2 + Pjave^2)/2} \tag{1}$$

where P_j is the Nemerow comprehensive pollution index at point j; P_{jmax} is the maximum value of the single pollution index of all elements at point j; P_{jave} is the average value of the single pollution index of all elements at point j. The evaluation criterion of the Nemerow comprehensive index is shown in Table 1.

Table 1. Classification of metal pollution degree based on the Nemerow comprehensive pollution index and geo-accumulation index.

Grade	p	Pollution Degree	Grade	I_{geo}	Pollution Degree
I	≤0.7	No pollution	I	≤0	No pollution
II	0.7–1	Slight pollution	II	0–1	Slight pollution
III	1–2	Moderate pollution	III	1–2	Moderate pollution
IV	2–3	Severe pollution	IV	2–3	Moderate severepollution
V	>3	Extremely Severe pollution	V	3–4	Severe pollution
			VI	4–5	Relatively severe pollution
			VII	>5	Extremely severe pollution

2.3.2. Geo-Accumulation Index

The geo-accumulation index(I_{geo}) has been widely used to evaluate the impact of heavy metals on the soil [26,27]. The formula is as follows:

$$I_{geo} = \log_2\left[\frac{C_i}{1.5 \times B_i}\right] \tag{2}$$

where C_i is the measured concentration of element i; B_i is reference value of element i. According to this parameter, soil pollution of heavy metals could be divided into 7 grades (Table 1).

2.3.3. Potential Ecological Risk Index

The potential ecological risk index of a single metal ($E_r{}^i$) was calculated according to the following formula:

$$E_r{}^i = T_r{}^i \times (C_s{}^i/C_n{}^i) \quad (3)$$

where $C_s{}^i$ is the detected concentration of element i; $C_n{}^i$ is the reference concentration of element i. $T_r{}^i$ is the toxic response factor of the element i. The values of $T_r{}^i$ for Ni, Cu, Zn, Cd, and Pb were 5, 5, 1, 30, and 5, respectively [28]. The combined hazard of metal elements (RI) is the sum of $E_r{}^i$ of each trace element. The degrees of $E_r{}^i$ and RI are shown in Figure 2.

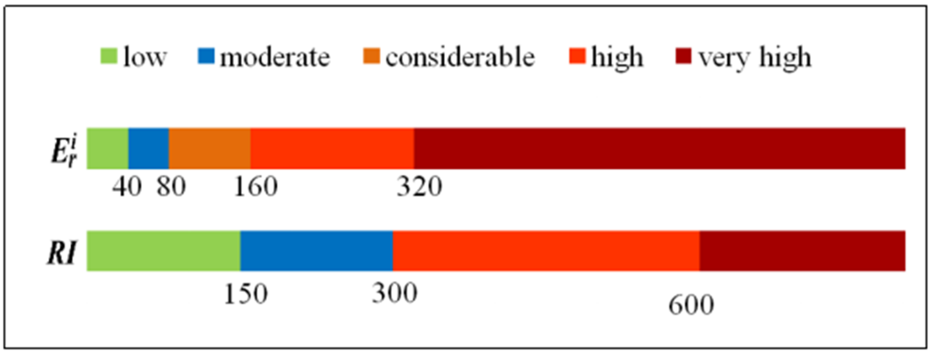

Figure 2. The degrees of $E_r{}^i$ and RI of heavy metals.

3. Results and Discussion

3.1. Analysis of Heavy Metal Content in the Soil

Concentrations of heavy metals in the soils from the winter jujube planting base in Binzhou City are shown in Table 2. The concentrations (mean ± SD) of Co, Ni, Cu, Zn, Cd, and Pb were 27.6 ± 6.0, 57.9 ± 12.8, 67.1 ± 10.3, 102.6 ± 23.4, 0.24 ± 0.07, and 25.1 ±5.9 mg/kg, respectively. Zn was the most abundant element, followed by Cu and Ni. Cd concentrations were 2–3 orders of magnitude lower than those of the other elements. In general, the magnitude orders of metal concentrations were in line with those of the background values for metals in soils of Binzhou City [22]. It could be observed that the average concentrations of the six metals all exceeded the soil background values, suggesting that extra input of heavy metals may occur in this area. Actually, concentrations of Co, Ni, and Cu were higher than related background values [22] at all the sampling points, and Zn, Cd, and Pb were observed with concentrations higher than associated background values at 16, 15, and 10 sampling points, respectively.

Our results were higher than those reported in the topsoil planting winter jujube in Binzhou City obtained about 10 years ago (the average values of Co, Ni, Cu, Zn, Cd, and Pb were 12.17, 30.28, 23.31, 68.36, 0.16, and 22.79 mg/kg, respectively), showing an increasing trend of metal levels [20]. Besides, concentrations of Cu, Zn, Cd, and Pb in the present study were higher than those of farmland soil in Binzhou City in 2011 (average values of Cu, Zn, Cd, and Pb were 23.51, 54.35, 0.057, and 22.28 mg/kg, respectively) [29]. Compared with the metal levels (the average values of Ni, Zn, Cd, and Pb in topsoil in 2014 were 33.4, 97.4, 0.21, and 23.3 mg/kg, respectively) in soils of another planting area of winter jujube in Dagang, Tianjin city [30], our results were observed with higher concentrations of Ni and similar levels of Zn, Cd, and Pb. Therefore, the long-term cultivation of fruit trees might be the major reasons for the enrichment of heavy metals in the soils of the Yellow River Delta.

Table 2. Heavy metal concentrations in soils from winter jujube farms of Binzhou City (mg/kg).

Sample Points	Co	Ni	Cu	Zn	Cd	Pb
J_1	31.1	66.4	71.7	106.9	0.27	29.8
J_2	20.4	34.2	56.2	73.9	0.18	17.7
J_3	35.5	65.2	69.1	126.1	0.38	31.6
J_4	38.6	81.1	70.0	130.6	0.33	32.5
J_5	39.1	83.4	76.2	139.4	0.34	35.0
J_6	30.0	62.4	53.6	96.4	0.27	30.5
J_7	29.6	63.4	71.2	97.4	0.27	28.8
J_8	27.4	57.8	50.2	85.1	0.23	26.4
J_9	28.3	57.9	68.7	111.6	0.23	28.4
J_{10}	26.5	62.4	59.7	99.6	0.18	20.7
J_{11}	26.6	56.7	62.9	83.7	0.13	19.7
J_{12}	22.6	50.0	96.4	101.1	0.22	20.2
J_{13}	20.5	45.8	72.5	143.0	0.25	19.5
J_{14}	19.7	44.3	68.1	69.9	0.13	16.2
J_{15}	26.4	49.9	62.3	120.4	0.21	22.2
J_{16}	25.9	60.5	65.6	90.8	0.23	27.4
J_{17}	21.2	43.3	66.7	67.5	0.16	19.8
Background value	12.2	29.5	23.8	69.1	0.147	22.1
Risk screening value	/	190	200	300	0.6	170
Exceeding standard rate (%)	-	0.0	0.0	0.0	0.0	0.0

In addition, the concentrations of Ni, Cu, Zn, Cd, and Pb in the soils were all lower than related risk screening values (Co was not involved) for agricultural soils in China (GB 15618-2018), indicating healthy planting conditions of the soils. The mean values of Co, Ni, Cu, Zn, Cd, and Pb were 1.1–2.8-times those of the related background values. This showed that the management practices, such as the selection, application amount, and frequency of fertilizer/pesticide, by the farmers in this region might be different.

3.2. Evaluation of Heavy Pollution in the Soils

The Nemerow comprehensive pollution index is a widely-used method to evaluate the pollution status of heavy metals in soils [31]. Values of this parameter in the 17 sampling stations ranged from 0.27 to 0.53, with an average value of 0.37, obviously lower than 0.7, suggesting a non-pollution status of heavy metals in the soils (Table 3).

Table 3. Nemerow comprehensive indexes of soil pollution from heavy metals.

Sample Points	p	Pollution Degree	Sample Points	p	Pollution Degree
J_1	0.40	No pollution	J_{10}	0.31	No pollution
J_2	0.27	No pollution	J_{11}	0.28	No pollution
J_3	0.53	No pollution	J_{12}	0.41	No pollution
J_4	0.48	No pollution	J_{13}	0.41	No pollution
J_5	0.49	No pollution	J_{14}	0.29	No pollution
J_6	0.39	No pollution	J_{15}	0.35	No pollution
J_7	0.39	No pollution	J_{16}	0.35	No pollution
J_8	0.34	No pollution	J_{17}	0.29	No pollution
J_9	0.36	No pollution			

The values of I_{geo} for different metal elements are shown in Figure 3. In detail, the I_{geo} values for Co, Ni, Cu, Zn, Cd, and Pb ranged from 0.11–1.09, −0.37–0.91, 0.49–1.43, −0.62–0.46, −0.76–0.80, and −1.04–0.08, respectively. In general, the average values of I_{geo} of Zn and Pb were less than 0, showing that the soils might have been barely affected by Zn and Pb. The average values of I_{geo} of Co, Ni, Cu, and Cd were between 0 and 1, showing a slight pollution of these above metals. In detail, the proportions of those stations without pollution of Ni, Zn, Cd, and Pb were 11.8%, 58.8%, 41.2%, and 94.1%, respectively, as well as those with slight pollution for Co, Ni, Cu, Zn, Cd, and Pb, which were 88.2%, 88.2%, 76.5%, 41.2%, 58.8% and 5.9%, respectively. It should be noted that the I_{geo} values of Co and Cu were observed to be greater than 1 at two stations and four stations, respectively, indicating moderate pollution.

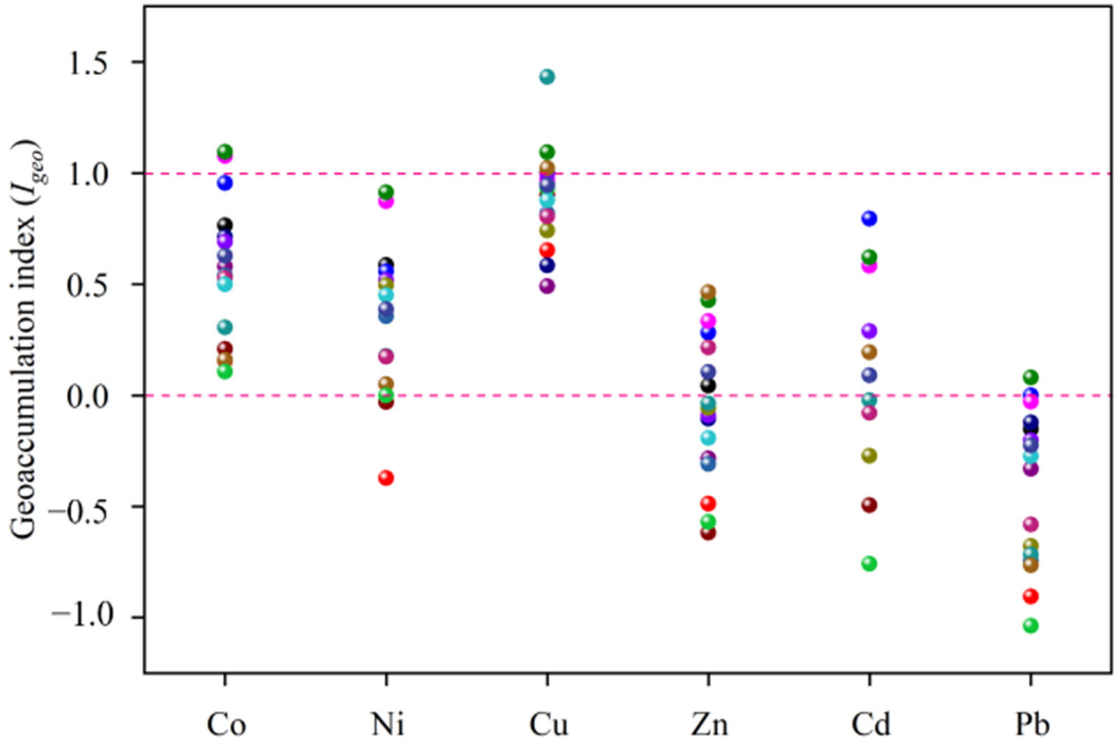

Figure 3. Geoaccumulation indexes for heavy metals in the sampling stations.

Besides, we also calculated the potential ecological hazard index ($E_r{}^i$) based on the risk screening values for soil pollution of agricultural land in China (Table 2.), to judge the pollution effects (ecological risk) of heavy metals in the soils. The results were shown in Table 4. The $E_r{}^i$ values of the Ni, Cu, Zn, Cd, and Pb ranged from 0.90–2.19, 1.25–2.41, 0.23–0.48, 6.52–19.13, and 0.48–1.03, respectively, with a generally decrease as Cd > Cu > Ni > Pb > Zn. All the values of $E_r{}^i$ of a single element in the soil samples were far below 40, demonstrating mild ecological hazards of these metals. As for the ecological hazards of multiple metals, the *RI* values far below 150 indicated extremely low risks in all the sampling stations.

Table 4. Potential ecological hazard index of a single element and comprehensive potential ecological hazard index of heavy metals in the soil.

	$E^i{}_r$					*RI*
	Ni	**Cu**	**Zn**	**Cd**	**Pb**	
Minimum	0.90	1.25	0.23	6.52	0.48	10.10
Maximum	2.19	2.41	0.48	19.13	1.03	23.92
Average	1.52	1.68	0.34	11.84	0.74	16.12
Standard deviation	0.34	0.26	0.08	3.54	0.17	4.06
Potential ecological hazard	Slight	Slight	Slight	Slight	Slight	Slight

3.3. Analyisis of Sources of Heavy Metals in the Soils

The occurrences of heavy metals in soil are mainly affected by natural and human factors, and the similarity of the sources could lead to certain correlations between different elements [32,33]. Correlation analysis between heavy metals is an important basis for inferring the source of heavy metals. The results of a Pearson correlation analysis among heavy metals in the soils are shown in Table 5. A significant positive correlation among Co, Ni, Zn, Cd, and Pb could be observed, and this might suggest that these metals have same sources or behaviors. Conversely, the correlation between Cu and other heavy metals were not significant, indicating that the source or environmental behavior of Cu was different from the other metals.

Table 5. Pearson correlation analysis results of heavy metals in the soil.

	Co	**Ni**	**Cu**	**Zn**	**Cd**	**Pb**
Co	1.00					
Ni	0.941 **	1.00				
Cu	0.060	0.131	1.00			
Zn	0.600 *	0.572 *	0.352	1.00		
Cd	0.819 **	0.720 **	0.220	0.749 **	1.00	
Pb	0.917 **	0.875 **	0.040	0.550 *	0.866 **	1.00

Note: * means significant correlation at $p < 0.05$ level; ** means extremely significant correlation at $p < 0.01$ level.

To further analyze the sources of the six heavy metals, a principal component analysis was performed. Firstly, Kaiser–Meyer–Olkin (KMO) and Bartlett's tests were conducted to test the concentration values of heavy metals. The KMO value was 0.687, and the associated probability of Bartlett's sphericity test was 0.000, meeting the requirements of a principal component analysis. The eigenvalues were greater than 1, and two principal components were screened out. The two principal components could explain 68.4% and 18.5% of the total variance of the variables, reflecting most of the information of the original data.

The factor load distributions of heavy metals in the soils are shown in Figure 4. Co, Ni, Zn, Cd, and Pb had a higher load in the first principal component, and the loads were 0.950, 0.916, 0.766, 0.924, and 0.935, respectively. In addition, as mentioned above, significant positive correlations among Co, Ni, Zn, Cd, and Pb were observed. These findings suggested that the five metals might have the same sources or behaviors. On the one hand, it was related to the natural soil parent material. On the other hand, the contents of the five heavy metals exceeded the background values, and this indicated that they were also greatly affected by human activities. Winter jujube planting could be described as the major sources of these metals. Irrigation could be ignored, because the water for occasional artificial irrigation comes from the Yellow River, whose quality is quite high. Fertilizers were commonly applied during the cultivation of winter jujube, and a local standard (T/ZHDZ 009-2019) was used to provide guidance for the management practices of the fertilizer. The proposed amounts of base fertilizer for decomposed manure, microbial fertilizer, medium and trace element fertilizer, and nitrogen phosphorus potassium compound fertilizer were 45,000–72,000 kg/ha, 2700 kg/ha, 225–450 kg/ha, and 900 kg/ha, respectively. Of course, in reality, the type, frequency, and amount of the fertilizer might vary at different orchards. A great deal of literature reported that different types of fertilizers (especially organic fertilizer) had a high content of heavy metals, for example, Cd and As enrichment in P fertilizers and Zn fertilizers [8,34,35]. Therefore, it is inferred that the extra inputs of the five heavy metals were related to the long-term application of fertilizers containing heavy metals.

Cu had a higher load in the second principal component, with a load of 0.926, which is consistent with the result of the correlation analysis, too. It is concluded that the source of Cu was different from the that of the other heavy metals. Cu is known as the main active ingredient of some pesticides, such as Bordeaux mixture, which is widely used in orchards, leading to the continuous accumulation of copper in the orchard soil [36,37]. Actually, during the cultivation processes of winter jujube, Cu-containing pesticides/fertilizers were generally sprayed in July or August, according to our talking with the farmers. Those Cu in the trees would enter the soils through rainfall. Therefore, Cu in the soils of the study area was mainly related to the use of Cu-containing pesticides. In the process of planting winter jujube, fertilizers and pesticides with a low content of heavy metals should be used, and the dosage and frequency of the fertilizers and pesticides can be adjusted to control the further input of heavy metals.

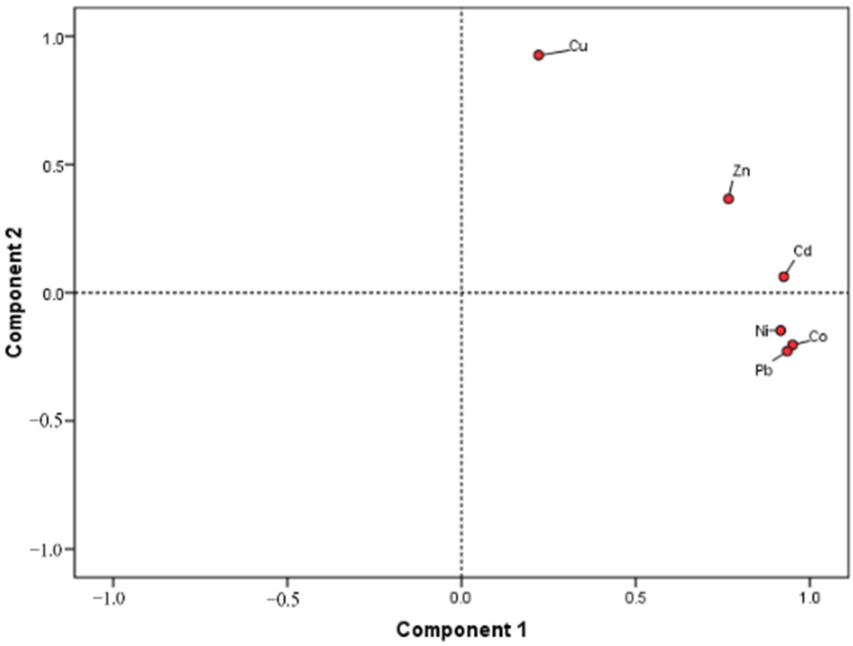

Figure 4. Distribution of heavy metal load in the soils.

4. Conclusions

In the present study, the occurrence of six heavy metals, including Co, Ni, Cu, Zn, Cd, and Pb, in soils of the winter jujube planting base were measured. The metal contents were observed to be exceeding the related soil background values of Binzhou City, showing an enrichment phenomenon. Besides, the metal levels in our study were higher than those in soils planting winter jujube in Binzhou City 10 years ago, presenting an increasing trend. On the other hand, these values were lower than the national risk screening values of metals in agricultural soil, revealing that the healthy/ecological risks caused by these metals were extremely low. The pollution degree and potential ecological risks were comprehensively discussed by calculating several indexes. The Nemerow comprehensive pollution indexes suggested no pollution of these metals. The I_{geo} values of metals revealed a non to slight pollution of Ni, Zn, Cd, and Pb, as well as a slight to moderate pollution of Co and Cu. Both the $E_r{}^i$ and RI values indicated that the ecological risks caused by these metals were extremely low. Co, Ni, Zn, Cd, and Pb might come from the long-term fertilization, and Cu tended to come mainly from the pesticide use during the cultivation process. In general, the soils from the planting base of winter jujube in the Yellow River Delta were safe for the cultivation of jujube. The rational utilization of pesticide and fertilizer could be efficient measurements proposed to control the fresh inputs of heavy metals.

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