

Research Article

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Study of solidification and stabilization of heavy metals by passivators in heavy metal-contaminated soil

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Abstract: In this study, the indoor constant temperature culture experiment was used to explore the mutual transformation of different forms of heavy metals. Appropriate types of passivating agents were screened, and the optimal addition amount was determined to realize the solidification and stabilization of heavy metals. The results showed that the dissolved copper (Cu), zinc (Zn), and lead (Pb) contents of the zeolite-treated soil decreased to the lowest. They were 219, 819, and 40 g/kg, respectively, which were 31.2, 6.5, and 38.5% lower than no passivating agent added (the control); the dissolved Cu, Zn, and Pb content gradually increased with the extension of time; 5% zeolite (Z4) treatment had the highest average content of Cu, Zn, and Pb in the residue state, respectively, 24, 48, and 19 mg/kg; and at the end of the test, the residual Zn content of Z4 treatment reached 50 mg/kg, which was 72.4% higher than that of the control. A comprehensive analysis of the changes in the dissolved state of the four heavy metals in the soil shows that Cu, Zn, cadmium (Cd), and Pb treated with zeolite have the best effect, followed by sepiolite, and finally palygorskite. Therefore, 5% zeolite can be used for the passivation restoration of heavy metals Cu, Zn, Cd, and Pb in the soil.

Keywords: heavy metals, contaminated soils, solidification and stabilization, passivators, constant temperature culture

1 Introduction

Soil is an important cornerstone for carrying the entire biological cycle; so, the cleanliness and safety of the soil have an immeasurable impact on animals, plants, and humans. With the acceleration of industrialization and urbanization in today's society, human beings have neglected environmental protection while developing their economy rapidly. This makes soil pollution by heavy metals increasingly prominent, especially agricultural soils polluted by heavy metals. Due to the migration of heavy metals in the food chain, they pose a great threat to human health and ecological environment security [1–3]. Only when the soil is contaminated with heavy metals and accumulated to a certain degree, can it be expressed through plant growth and human health. Therefore, once the soil is polluted by heavy metals, it will change the structure, properties, and functions of the soil and be transported to microorganisms and plants through the food chain, causing harm to human health [4–6]. At present, soil heavy metal pollution has become a global problem, especially in developing countries, and China is the largest developing country [7,8]. Therefore, it is imminent to study the large-scale remediation method of heavy metal-contaminated soil.

Heavy metal pollution is an extremely severe type of pollution that is long-lasting, highly toxic, and easy to migrate. Therefore, the cost, cycle, and difficulty of repairing are extremely high [8,9]. Physical repair can take effect quickly, but it requires a huge cost. Phytoremediation has a long-term effect, but its repair time is longer [10]. Therefore, chemical passivation is the most practical method for remediation of soil contaminated by heavy metals. It is relatively fast and can ensure the safe production of crops [11,12]. Chemical passivation remediation refers to the addition of passivators to contaminated soil; passivators convert heavy metals from active forms to stable forms. This is beneficial to reduce the migration and bioavailability of heavy metals and to achieve a method of repairing soil contaminated by heavy metals [13,14]. This repair technology has the

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advantages of rapid repair and simple operation, so it is more suitable for large areas of farmland contaminated by heavy metals [15,16]. At present, the passivator materials that can be used mainly include various minerals, biochar and organic matter, and so on. There are certain differences in their remediation results for different types of pollutants, soil types, and pollution levels [17,18]. Inactivation agents repairing the soil can improve the physical and chemical properties of the soil and supplement nutrients. At the same time, through adsorption, precipitation, and complexation, heavy metals are changed from exchangeable state to organic combined state and residual state. This reduces the mobility and biocombination of heavy metals [19].

There are many types of chemical passivators. The price of the passivator and the possibility of recontamination should be fully considered. Minerals are cheap materials with the best environmental compatibility. In recent years, nonmetallic minerals are often used as passivators for heavy metals in the soil. This research has achieved good results [20,21]. Studies have shown that the exchangeable cadmium (Cd) content in vegetable soil can be reduced by 23.1–41.2%. The maximum reduction of Cd in the edible parts of lettuce, rapeseed, and radish reached 51.8, 47.0, and 24.9%, respectively [22]. Studies have shown that under the repairing effect of sepiolite, the absorption and accumulation of Cd in the edible parts and roots of pakchoi can be significantly reduced [23]. Some scholars have studied the control effects of different passivators on the soil-ryegrass system. It was found that passivators can change the chemical form and biological activity of the heavy metals and reduce the migration and transformation of heavy metals into plants [24,25].

The remediation effect of passivators on heavy metals is restricted by factors such as climate, soil physical and chemical properties, water and fertilizer management, and crop types and varieties. In actual research, passivation materials should be selected according to local conditions [26]. Therefore, it has two objects in this study: (1) To identify three mineral materials that were selected as passivation agents for remediation of heavy metal-contaminated soil. Through constant temperature cultivation, the different forms of heavy metals in the soil at different cultivation times were studied. (2) To discuss the best type and amount of passivator materials needed to change the chemical form and activity of soil heavy metals. It is conducive to the rational and effective use of soil polluted by heavy metals under the condition of ensuring the quality of crops. Moreover, these objectives should be technically written.

2 Experimental materials and methods

2.1 Experimental materials

The test soil was the clay around Xi'an city, Shaanxi Province. The sampling depth was 0–20 cm. The soil samples were air-dried and passed through a 0.149 mm sieve for use. The contents of total copper (Cu), total Cd, total zinc (Zn), and total lead (Pb) in the soil samples were 14, 0, 28, and 16 mg/kg, respectively. The passivating agent is sepiolite, zeolite, and palygorskite. Heavy metal-contaminated soils are expanded by five times in accordance with the second level of soil environmental quality standards (GB 15618-2018), and appropriate heavy metal salts are added to make the total Cu, total Cd, total Zn, and total Pb contents in the soil reach 500, 1.5, 1,250, and 600 mg/kg, respectively.

2.1.1 Preparation of heavy metal compound-contaminated soil

Prepared soil sample of 20 kg was added to a 4 L plastic pot. There are five plastic basins in total. The water content was controlled to 60% of the field water-holding capacity. The soil bulk density is 1.21 g/cm³. The soil samples were cultured in a constant temperature incubator at 25°C for 7 days. According to the second level of soil environmental quality standards (GB 15618-2018), heavy metals in the form of sulfates are added to the soil. The total Cu, total Cd, total Zn, and total Pb contents in the soil are 500, 1.5, 1,250, and 1,500 mg/kg, respectively, and water was added according to 65% of the field water-holding capacity. The soil is contaminated with heavy metals required for the test. The contaminated soil is then subjected to aging treatment. The soil moisture is adjusted by the weighing difference method every 3 days. The soil moisture content is maintained at 65%. After 30 days, a constant temperature incubation test was carried out.

2.2 Experimental method

The constant temperature incubation test was adopted. In the experimental setting, the ratio of passivation agent to air-dried soil sample was 1, 2, 3, and 5%. A mineral

passivator was added to 150 g of heavy metal compound-contaminated soil. The heavy metal compound-contaminated soil after adding the passivator is thoroughly mixed. The mixed sample was placed in a 250 mL plastic bottle. The mouth of the bottle was sealed with plastic wrap, and several small holes were left in the middle of the plastic wrap. The treated soil samples were cultured in a constant temperature incubator at 25°C. During the cultivation process, a weighing method was used to supplement deionized water to 65% of the field water-holding capacity. About 10 g soil samples were collected at 20, 40, and 50 days of culture. After natural air drying, the soil samples were passed through a 0.149 mm sieve for later use. Palygorskite was recorded as P1–P4, Zeolite was recorded as Z1–Z4, and sepiolite was recorded as S1–S4. The control without passivation agent was recorded as the control. There were 13 treatments in total, with three repetitions for each treatment.

The contents of different forms of Cu, Cd, Zn, and Pb in the soil were continuously extracted with 0.1 mol/L acetic acid, 0.1 mol/L hydroxylamine hydrochloride, and 8.8 mol/L H₂O₂. The leaching residue was digested with nitric acid–hydrofluoric acid–perchloric acid. Finally, the dissolved, reducible, and oxidizable leachate and residue states were obtained. The contents of Cd, Cu, Pb, and Zn in the extract and digestion solution were determined by atomic absorption spectrophotometry (Analytical Instruments AG in Jena, Germany. AAS ZEEnit700plus).

3 Results and discussion

3.1 Study the change trend of heavy metal forms in different culture time

At the beginning of the experiment, there was no significant difference in heavy metal content between different treatments. The content of dissolved heavy metals showed a downward trend with time. At the end of the experiment, the dissolved Cu, Zn, and Pb contents of the zeolite treatment reached the lowest. They were 219, 819, and 40 mg/kg, which were 31.2, 6.5, and 38.5% lower than those of the control, respectively. The content of sepiolite in dissolved Cd is the lowest, which is 1.08 mg/kg, followed by the contents of palygorskite and zeolite, which both are 1.09 mg/kg. They were reduced by 10.7% and 9.9% respectively compared to the control. The content of the reduced and oxidized states of the passivator treatment increases first and then decreases with the increase of time. The control showed a continuous decline. The content of heavy metals in the residue of different treatments gradually increased with time; among them, the contents of Cu, Zn, and Pb in the residue of zeolite reached the highest 48, 70, and 41 mg/kg, which were 1.92, 2.41, and 1.86 times that of the control, respectively. However, the content of residual Cd was treated with sepiolite, which was 0.18 mg/kg, which was twice that of the control (Table 1). It shows that the form of heavy metals treated with passivating

Table 1: Changes in the forms content of four heavy metals treated (mg/kg) with different passivates over time

Time	Dissolved state				Reduced state				Oxidized state				Residual state				
	12.1	12.21	1.10	1.21	12.1	12.21	1.10	1.21	12.1	12.21	1.10	1.21	12.1	12.21	1.10	1.21	
CK	Cu	367	357	346	321	214	106	67	58	38	31	27	14	15	20	23	25
	Zn	972	886	874	868	115	162	95	91	58	51	46	35	16	22	26	29
	Cd	1.35	1.31	1.26	1.21	0.10	0.11	0.11	1.09	0.02	0.04	0.02	0.01	0	0	0.06	0.08
	Pb	80	77	69	65	67	127	108	99	54	46	34	30	11	15	17	22
P	Cu	375	346	287	257	82	86	94	88	24	38	25	16	14	15	23	32
	Zn	973	923	865	841	126	134	130	122	41	57	40	27	28	32	43	48
	Cd	1.34	1.26	1.11	1.09	0.12	0.12	0.11	0.10	0.02	0.03	0.03	0.02	0.04	0.08	0.12	0.14
	Pb	84	69	66	63	81	113	109	103	56	69	30	18	14	14	22	36
Z	Cu	371	325	252	219	85	81	87	84	27	38	28	24	15	18	22	48
	Zn	989	912	850	812	117	153	145	132	45	51	43	37	32	38	49	70
	Cd	1.35	1.31	1.19	1.09	0.10	0.13	0.12	1.12	0.03	0.05	0.03	0.02	0.01	0.11	0.13	0.16
	Pb	84	77	61	40	84	101	96	93	61	73	40	19	16	17	25	41
S	Cu	372	318	347	265	81	86	82	880	17	26	28	15	17	19	20	35
	Zn	978	945	868	821	119	150	138	127	32	42	45	27	35	36	37	52
	Cd	1.31	1.29	1.16	1.08	0.10	0.12	0.10	0.10	0.01	0.03	0.02	0.01	0	0.12	0.15	0.18
	Pb	87	81	74	59	90	112	97	91	52	61	42	15	14	16	24	37

Note: The treatments in the table are CK-control without passivation agent, P-palygorskite, S-sepiolite, Z-zeolite, and the values of P, Z, and S represent the average heavy metal content of different proportions.

agent changes to a low-activity oxidizable state and a residue state. The content of heavy metals in the residues of different treatments gradually increased with time. This is consistent with the results of Gao *et al.* [27]. Using montmorillonite to passivate and remediate heavy metal compound-contaminated soil, the weakly acid-extracted Pb, Zn, and Cd in the soil were reduced by 12.0–15.9%, whereas the residue content increased by 62.5–110.1%. The results show that the bioavailability and mobility of these four heavy metals have been reduced. The main reason is that the addition of passivators can convert heavy metals in the soil from the dissolved state to the residue state, and the dissolved heavy metals are finally formed into a fixed residue form through the process of reduction and oxidation. Studies have shown that the types of passivators and soil pH and Eh have a close influence on the occurrence of heavy metals, coordination properties, and charge adsorption. This can change the form of heavy metals in the soil and affect their migration and transformation in the soil environment [28–30]. Therefore, the dissolved heavy metals in the soil treated by the passivator quickly converted to the residue state, resulting in the lower content than the control treatment.

3.2 Study the change trend of dissolved heavy metals in different addition levels

Dissolved heavy metals are heavy metals that are exchange-adsorbed on soil clay minerals and other components, such as iron hydroxide, manganese hydroxide, and humus. This form is most sensitive to changes in the soil environment. It has high activity and is easy to migrate and transform through leaching and leakage of irrigation, rainwater, etc. Eventually, it will seriously pollute the underlying soil, groundwater, and growing plants. It can be seen from Figure 1 that the content of dissolved Cu, Zn, Cd, and Pb

of the control is the largest. After adding different passivators, the content of dissolved heavy metals decreased to varying degrees. This is consistent with the results of existing studies. After adding passivation materials, the form of each heavy metal changes to an oxidizable state with low activity and a residue state. With the prolongation of the passivation time, the content of forms with high bioavailability gradually decreases. It shows that the passivation material added can effectively reduce the mobility and bioavailability of Pb, Cd, Zn, and arsenic [31,32]. In this study, P3 has the lowest Zn content, which is 814 mg/kg, which is lower than the other passivation treatments. When the dissolved heavy metals are treated with sepiolite, the effect of treatment with S3 (3% sepiolite) is the best, of which Cu, Zn, and Pb are 286, 856, and 60 mg/kg, respectively. For zeolite treatment, Z4 (zeolite 5%) performed best, and the dissolved heavy metals Cu, Cd, and Pb are the lowest, which are 269, 0.94, and 55 mg/kg, respectively. Therefore, among the three passivating agents, palygorskite has the best passivation effect on Cu, and 5% zeolite has the best comprehensive passivation effect on heavy metals.

3.3 Study the change trend of residual heavy metal in different addition levels

Residual heavy metals are stable in crystal lattices such as quartz and clay minerals. Its nature is stable. It has little effect on the migration and bioavailability of heavy metals in the soil. Residual heavy metals in the soil will not be absorbed by plants, and they will not easily leak through rain or other means. It presents an extremely stable state. It can be seen from Table 2 that different passivation treatments significantly increased the content of residual heavy metals. The results are consistent with the conclusions of earlier reports [32,33]. In this

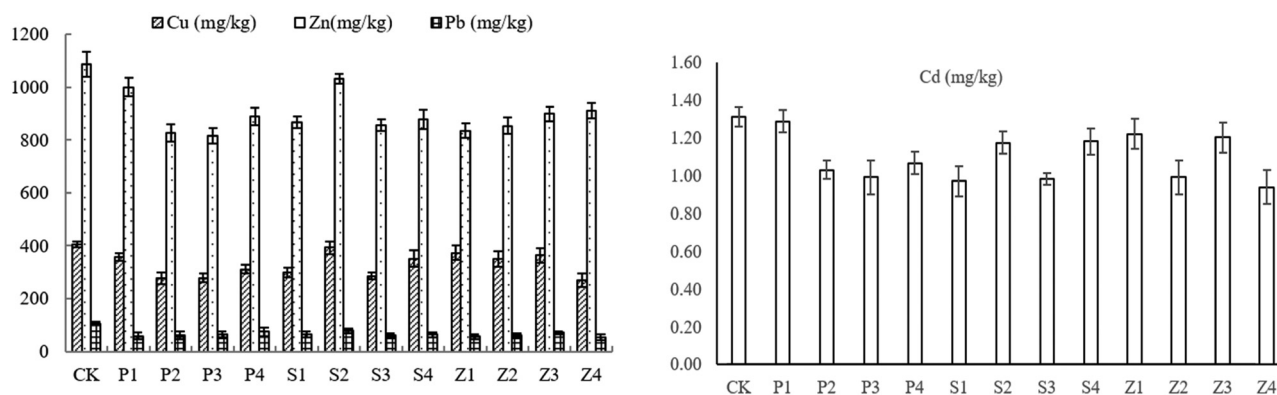


Figure 1: Changes in dissolved heavy metal content in different treatments. Note: The treatments in the figure are CK-control without passivation agent, P1-palygorskite 1%, P2-palygorskite 2%, P3-palygorskite 3%, P4-palygorskite 5%, S1-sepiolite 1%, S2-Sepiolite 2%, S3-Sepiolite 3%, S4-Sepiolite 5%, Z1-Zeolite 1%, Z2-Zeolite 2%, Z3-Zeolite 3%, and Z4-Zeolite 5%.

study, the heavy metal Cd is not easy to be solidified, and the residual Cd of S4, Z4, and S3 is 0.21, 0.18, and 0.17 mg/kg, respectively. They are significantly different from the control ($P < 0.05$). Among them, S4 and Z4 have the best fixation effect. This is consistent with the results of existing studies. The application of sepiolite in Cd-contaminated soil can significantly increase soil pH. It can promote the conversion of Cd in the soil from the exchange state with high activity to the residue state with low activity, thereby effectively reducing its bioavailability and migration ability [34,35]. The maximum Pb content in the residue of P2 treatment is 24 mg/kg. The residual Cu, Zn, and Pb contents of Z4 treatment are the highest, which are 24, 48, and 19 mg/kg, respectively. They are significantly higher than those from other treatments. Therefore, the effect of zeolite treatment is the best, followed by sepiolite. The main reason is that the application of zeolite powder can promote the formation of soil aggregates and increase soil organic matter [36]. In addition, zeolite powder belongs to clay minerals. Due to its large specific surface area and abundant negative charges on the surface, it has strong adsorption and ion-exchange capabilities for metal ions [37].

3.4 Changes in the content of heavy metals in polluted soil at different treatment times

3.4.1 Changes in the content of dissolved heavy metals in contaminated soil

It can be seen from Figure 2 that the dissolved Cu, Zn, Cd, and Pb in the soil showed a downward trend with an increase in the cultivation time under constant temperature

culture conditions. The dissolved heavy metal content of each passivator treatment was basically less than the control. With the increase of the passivation dose in each treatment, the dissolved Cu, Zn, Cd, and Pb contents in the soil showed a downward trend. The research results of Ren [38] showed that the mineral treatment significantly reduced the available Zn, Cd, Cu, and Pb contents of heavy metals in the soil ($P < 0.05$). The reduction rate was 99.1, 91.4, 85.6, and 46.1%. This is consistent with the results of this study. The Cu content of Z4 treatment decreased from 413 to 299 mg/kg. The rate of decline is highest at 27.6%. Second, the Cu content of P2 and S4 treatment decreased from 367 and 377 mg/kg to 278 and 286 mg/kg, respectively. The decline rates were 24.3 and 24.1%. The difference between the two is not significant. There was no significant difference in the dissolved Zn content of the control. The rate of decline is only 0.6%. The content of dissolved Zn in the soil treated with P4 was the lowest at 814 mg/kg, and the rate of decline also reached the maximum at 23.2%. Then it is Z4 processing. The dissolved Zn content decreased from 1077 to 835 mg/kg. The decline rate reached 22.5%, and the difference between P4 and Z4 is not significant. In this study, only the dissolved Zn was reduced by 8.4–23.2%. This is consistent with the study by Liang et al. [39]. The effective state of Zn in the soil was reduced by 23.48–49.61% after the passivation agent treatment. The passivation rate in this study is low, mainly due to shorter passivation time. Research by Zhao [26] showed that the reduction range of soil available heavy metals increased with the extension of the test time. The overall dissolved content of Cd and Pb in the contaminated soil is the control > palygorskite > sepiolite > zeolite. Comprehensive analysis of the changes in the dissolved state of the four elements in the soil shows that Cu, Zn, Cd, and Pb treated with zeolite have the best effect, followed by palygorskite, and finally sepiolite.

Table 2: Effects of different passivators on the concentrations of residual heavy metals (mg/kg) in soil

Treatment	Cu	Zn	Cd	Pb
CK	12 ± 0.54^d	29 ± 1.02^{ef}	0.08 ± 0.01^d	8 ± 0.22^e
P1	12 ± 0.43^d	30 ± 1.15^e	0.09 ± 0.01^d	12 ± 0.45^d
P2	17 ± 0.64^{bc}	41 ± 1.26^{bc}	0.10 ± 0.01^d	16 ± 0.41^c
P3	19 ± 0.93^b	42 ± 1.32^{bc}	0.11 ± 0.01^{cd}	17 ± 0.43^c
P4	25 ± 1.00^a	43 ± 0.89^c	0.15 ± 0.01^{bc}	25 ± 0.54^b
Z1	15 ± 0.25^c	34 ± 1.07^d	0.10 ± 0.01^d	12 ± 0.27^d
Z2	16 ± 0.42^{bc}	33 ± 1.01^{de}	0.12 ± 0.01^{cd}	12 ± 0.32^d
Z3	18 ± 0.66^b	34 ± 1.20^d	0.13 ± 0.01^{cd}	24 ± 0.66^b
Z4	24 ± 1.21^a	48 ± 1.52^a	0.18 ± 0.01^{ab}	19 ± 0.76^c
S1	12 ± 0.27^d	27 ± 0.68^f	0.13 ± 0.01^{cd}	9 ± 0.22^{de}
S2	16 ± 0.48^{bc}	35 ± 1.11^d	0.15 ± 0.01^{bc}	11 ± 0.38^{de}
S3	15 ± 0.39^c	39 ± 1.04^c	0.17 ± 0.01^{ab}	17 ± 0.47^c
S4	23 ± 0.72^a	50 ± 1.30^a	0.21 ± 0.01^a	30 ± 0.90^a

Note: Different lowercase letters in the same column represent significant differences between treatments ($P < 0.05$).

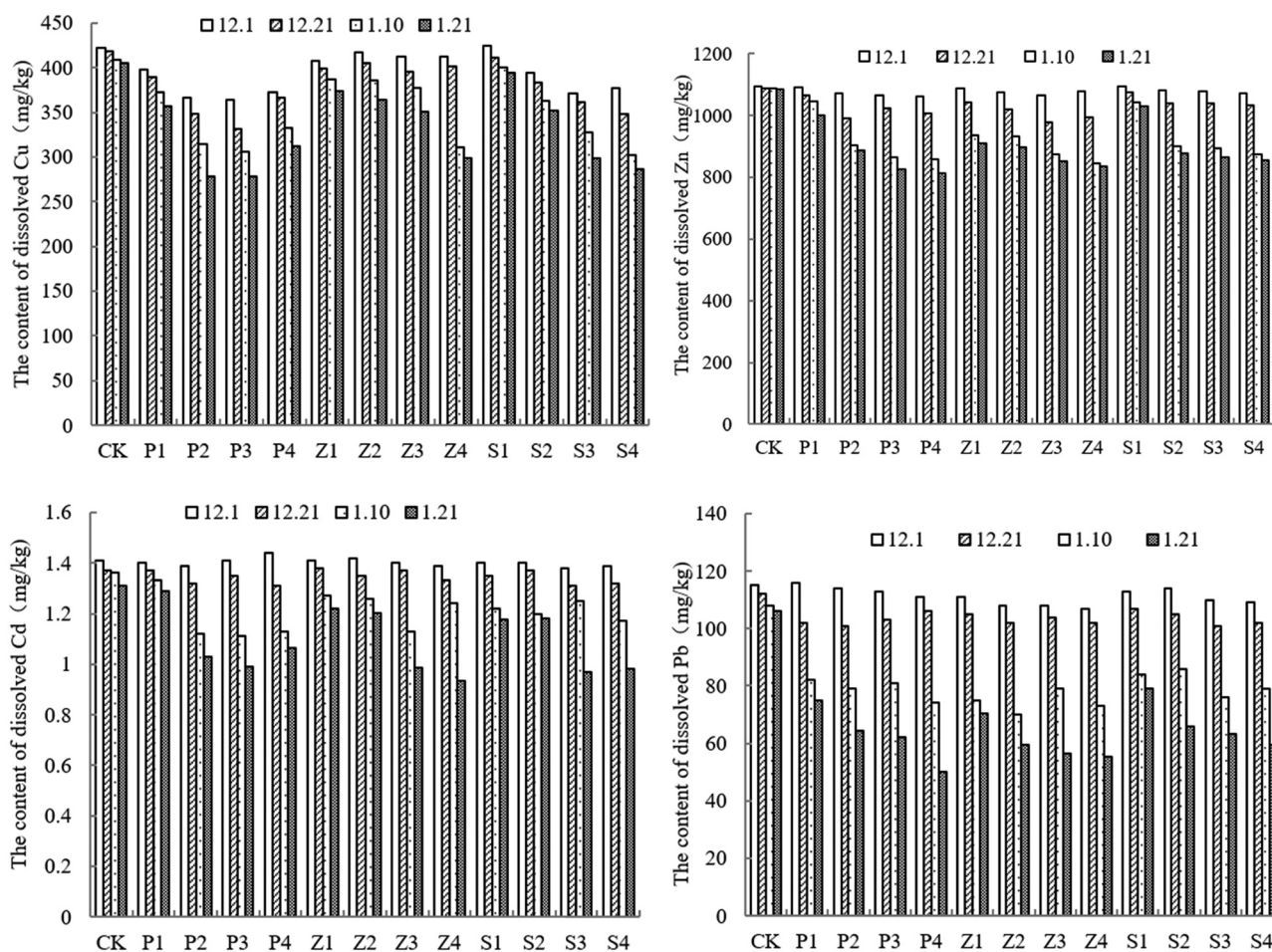


Figure 2: Changes in the content of dissolved heavy metals in the soil at different incubation times.

3.4.2 Changes in residual heavy metal content in contaminated soil

It can be seen from Figure 3 that the residual Cu, Zn, Cd, and Pb in the soil showed an upward trend with an increase in the cultivation time under constant temperature culture conditions. The residual heavy metal content of each treatment is greater than that of the control. With the increase of the passivation dose in each treatment, the content of residual Cu, Zn, Cd, and Pb in the soil showed an upward trend. The overall Cu content in the soil residue is palygorskite > sepiolite > zeolite > the control. Among them, the residual Cu content of the control is 12 mg/kg. The residual Cu content of P4, S4, and Z4 treatments is 25, 24, and 23 mg/kg. They are 2.1, 2.0, and 1.9 times of the control, respectively, and it is significantly higher than the other treatments, but the difference between the three is not significant. Residual Zn content changed from 23 to 29 mg/kg. The fixation rate has increased by 2.1%. Z4 treatment has the highest residual Zn content of

50 mg/kg. Compared with the control, it increased by 72.4%, and it was significantly different from other zeolite treatments; followed by S4 treatment, the content of residual Zn in the soil was 48 mg/kg, which was an increase of 65.5% compared with the control. From P2 to P4, the content of residual Zn in the soil is 41, 42, and 43 mg/kg, respectively. Compared with the control, it increased by 40.0, 44.8, and 48.3%, respectively, which is consistent with Wang Xiaoyu's research results [31]. With the increase of passivation time, the passivation rate of soil Zn increased. From 30 to 60 days, the passivation rate of soil Zn increased from 32.2–55.5 to 46.0–63.9%. The residual Cd of Z4, S4, and S3 is 0.21, 0.18, and 0.17 mg/kg, respectively. Compared with the control, it increases by 90.1, 63.6, and 54.5%, which is consistent with the research results of Guo *et al.* [40]. By applying passivating agent to the soil, the effective Cd content is significantly reduced, and the fixed content is increased. The content of residual Pb in the soil remains as zeolite > palygorskite > sepiolite > the control. The residual Pb content of Z4 reaches 30 mg/kg, which is

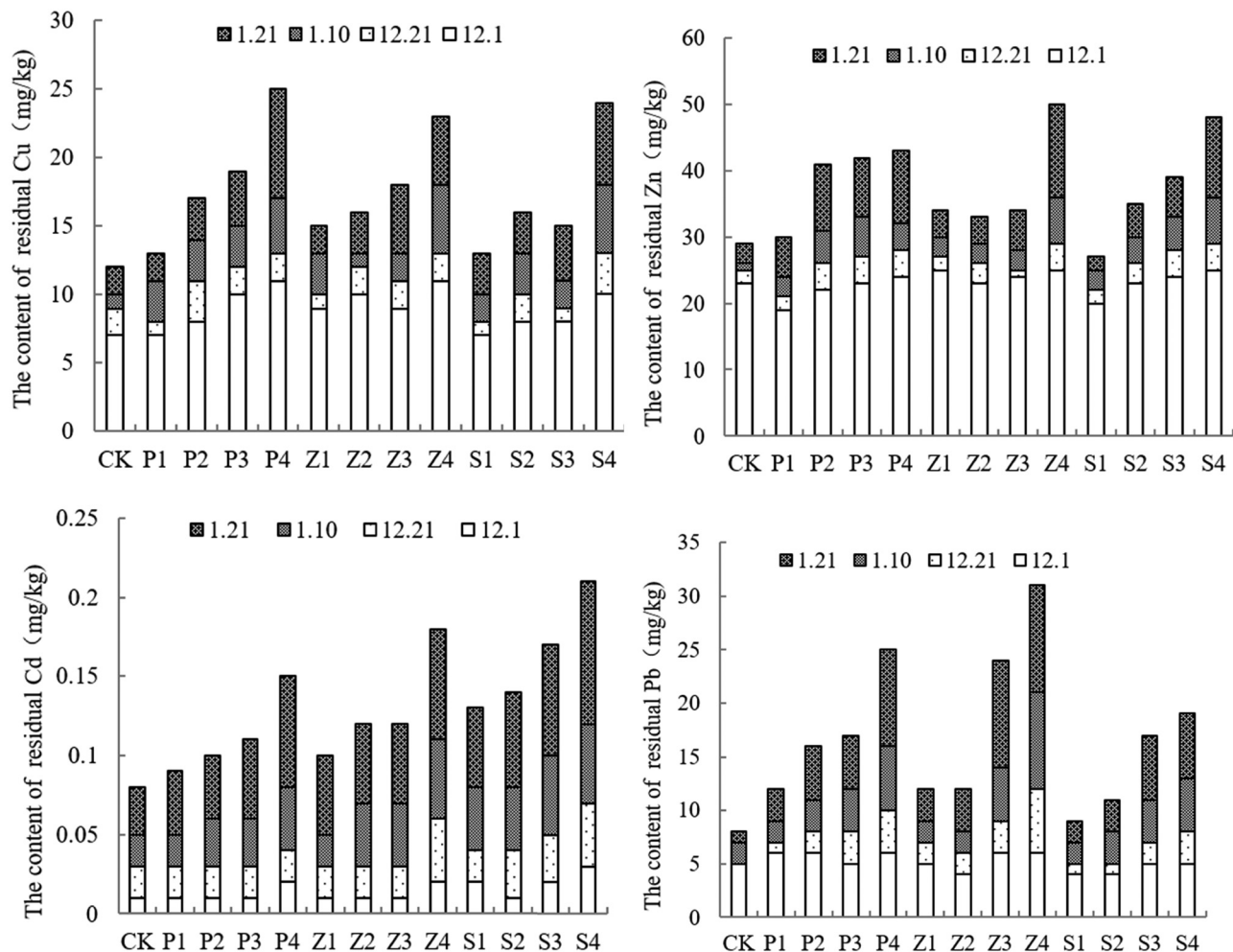


Figure 3: Changes in the content of heavy metal residues in the soil at different cultivation times. Note: The bar chart in the figure uses December 1, 2020 as the base, showing the increase in the content of heavy metals in residues in the soil with different treatments over time.

3.75 times higher than that of the control, and it is significantly different from the other treatments, followed by P4 and Z3 treatments, which are 25 and 24 mg/kg, respectively, which are 3.13 and 3.00 times higher than those of the control. A comprehensive analysis of the changes in the content of the residues of the four elements in the soil shows that Cu, Zn, Cd, and Pb treated with zeolite have the best effect, followed by sepiolite, and finally palygorskite.

4 Conclusion

In this experiment, the constant temperature culture experiment found that different kinds of passivators have a good fixation effect on heavy metal-contaminated soil.

(1) With different passivating agents, the dissolved Cu, Zn, and Pb contents of the zeolite treatment have the highest decrease rate. The sepiolite treatment has the highest decrease rate of dissolved Cd. The content of

heavy metals in the residue state of each treatment gradually increased with time.

- (2) Different addition amounts of passivators reduce the content of dissolved heavy metal and increase the content of residual heavy metal. Among the three passivating agents, 5% zeolite has the best passivation effect on heavy metals.
- (3) With different amounts of passivation agents, the dissolved content of Cd and Pb in contaminated soil is the control > palygorskite > sepiolite > zeolite. The curing effect of Cu, Zn, Cd, and Pb treated with zeolite is the best, followed by sepiolite, and finally palygorskite.

Comprehensive analysis shows that 5% zeolite has the best effect and can be used for passivation restoration of heavy metals Cu, Zn, and Pb in the soil in the single-passivation treatment. The low concentration of sepiolite can repair Cd better than the other treatments.

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Conflict of interest: The authors declare they have no conflict of interests.

Ethical approval: The conducted research is not related to either human or animal use.

Data availability statement: All data generated or analyzed during this study are included in this published article.

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