

Article

Lime and Phosphate Amendment Can Significantly Reduce Uptake of Cd and Pb by Field-Grown Rice

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Abstract: Agricultural soils are suffering from increasing heavy metal pollution, among which, paddy soil polluted by heavy metals is frequently reported and has elicited great public concern. In this study, we carried out field experiments on paddy soil around a Pb-Zn mine to study amelioration effects of four soil amendments on uptake of Cd and Pb by rice, and to make recommendations for paddy soil heavy metal remediation, particularly for combined pollution of Cd and Pb. The results showed that all the four treatments can significantly reduce the Cd and Pb content in the late rice grain compared with the early rice, among which, the combination amendment of lime and phosphate had the best remediation effects where rice grain Cd content was reduced by 85% and 61%, respectively, for the late rice and the early rice, and by 30% in the late rice grain for Pb. The high reduction effects under the Ca + P treatment might be attributed to increase of soil pH from 5.5 to 6.7. We also found that influence of the Ca + P treatment on rice production was insignificant, while the available Cd and Pb content in soil was reduced by 16.5% and 11.7%, respectively.

Keywords: soil amendments; soil remediation; heavy metal; paddy soil; stabilization

1. Introduction

With the rapid urbanization and industrialization over the past decades in China, a large amount of heavy metals had been released into the environment with much being retained in the soil through atmospheric deposition and irrigation [1,2]. Chinese agricultural soils are suffering from increasing heavy metal pollution, among which paddy soil polluted by heavy metals is frequently reported and has drawn great public concern [3]. For instance, Du et al. (2003) reported that in one prefecture of Hunan Province, 58% of the paddy soils were polluted by Cd ($>0.3 \text{ mg} \cdot \text{kg}^{-1}$), and 60% of the randomly sampled rice grains exceeded $0.2 \text{ mg Cd} \cdot \text{kg}^{-1}$, the national standard [4].

The heavy metal polluted soil poses high risks to ecosystem and to human health through the food chain [5,6]. To eliminate the heavy metals in soil or reduce the concentration in food to a tolerable reference dose, contaminated soil remediation technologies including physical processes, biological processes and chemical processes have been applied to recover land productivity [7]. Currently, physical processes such as excavation and disposal to landfill, thermal desorption and electrokinetic remediation are generally adopted but these methods are less attractive for widespread use because of high cost, manpower and material resources. Although the cost of bioremediation and

phytoremediation is lower than other remediation technologies, they are time-consuming and cannot satisfy the urgency of land development [8–10]. Compared with other remediation technologies, in-situ chemical stabilization/solidification is considered as a promising strategy with numerous advantages such as reduced risk to site workers, low cost and speed of implementation [11]. This method uses soil amendments to change soil pH, increase absorption sites or promote co-precipitation process between heavy metal ions and soil components/amendments to reduce bioavailability of heavy metals, and is gaining increasing prominence in heavy metal contaminated land remediation [12–15].

Many pot and field experiments have demonstrated that soil amendments such as lime, silicon fertilizer and phosphate could reduce rice uptake of heavy metals [16–18]. However, the inhibition effects varied according to the type and dose of soil amendments, experimental conditions and heavy metals. In addition, the current studies mainly focus on indoor pot experiments in the short term, which cannot reflect the real amelioration effects of the complex environmental conditions in the rice field. Therefore, field experiments covering the whole growth period of rice are needed for directing regional paddy soil remediation.

In this research, we selected heavy metal contaminated cropland around a Pb-Zn mine of Guangdong for field experiments to identify the most effective amendments to stabilize Cd and Pb in contaminated paddy soil. The amelioration effects of lime, silicon fertilizer, phosphate and combination of lime and phosphate amendments on field Cd and Pb uptake by the early and late rice were studied to provide information for paddy soil heavy metal remediation, particularly for combined pollution of Cd and Pb.

2. Materials and Methods

2.1. Field Experiment

The field is located 2 km from the mining zone, a typical lead-zinc mine in Guangdong province. The mine had been in operation for about 50 years. The study area has a humid subtropical climate with an annual average temperature of 19.6 °C and rainfall of 1619.6 mm. The number of frost-free days is approximately 305. The field is flat for farming with loam soil. The field experiments were conducted for two continuous rice growth seasons. The first growth season (late rice) was cultivated from July 2013 to November 2013 while the second growth season (early rice) was cultivated from April 2013 to July 2014. Before the rice cultivation, the fields were plowed (deep to 20 cm) and divided into fifteen 12 m² (3 m × 4 m) plots. 0.5 m width aisles were set between plots. Five treatments were designed including control (CK), lime amendment (Ca), phosphate amendment (P), silicon fertilizer amendment (Si) and phosphate plus lime amendment (Ca + P). The designed treatments were randomly assigned to the 15 field plots with three replicates for each treatment. Soil amendments were added once before the field experiment and completely mixed with the topsoil. The addition amount of each amendment was shown in Table 1. The rice type is Guanghui 998, a type of *indica* rice which is widely planted near the field. The rice cultivation and management followed common local practices.

Table 1. Addition amount of each amendment (kg·mu^{−1}).

Treatment	Amendments	Addition
CK	Control	—
Ca	Lime	6000
P	phosphate	165.6
Si	silicon fertilizer	1500
Ca + P	Lime + phosphate	6000 + 165.6

2.2. Sampling and Analysis

Soil samples were collected one day before the late rice plantation and during the growing season to examine the effects of soil amendments on soil pH and bioavailability of Cd and Pb. Soil pH was determined in the 1:2.5 weight vs. volume aqueous soil suspension through potentiometry.

The bioavailability of Cd and Pb was determined based on extraction of $0.1 \text{ mol}\cdot\text{L}^{-1}$ hydrochloric acid at the 1:5 soil/water ratio [19]. Total soil Cd and Pb content was determined based on a $\text{HCl-HNO}_3\text{-HF-HClO}_4$ digestion [15].

Rice tissue samples including roots, plants and grain were collected at the maturity stage, 16 October 2013 for the late rice, 14 July 2014 for the early rice. The rice samples were washed with tap water followed by rinsing with deionized water for 2 to 3 times. After that, samples were heated in 105°C for half an hour and in 80°C until they reached a constant weight. Then the samples were weighed and ground for further analysis.

The contents of Cd and Pb of plant samples were determined by the $\text{HNO}_3\text{-HClO}_4$ wet digestion method [20]. The concentration of Cd and Pb was determined by the graphite furnace atomic absorption spectrophotometer (AA800, Perkin-Elmer, Wellesley, MA, USA). National standard material GBW07604 (GSV-3) (National Standard Material Center, Beijing, China) was referenced to ensure the quality of analysis. The recovery ratio of standards ranged from 79.3% to 112.1%, and 88.3% to 105.8% for heavy metals in soil and rice samples, respectively.

2.3. Data Analysis

Data were processed in Microsoft Office Excel (version 2010, Microsoft, Washington, DC, USA) and statistically analyzed using SPSS (version 17.0, IBM, New York, NY, USA). The figures were drawn by Origin (version 8.1, OriginLab, Northampton, MA, USA). The results of the pot experiment were analyzed by a two-way analysis of variance ($p < 0.05$).

3. Results

3.1. Basic Information and Rice Yields

Table 2 summarizes the basic soil properties of the field. The field is acidic with soil pH ranging from 4.5 to 5.5, and is polluted by Cd and Pb. The averaged soil Cd and Pb concentration were 0.73 and $136 \text{ mg}\cdot\text{kg}^{-1}$, respectively, exceeding the secondary standard of national environmental quality standards for soils, which is $0.3 \text{ mg}\cdot\text{kg}^{-1}$ for Cd and $80 \text{ mg}\cdot\text{kg}^{-1}$ Pb.

Table 2. Selected soil properties of the field ($n = 15$).

	Mean	Standard Deviation
pH	4.80	0.6
SOM ($\text{g}\cdot\text{kg}^{-1}$)	27.62	3.8
CEC ($\text{cmol (+)}\cdot\text{kg}^{-1}$)	8.17	0.9
Cd ($\text{mg}\cdot\text{kg}^{-1}$)	0.76	0.13
Pb ($\text{mg}\cdot\text{kg}^{-1}$)	136	15
Zn ($\text{mg}\cdot\text{kg}^{-1}$)	176	17
As ($\text{mg}\cdot\text{kg}^{-1}$)	18	3.2

Figure 1 illustrates the variation of rice yields under different treatments. The rice yields varied from 6.09 to $8.51 \text{ ton}\cdot\text{ha}^{-1}$, within the medium production level of local paddy rice. For the late rice, yield of all treatments had no significant difference compared with control treatment except for the phosphate treatment ($p < 0.05$), under which the rice production reduced significantly by 28.1%. For the early rice, the phosphate and silicon fertilizer treatment resulted in significant reduction in rice production by 20.1% and 14.8%, respectively, in comparison with the control.

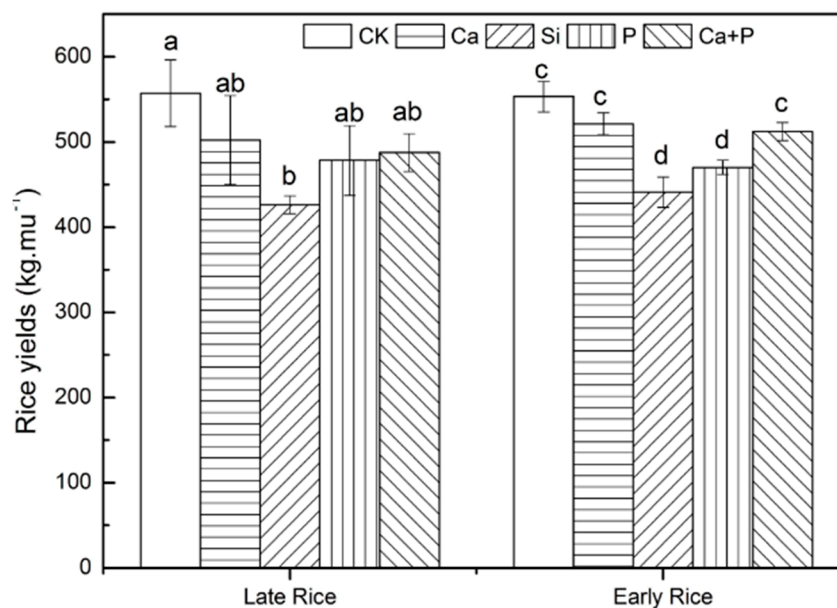


Figure 1. Brown rice yields under different amendments. CK, Ca, P, Si, and Ca + P represents the treatment of: control, lime amendment, phosphate amendment, silicon fertilizer amendment, and phosphate plus lime amendment, respectively. The different lower case letters indicate a significant difference ($p < 0.05$).

3.2. Soil pH and Metal Availability

Figure 2 shows the variation of soil pH under different treatments. All the soil amendments increased the soil pH to some extent. Among them, the influence of Ca + P and Ca treatment on soil pH was significant, and the soil pH was increased from around 5.5 to 6.7. While soil pH varied at the five sampling times, it maintained a relatively stable level during the study period, suggesting that the pH amendment effect could last for a certain period.

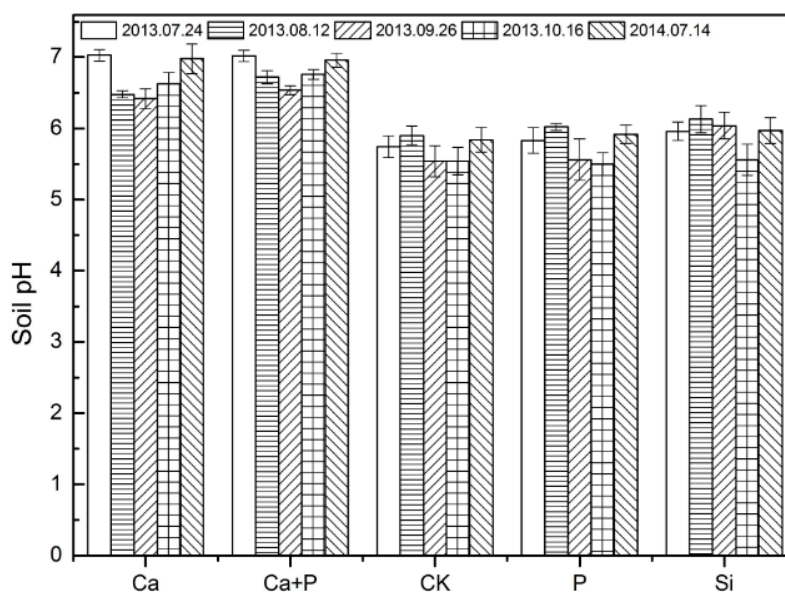


Figure 2. The variation of soil pH under different treatment. CK, Ca, P, Si, and Ca + P represents the treat of: control, lime amendment, phosphate amendment, silicon fertilizer amendment, and phosphate plus lime amendment, respectively. The different lower case letters indicate a significant difference ($p < 0.05$).

Table 3 summarizes the available content of Cd and Pb under different treatment. For the late rice, the available Cd and Pb content in soil was significantly reduced by 16.5% and 11.7%, respectively, under the Ca + P treatment. The other soil amendment treatment can reduce the metal availability to a certain extent, but was insignificant in comparison to the control. Similar to the later rice, the Ca + P treatment also had significant influence on soil metal availability for the early rice such that soil available Cd and Pb content was reduced by 24.8% and 13.9%, respectively, and the influence of other treatment was insignificant.

Table 3. Effect of different treatment on concentration of available heavy metals (mg·kg^{−1}).

Treatment	Late Rice (n = 15)		Early Rice (n = 15)	
	Cd	Pb	Cd	Pb
CK	0.186 ± 0.058 a	45.1 ± 2.41 a	0.173 ± 0.06 a	35.2 ± 3.12 a
Ca	0.169 ± 0.023 a	42.1 ± 2.44 a	0.137 ± 0.012 b	34.2 ± 2.56 a
P	0.182 ± 0.011 a	47.3 ± 0.41 a	0.163 ± 0.015 a	36.5 ± 0.64 a
Si	0.160 ± 0.01 a	42.2 ± 0.429 a	0.158 ± 0.31 a	31.1 ± 1.38 a
Ca + P	0.155 ± 0.06 b	39.8 ± 1.021 b	0.130 ± 0.13 b	30.3 ± 0.98 a

Notes: The same column with the same letter indicates no significant difference between processing ($p < 0.05$). The CK, Ca, P, Si, and Ca + P represents the treat of including control, lime amendment, phosphate amendment, silicon fertilizer amendment, and phosphate plus lime amendment, respectively. The different lower case letters indicate a significant difference ($p < 0.05$).

Table 4 summarizes the correlation between soil pH and available heavy metal contents in soil as well as metal content of rice plant. Increase of soil pH resulted in significant decrease of Cd content in grain, shoots and roots. Some physical and chemical processes including precipitation, absorption, complexation, redox reaction and ion exchange occur with changes of soil pH which can influence the available Cd in soil, and thus reduce rice uptake at higher soil pH [21]. In contrast to Cd, changes of soil pH only had a negative influence on Pb content in roots, suggesting that absorption and translocation mechanisms of Cd and Pb by paddy rice were quite different. Furthermore, changes of soil pH had no significant correlation with available heavy metal contents in soil, and there was also no significant correlation between soil available heavy metal content and metal uptake, except for between available Pb in soil and Pb in roots. These results indicated a complex relationship between soil and plant uptake of heavy metals. Better soil measurement approaches are needed to predict the rice uptake potential of heavy metals. In accordance with the research of Wang et al., available Cd and Pb in soil was found to be highly correlated [22], suggesting that there might be a synergistic function between available Cd and Pb in soil under these amendment treatments.

Table 4. Correlation between soil pH and available heavy metals contents and heavy metal content of different plant tissues (n = 15).

Index	Cd in Rice	Pb in Rice	Available Cd	Available Pb	Cd in Roots	Pb in Roots	Cd in Plants	Pb in Plants
pH	−0.86 **	−0.173	−0.234	−0.401	−0.764 **	−0.526 *	−0.825 **	−0.29
Cd in rice		−0.181	0.388	0.466	0.853 **	0.767 **	0.956 **	0.158
Pb in rice			−0.284	−0.387	−0.221	−0.285	−0.126	−0.475
Available Cd				0.949 **	0.178	0.531 *	0.510	0.60
Available Pb					0.244	0.547 *	0.556 *	0.138
Cd in roots						0.767 **	0.789 **	0.237
Pb in roots							0.714 **	0.121
Cd in plants								0.135

Notes: * Means $p < 0.05$, ** means $p < 0.01$.

3.3. Uptake of Cd and Pb by Rice

As illustrated by Figure 3a, all amendments can significantly reduce the Cd content in brown rice ($p < 0.01$). For the late rice, the food chain transfer risk of Cd was quite high, the Cd concentrations of

rice grain under the control treatment reached $1.75 \text{ mg} \cdot \text{kg}^{-1}$, about 9 times higher than the national food safety standard in China ($0.2 \text{ mg} \cdot \text{kg}^{-1}$). Under the Ca + P and Ca treatments, the Cd content in rice grain was reduced significantly ($p < 0.01$) by 84.6% and 83.9%, respectively, just a little higher than the national food safety standard, suggesting that the Ca + P or Ca treatment is quite efficient in soil Cd risk control. The Si and P treatments could also significantly reduce the Cd uptake by late rice, but the magnitude was much smaller, resulting in a 12.8% and 10.3% decrease of rice Cd content, respectively.

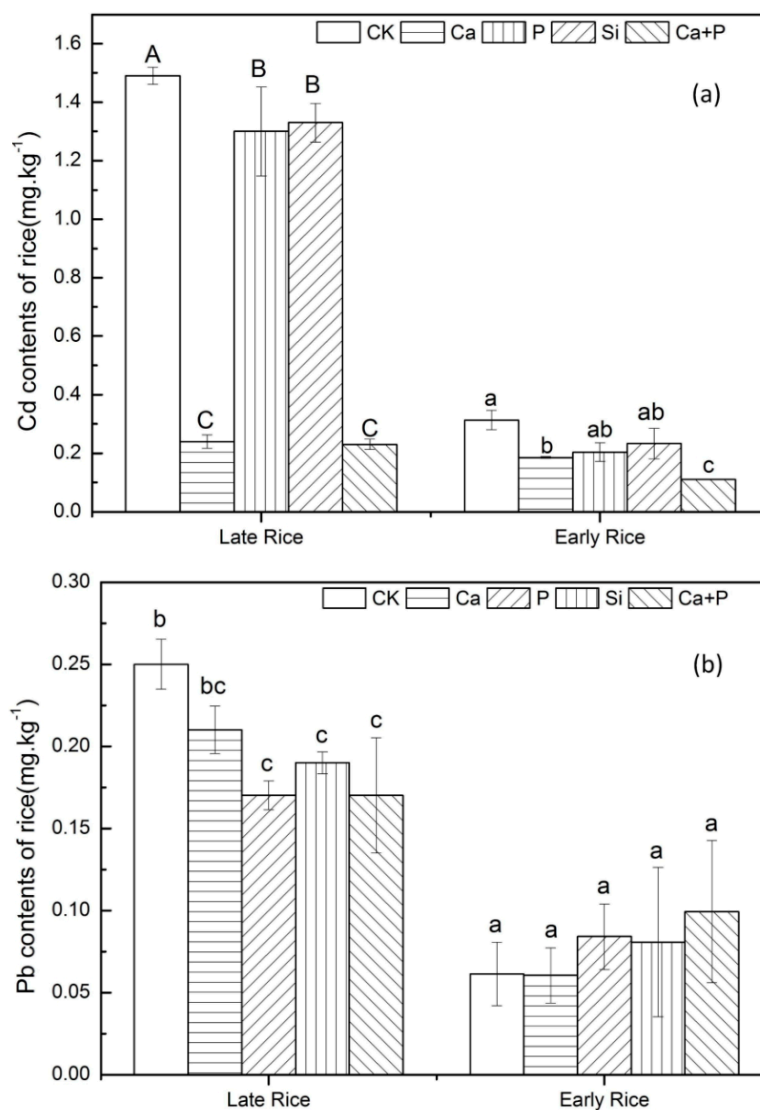


Figure 3. Rice grains Cd (a) and Pb (b) contents under different amendments. The CK, Ca, P, Si, and Ca + P represents the treat of including control, lime amendment, phosphate amendment, silicon fertilizer amendment, and phosphate plus lime amendment, respectively. The different letters indicate a significant difference ($p < 0.05$).

For the early rice, the food chain transfer risk of Cd was much smaller, but the Cd concentration of rice grain under the control treatment ($0.31 \text{ mg} \cdot \text{kg}^{-1}$) was still higher than the national food safety standard. Under the Ca + P and Ca treatments, the rice Cd content was significantly reduced by 61% and 32%, respectively, lower than the food safety standards. The Si and P treatments reduce the Cd uptake by early rice to some extent, but did not reach a significant level, and the rice Cd content was a little higher than the food safety standards.

In comparison with Cd, the food chain transfer risk of Pb was much lower (Figure 3b). The rice grain Pb content under different treatments ranged from 0.17 to $0.25 \text{ mg} \cdot \text{kg}^{-1}$ for the late rice,

and from 0.06 to 0.1 $\text{mg}\cdot\text{kg}^{-1}$ for the early rice. The Ca + P, P and Si treatments significantly reduced the Pb uptake by late rice to below the food safety standards in China ($0.2 \text{ mg}\cdot\text{kg}^{-1}$). For the early rice, the difference among treatments was insignificant.

Overall, our field experiments showed that lime plus phosphate amendment was quite efficient for the combination control of Cd and Pb food transfer risk by rice, and uptake of Cd and Pb by the early rice was much lower than by the late rice.

The four soil amendment treatment not only reduced accumulation of Cd and Pb in brown rice, but also in other organic of rice like roots and plants. As showed by Figure 4, all the four soil amendment treatment can inhibit accumulation of Cd in rice plants and roots. Among them, the influence of Ca + P and Ca treatment was significant, and Cd content in rice plants and roots was reduced by about 88% and 76%, respectively, under these two treatments (Figure 4a). The Ca + P and Ca treatment also resulted in much lower root accumulation of Pb, while the difference among Pb in plants was much smaller and was insignificant (Figure 4b). The average root to grain transfer ratio is about 1/7 for Cd, 1/442 for Pb, suggesting the high Cd translocation ability of rice. Therefore, for the combined pollution of Cd and Pb paddy soil, the risk control and remediation should mainly focus on Cd.

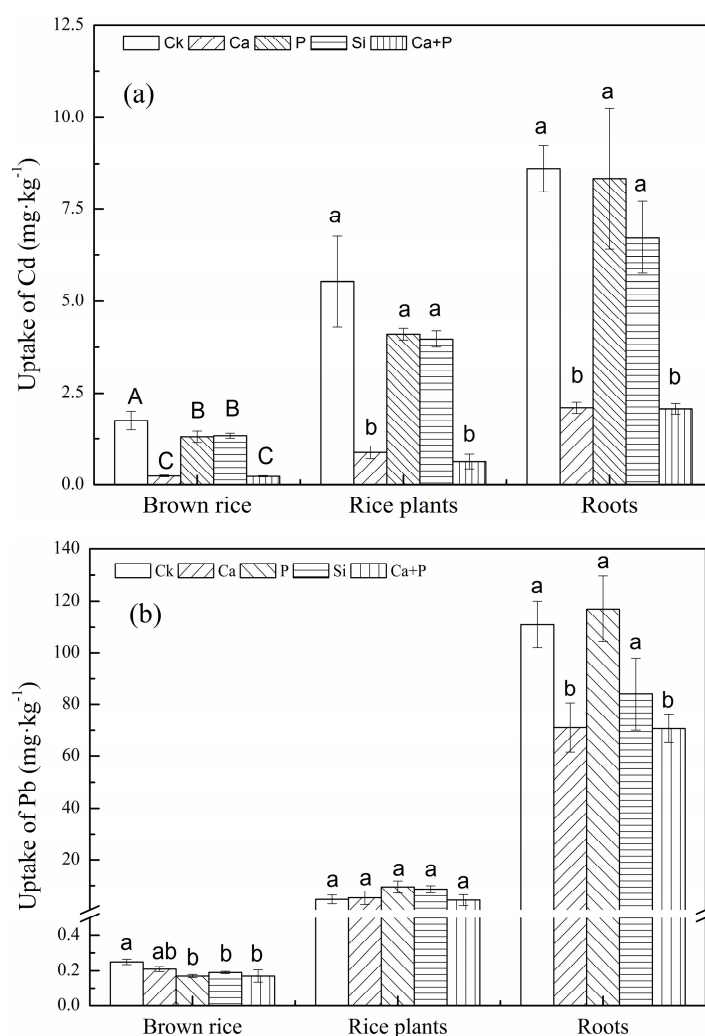


Figure 4. Accumulation of Cd (a) and Pb (b) in different rice tissues. CK, Ca, P, Si, and Ca + P represents the treatment of: control, lime amendment, phosphate amendment, silicon fertilizer amendment, and phosphate plus lime amendment, respectively. The different letters indicate a significant difference ($p < 0.05$).

4. Discussion

Our field experiments illustrated that phosphate and silicon fertilizer could have a significant impact on rice production. With addition of these amendments, soil pH was elevated, which in turn decreased the plant uptake of nutritious elements, thus the rice yield [23]. Furthermore, large amount of phosphate in the soil can inhibit the uptake of Zn to some extent, thus affecting the rice production [24]. It is also possible that due to microenvironment changes associated with these soil amendments such as reactive oxygen species, oxidative stress, and cell membrane lipid peroxidation [25]. Thus, additional or combined amendments may be necessary to assure the normal rice production when using phosphate or silicon fertilizer to stabilize the Cd and Pb in paddy soil.

As illustrated by Table 4, the correlation between soil pH and available heavy metal contents in soil as well as metal content of rice plant was complex. Increase of soil pH resulted in a significant decrease of Cd content in grain, shoots and roots. Some physical and chemical processes including precipitation, absorption, complexation, redox reaction and ion exchange occur with changes of soil pH which can influence the available Cd in soil, thus reduce uptake by rice at higher soil pH [21]. In contrast to Cd, changes of soil pH only had a negative influence on Pb content in roots, suggesting that the absorption and translocation mechanisms of Cd and Pb by paddy rice were quite different. Furthermore, changes of soil pH had no significant correlation with available heavy metal contents in soil, and there was also no significant correlation between soil available heavy metal content and metal uptake, except for between the available Pb in soil and Pb in roots. These results indicated the complex relationship between soil and plant uptake of heavy metals. A better soil measurement approach is needed to predict the uptake potential of heavy metal by rice. In accordance with the research of Wang et al., available Cd and Pb in soil was found to be highly correlated [22], suggesting that there might be a synergistic function between available Cd and Pb in soil under the amendment treatments.

Our field experiments showed that all the four soil amendments could inhibit the absorption of Cd and Pb by paddy rice to some extent. Among them, Ca + P treatment had the highest influence on reduction of both Cd and Pb in the rice grain. Alkaline phosphate can reduce the acidity of the soil through interaction with exchangeable ions in clay or hydroxyl groups of organics. Meanwhile, the co-precipitation reaction between alkaline phosphate materials and calcium compounds can lead to metal oxide precipitation [26], and phosphates and heavy metals can form insoluble phosphates to weaken migration of Cd and Pb in the soil [27]. All these reactions would help to inhibit the uptake of heavy metal by rice. Addition of lime can increase soil pH, thus improve absorption of heavy metal ions by soil. It can also promote the formation of heavy metal hydroxide or carbonate minerals precipitate which is conducive to reduce the bioavailability of heavy metals in soil [22].

In addition, our field experiments also illustrated that the uptake of Cd and Pb by the early rice was much lower than by the late rice, which may be attributed to different field water conditions. During the period of late rice planting, the rain in the whole growth period decreased, while the situation was the opposite for the early rice. Cadmium uptake by rice occurred mostly from the earing stage to maturity stage. During this key period, the early rice had more water than the late rice. It is well known that field water conditions have important influence on the accumulation of Cd in rice [28]. When acidic paddy soil was flooded, the soil redox potential reduced, the soil pH increased, and the bioavailability of the heavy metal would decrease through the competitive absorption and co-precipitation interaction [29]. Therefore, Cd and Pb content in the early rice grain decreased greatly with increased soil moisture during the key metal absorption period. The results indicate that water management is very important for mitigating food transfer of heavy metal by rice.

5. Conclusions

Field experiments were conducted to investigate the influences of four typical stabilization amendments on rice uptake of Cd and Pb grown on paddy soil around a Pb-Zn mine. The results showed that the combination amendment of lime and phosphate can significantly reduce the uptake of Cd and Pb by field-grown rice. The rice grain Cd content reduced from about 9 times higher to just a little higher than the food safety standard, while the Pb content in rice grain was reduced to

below the food safety standard under the Ca + P treatment. Soil analyses showed the Ca + P treatment could increase the soil pH by 1.2 per unit, and reduce the available Cd and Pb content in soil by 16.5% and 11.7%, respectively. In addition, influence of the Ca + P treatment on rice production is negligible. All these facts led us to conclude that the Ca + P treatment could be an efficient approach to control the risks associated with combined pollution of Cd and Pb in paddy soil. The field experiment results also illustrated that transfer of heavy metal from soil to rice was governed by many complex processes. Uptake of Cd and Pb by the later rice was much higher than by the early rice, and absorption and translocation ability of Cd was much higher than that of Pb. Further research is needed to reveal the mechanisms and processes associated with soil amendments to provide recommendations for regional soil heavy metal pollution remediation.

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Conflicts of Interest: The authors declare no conflict of interest.

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