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Article

In situ passivation effect of fertilizer passivation solutions with various pH on Pb-F contaminated soil

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Abstract

The Maoniuping rare earth mine in Mianning, Liangshan Prefecture, is the largest rare earth deposit in China and the second largest rare earth deposit in the world. During the mining of rare earths, F and Pb, both types of heavy metal waste can directly or indirectly enter the soil, causing local soil contamination, which in turn poses a threat to the health of local residents. In this study, soil samples were incubated with fertilizer passivation solutions with pH values of 4, 5, 6, 7, 8, and 9, with the soil water content adjusted to 50% of the maximum water holding capacity in the field. The Pb-F contamination of the soil samples was analyzed to determine the existing states of the soil F and Pb and to study the remediation effect of fertilizer passivation solutions on the Pb-F contaminated soil. The results showed that under different passivation conditions, the pH, Pb, and F of the soil substantially changed and the passivation effect was enhanced over time. The pH of the soil significantly increased. The analysis of the effects of F and Pb treatment revealed that when the passivation fertilizer solution of pH 5 was used, the available Pb and F in the soil decreased the most, and the residual state of Pb and F increased to 77.86% and 57.24%, respectively.

Key words: soil, F contamination, Pb contamination, in situ passivation, pH

Résumé

La mine Maoniuping à Mianning, dans la préfecture du Liangshan, abrite un des plus vastes gisements de terres rares de Chine, le deuxième en importance dans le monde. Lors de l'excavation de ces minerais, il arrive que les déchets de deux métaux lourds (F et Pb) pénètrent directement ou indirectement dans le sol et entraînent une contamination locale susceptible de mettre la santé des habitants de la région en danger. Les auteurs ont incubé des échantillons de sol avec des solutions de passivation fertilisantes à pH 4, 5, 6, 7, 8 ou 9, et ajusté la teneur en eau du sol afin qu'elle corresponde à la moitié de la capacité de rétention maximale relevée sur le terrain. Ensuite, ils ont analysé la contamination des échantillons de sol par les deux métaux lourds pour établir la concentration de F et de Pb existante et déterminer la mesure dans laquelle les solutions de passivation fertilisantes atténuent le problème. Les résultats indiquent une modification notable du pH ainsi que de la concentration de Pb et de F dans le sol, selon le degré de passivation. Les effets de la passivation s'accentuent avec le temps et le pH du sol s'élève de manière significative. L'analyse des effets du traitement du F et du Pb indique que la solution de passivation fertilisante à pH 5 réduit le plus la quantité de Pb et de F disponible dans le sol, la proportion des résidus des deux éléments passant respectivement à 77,86 % et à 57,24 %. [Traduction par la Rédaction]

Mots-clés : sol, contamination par le F, contamination par le Pb, passivation in situ, pH

Introduction

The Maoniuping rare earth mine in Mianning County, Sichuan Province, is the second largest light rare earth deposit in China and an important part of the world's rare earth resources. The development of rare earth mineral resources in Mianning began in 1988. Due to the excessive pursuit of resource development and economic interests, as well as the lack of an organized mining plan, rare earth development grew exponentially, resulting in more than one hundred rare earth mining enterprises at the peak of mining activities. This chaotic exploitation and mining resulted in serious ecological damage. Additionally, waste containing F and heavy metals, such as Pb, was directly and indirectly transferred to the surrounding environment, causing soil contamination in mining areas and their surroundings (Hu et al. 2017; Zhao et al. 2019). Therefore, urgent studies are needed for the remediation of Pb and F contamination in the soil around rare earth tailings.

The research on efficient remediation techniques for Pb-contaminated soils has been a popular, challenging topic in recent years. At present, the commonly used methods are soil dressing, isolation, encapsulation, leaching, electrochemical remediation, and microbial remediation (Yang et al. 2018; Li et al. 2019; Wang et al. 2019; Gao et al. 2020). Commonly used soil remediation techniques in China include in situ passivation remediation, phytoremediation, and agronomic regulation and control (Xiong et al. 2019). In situ passivation remediation technology is suitable for the remediation and treatment of vast cropland areas that have been lightly or moderately contaminated by heavy metals due to its low cost, ease of application, and high efficiency (Yang et al. 2015). In the solid phase of rare earth ore and in the soil, F (and other metal ions) readily forms solutions containing complex bioavailable ions, which can cause even more severe harm to the environment, ecosystems, and human health.

The toxic effects can be mitigated by adding passivation materials to the soil to change the existing form of Pb-F, reducing its bioavailability and mobility (Wołejko et al. 2020; Zhao 2020). The existing states of Pb and F in the soil are greatly impacted by pH, with a boost in F content in a more effective exchangeable state or Fe-Mn oxide-bound state following an increase in pH and an elevated F content in a less effective organic-bound or residual state following a decline in pH value (Blomberg et al. 2017). Currently, there are both single and composite soil passivators. Due to the complex composition of the soil, the remediation mechanisms of various passivators on soil contaminated by multiple heavy metals have not been completely elucidated. For instance, when activated carbon is used as a passivator, it can cause secondary contamination of the soil among other problems (Zhou et al. 2020).

The use of fertilizer passivation solution to remediate Pb-F contaminated soil has not been studied. The preparation of an efficient and environmentally friendly soil fertilizer passivation solution not only reduces the environmental and human hazards of Pb-F contamination in soil but also improves soil fertility and eliminates secondary contamination in the soil. Therefore, this study set up indoor soil incubation tests with acid and alkaline fertilizers of different pH levels as passivating agents. The effects of the passivating agents on the pH, the total Pb and F, and the extractable contents of the soil were investigated. Furthermore, the content changes of different forms of Pb and F in the contaminated soil were obtained using the sequential extraction method to compare the effects of different pH values on the existing forms of Pb and F. To find the most suitable pH fertilizer passivation solution that can reduce the available state content of Pb and F in soil, we provide a theoretical reference for the preparation of efficient and environmentally friendly soil passivation remediation agents.

Materials and methods

Experimental design

The study area was located in the Maoniuping rare earth mining district in Mianning County, Xichang City, Liangshan Yi Autonomous prefecture, Sichuan Province. A highly polluted farmland sample plot (102°1′22″E, 28°24′29″N, elevation 2140 m) was selected to collect 0–20 cm soil using diagonal sampling method, and uncontaminated soil samples were collected as background soil samples. The soil was sieved and taken at 50 g each and placed in 150 mL beakers for indoor simulated soil incubation. Ammonium chloride and ammonium carbonate were used as acid and basic passivation solutions, respectively, with the pH of the solutions adjusted to 4, 5, 6, 7, 8, and 9 (see Table 1 for fertilizer dosage) and triplicated for each pH level. The water content of the soil was adjusted to 50% of the maximum holding capacity in the field. A control group was set up at the same time without the addition of fertilizer passivation solution. Soil samples were rehydrated every 2 days with fertilizer passivation solution with the same pH to maintain the soil moisture content at 50% of the water holding capacity. Samples were kept under atmospheric conditions at room temperature. Samples were taken at days 0, 15, and 25 and were dried, ground, and sieved through a 60-mesh sieve. The soil samples were then analyzed for basic physicochemical properties and changes in Pb and F content in the relevant existing forms.

Analysis methods

Total fluoride was determined using an ion-selective electrode (GB/T 22104-2008; Ministry of Agriculture of the People’s Republic of China 2008); F content in other forms was determined by sequential extraction; total Pb was determined by ICP-MS after triacid digestion; the chemical speciation of Pb was determined by BCR sequential extraction (GB/T 25282-2010; National Technical Committee for Standardization of Land and Resources 2011); soil pH was determined using a potentiometer (soil to water ratio of 1:2.5); and soil organic matter (OM) was determined by potassium dichromate oxidation (OM).

The data were processed and plotted using Excel 2010 and Origin 2019. SPSS was used for statistical analysis, difference significance analysis, and correlation analysis.

Results and discussion

Effect of fertilizer passivation solutions with different pH values on the soil pH

To accurately delineate the Pb and F contamination in the soil around the rare earth mine, the basic physicochemical properties, Pb and F contents, and their relevant species in the soil samples were analyzed, with the results shown in Table 2.

According to the Soil Environmental Quality Standard (GB 15618-2018) promulgated by the State Environmental Protection Administration, the mining area had serious Pb and F contamination, as the concentration of Pb ions and F in the highly contaminated farm soil around the Maoniuping rare earth mine in Mianning County, Sichuan Province, were 503 mg/kg and 2776.0380 μg/kg, respectively, while their concentrations were 84 mg/kg and 966.4455 μg/kg in the background soil.

Soil systems have complex compositions and a large buffering capacity, and their pH reflects (to some extent) their pH buffering capacity. Therefore, the pH of the contaminated soil after passivation can approximately reflect the buffering capacity of the soil at the sampling site (Wang et al. 2019). In

Table 1. Fertilizer dosage of various pH gradients.

pH	4	5	6	7	8	9
Dosage	4 g NH_4CL HCl-adjusted pH	2 g NH_4CL HCl-adjusted pH	0.063 g NH_4CL	0.001 g $(NH)_2CO_3$	0.007 g $(NH)_2CO_3$	0.01 g $(NH)_2CO_3$

Table 2. Basic physicochemical properties, Pb and F contents, and their relevant species in the tested soil samples.

State	pH	Organic matter	Pb in reducible state	Pb in oxidizable state	Pb in acid soluble state	Total Pb	F in water	F in exchangeable soluble state	F in iron-manganese bonded state	F in organic-bound state	F in residual state	Total F
Contaminated soil	5.9	25.63	72.26	33.02	1.56	503.00	0.064	0.176	0.49	0.53	2774.77	2776.038
Background soil	5.0	20.49	22.22	32.47	0.72	84.00	0.03	0.05	0.19	0.12	966.05	966.44

Fig. 1. Trend of pH in the background soil group. [Colour online]

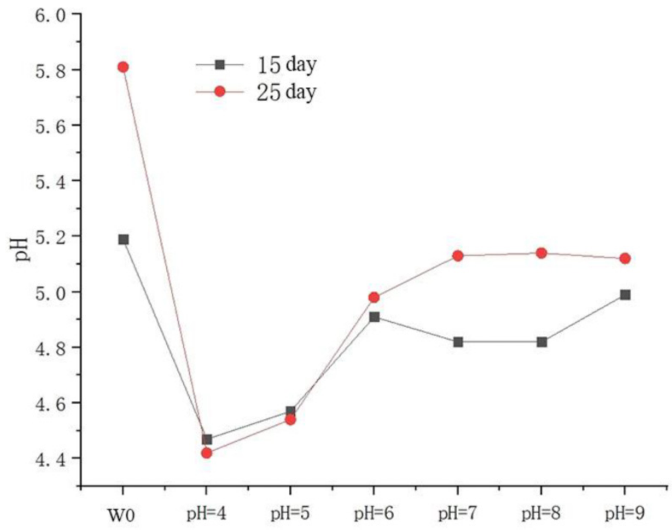
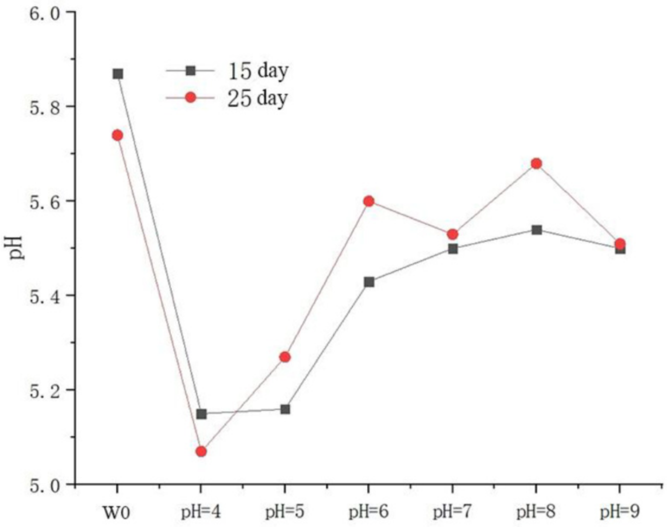


Fig. 2. Trend of pH in the contaminated soil group. [Colour online]



general, soil pH is negatively correlated with the exchangeable heavy metal content and positively correlated with the heavy metals that are carbonate bound, organic bound, or in Fe-manganese oxides, with insignificant correlation with the residual heavy metal content. Figures 1 and 2 show the variation in pH with increasing passivation time when the soil was passivated with fertilizer solutions of pH 4, 5, 6, 7, 8, and 9. The graphs indicate that the pH of the soil increased with increasing passivation time, and the pH of the treated samples was around 4.4–6.0 after 25 days of passivation, being weakly acidic. Comparison of the contaminated soil and the background soil prior to passivation revealed that the pH of the contaminated soil was higher than that of the background soil, and so was the content of Pb and F in the contaminated soil. When other parameters were identical, soil samples were passivated with liquid fertilizer of various pH values and subsequently analyzed. It was observed that the pH increased in both contaminated soil and background soil, changing from acid soil to weakly acid soil, and available Pb and F were significantly reduced, especially for the contaminated soil.

Cai et al. (2017) found that soil pH had a significant influence on the heavy metals associated with Fe-Mn oxides in soil. When pH was relatively high, the association with Fe-Mn oxides was favored. Limiting the soil pH to the range of 6.5–7.5 reduced F uptake by crops. In alkaline soils with high pH, F is more bioavailable, resulting in relatively strong accumulation of F by crops, which subsequently causes crop damage. Under acidic conditions, Pb is more bioavailable and mobile. In this experiment, the pH of the contaminated soil was approximately 5.0–6.0 after 25 days of passivation, slightly higher than the pH after 15 days of passivation. Additionally, the pH was higher in the soil treated with passivation solution of a higher pH. The pH in the background soil ranged from approximately 4.4 to 6.0 after 25 days, which was also higher than 15 days of passivation, and the pH increased with the increasing pH of the passivation solution. In general, the pH of the soil did not change significantly and re-

Fig. 3. Contamination soil group with 15 days passivation.

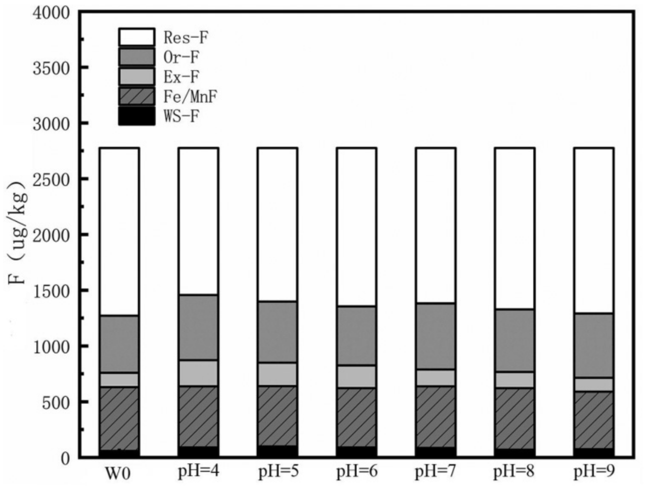


Fig. 4. Contamination soil group with 25 days passivation.

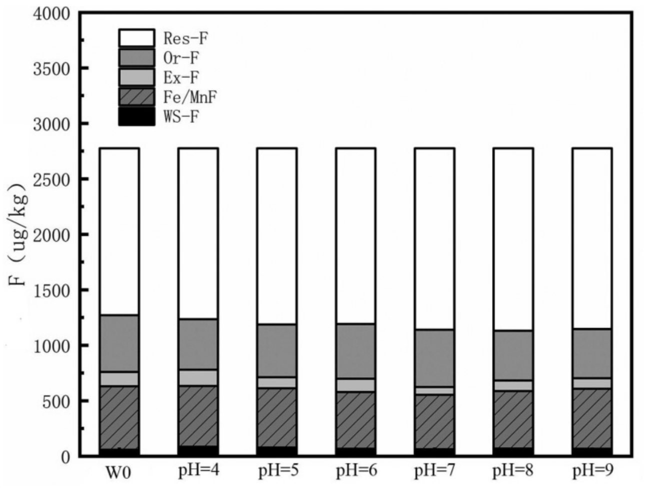


Fig. 5. Background soil group with 15 days passivation.

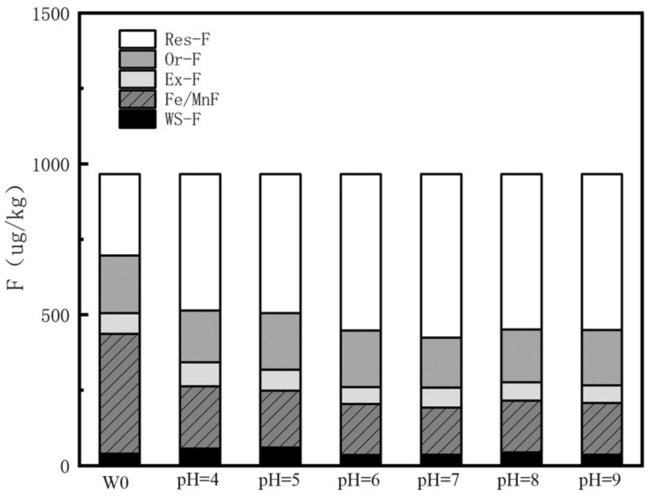
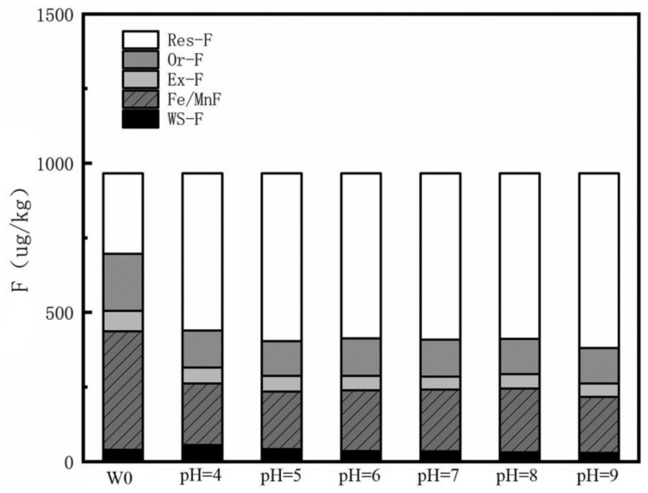


Fig. 6. Background soil group with 25 days passivation.



mained acidic, which may be related to the passivation time. The comparison between 15 and 25 days of passivation revealed that the pH of the soil was elevated with increasing passivation time, except for the passivation solution of pH 4. Considering that soil has a complex composition with strong buffering properties, longer passivation time may result in a neutral pH in the soil (Cao and Huang 2017).

Effect of fertilizer passivation solutions with different pH values on F species in soil

The variations in F species with increasing passivation time are shown in Figs. 3–6. In the contaminated soil treated with passivation solutions, the proportion of Fe-Mn bound F content within the total F decreased and approached the corresponding level in the background soil. In this study, the residual F content was defined as the difference between the total F and the sum of other forms of F. Residual F was often found in the lattice of mineral grains and was not bioavailable (Duan et al. 2021). The proportions of water-soluble F, Fe-Mn bound F, exchangeable F, and organic-bound F within the total F content of the tested soil were reduced to different degrees after passivation, while the residual F, which was not bioavailable, was enhanced. The results indicate that in situ passivation remediated F contamination of the test soil to some extent. On day 25, the highest Res-F content was 1589.07 mg/kg, observed in background soil and contaminated groups when treated with the fertilizer solution of pH 5. The effect of the passivation became more pronounced with increasing passivation time.

The proportions of WS-F, Fe/Mn-F, Ex-F, and Or-F to total fluorine decreased in both the 15 and 25 days passivation-treated contaminated soils, while the proportion of Res-F (not bioavailable) within the total F increased. The Res-F content after 25 days of passivation increased substantially compared with that after 15 days of passivation, and the best remediation effect was achieved when the treatment was passivated with a pH5 fertilizer solution.

Exactly the same results were observed in the background soil group as well. The best remediation results were achieved when passivation was carried out with a pH 5 fertilizer solution in both the contaminated soil group and the background soil group. It is likely that under acidic conditions, F tends

Fig. 7. Contamination soil group with 15 days passivation.

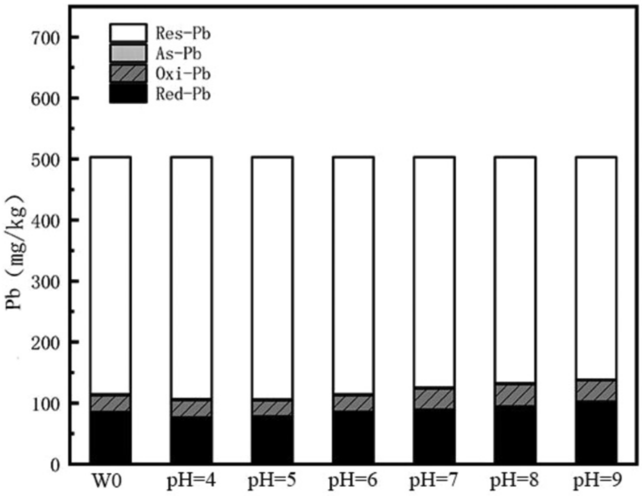
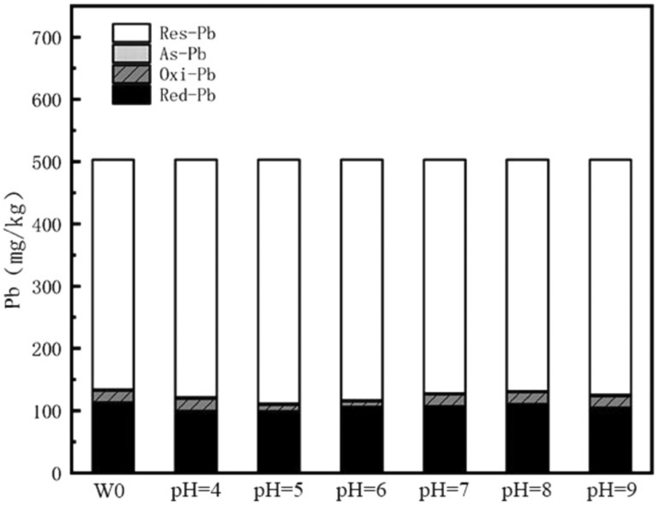


Fig. 8. Contamination soil group with 25 days passivation.



to form stable compounds with many elements, such as aluminum, silicon, iron, calcium, magnesium, boron, niobium, tantalum, beryllium, and lithium. In addition to this, soil colloids and oxides have a strong adsorptive effect on F (Yuan et al. 2019). Humus in the soil is also an important adsorbent for fluorine. The mechanism of adsorption is mainly through the change of fluoride ions with functional groups, such as carboxyl and hydroxyl groups, in humus, thus releasing hydroxyl groups (Tu et al. 2019). The effect of organic acids on F adsorption is complicated by the competition between anions and fluoride ions on the one hand and promotion under acidic conditions on the other hand. The time of passivation also affected the passivation effect, and there was no clear pattern in the fluorine content of various forms at 15 days of passivation.

Effect of fertilizer passivation solutions with different pH values on Pb species in soil

Heavy metals contaminate soil and harm the biosphere in their bioavailable states. Hence, according to the bioavailability of the different forms of heavy metals, they are divided into three major categories, i.e., active states, potentially active states, and unavailable states (Zhou et al. 2020). In the present experiment, the changes in the species of Pb in different treatment groups with increasing passivation time are shown in Figs. 7–10. It can be seen from the figures that for different initial pH values and passivation times, the overall oxidizable, acid soluble, and reducible Pb contents slightly increased and the residual Pb contents slightly decreased in the contaminated soil group, while the overall oxidizable, acid soluble, and reducible Pb contents showed a minor decline and the residual Pb contents displayed a slight rise in the background soil group.

The Pb content in oxidizable, acid soluble, and reducible states, which were potentially bioavailable, increased slightly in the contaminated soil after 15 and 25 days of passivation, while the proportion of residual Pb, which was not bioavailable, decreased relative to the total F. This may be due to the

Fig. 9. Background soil group with 15 days passivation.

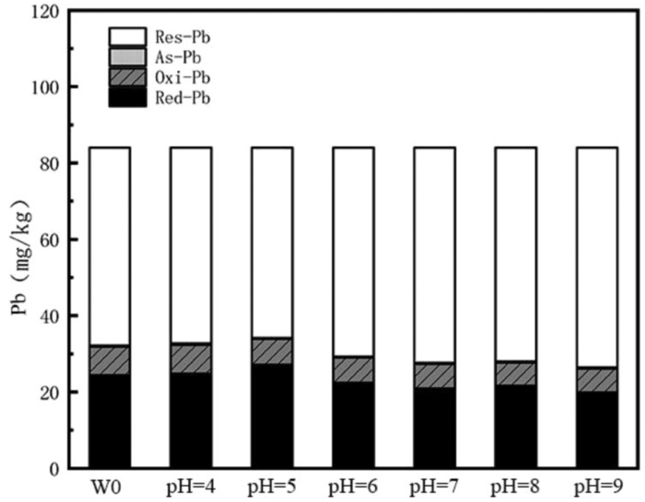


Fig. 10. Background soil group with 25 days treatment.

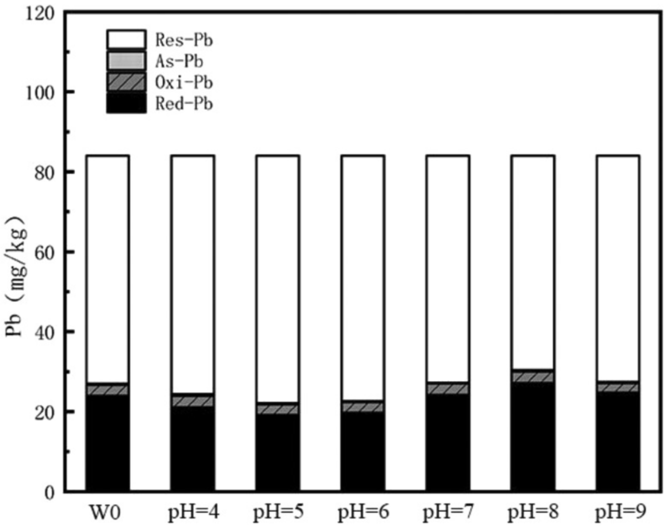


Table 3. Organic matter contents of soils at 15 and 25 days of passivation.

Fertilizer pH	Contents of organic substances	
	15 days content (mean ± SD)	25 days content (mean ± SD)
Contamination blank	26.65	18.12
Contamination pH = 4	28.46 ± 1.02	21.78 ± 0.75
Contamination pH = 5	25.67 ± 5.51	8.85 ± 156.53
Contamination pH = 6	25.96 ± 4.02	22.63 ± 1.93
Contamination pH = 7	22.53 ± 0.01	24.59 ± 0.18
Contamination pH = 8	19.95 ± 1.31	21.31 ± 0.00
Contamination pH = 9	30.75 ± 124.17	24.51 ± 0.75
Background blank	3.5	9.79
Background pH = 4	11.95 ± 2.59	10.00 ± 1.93
Background pH = 5	19.90 ± 209.16	7.04 ± 8.74
Background pH = 6	4.20 ± 35.21	9.54 ± 19.76
Background pH = 7	10.45 ± 1.22	9.54 ± 5.92
Background pH = 8	10.53 ± 10.36	10.47 ± 0.23
Background pH = 9	11.45 ± 0.09	11.11 ± 6.94

fact that the residual Pb in the soil gradually desorbed from the soil to become acid soluble and reducible Pb during passivation with the addition of fertilizer solutions of different pH (Wang et al. 2019). In addition, it may be due to the replacement of OH^- by F^- in the soil to form PbF^- and PbF_2, resulting in an increase in the proportion of potentially bioavailable Pb to total Pb after passivation (Yang et al. 2018). The opposite experimental results were observed in the background soils, in which the contents of oxidizable, acid soluble, and reducible Pb, all potentially bioavailable, showed an overall slight decrease after 15 and 25 days of passivation, while the proportion of residual Pb (not considered bioavailable) to the total Pb increased. This may be due to the low content of Pb and F in the background group, which was less likely to form Pb-F complex contamination, and the residual Pb content in the soil grew after a period of passivation.

Effect of fertilizer passivation solutions with different pH values on soil organic matter content

Organic matter enhances soil fertility and improves its ability to adsorb heavy metals. The buffering and purification effects of organic matter are shown in the following aspects: they participate in ion exchange in the soil, stabilize soil structure, and provide microbial active substances, which can form metal–organic complexes of different chemical and biological stability with metal oxides, metal hydroxides, and metal ions of minerals (Zhou et al. 2018). Table 3 shows the organic matter content in the contaminated and background soils after 15 and 25 days of passivation. The soil organic matter decreased continuously with increasing passivation time and was slightly higher in the contaminated group than in the background group.

It can be observed from the table that the organic matter content of the contaminated soil group decreased significantly with an increase in passivation time. Organic matter contains a large number of active functional groups, such as hydroxyl and carboxyl groups, which are negatively charged after the release of H^+ and have a strong adsorption and ion exchange effect on positively charged metal ions, thus forming compounds of different solubility with heavy metal ions. This is how organic matter impacts the states of heavy metals in soil. On the other hand, organic matter can indirectly increase the pH value of the soil and enhance the adsorption of heavy metal ions (Yang et al. 2018). The organic matter in the contaminated soil decreased after passivation due to adsorption and ion exchange with F and Pb ions in the soil. The organic matter content of the background group decreased slightly with an increase in passivation time, likely because the metal ion content of the background soil was low and the degree of surface adsorption and ion exchange with the organic matter was also low. Therefore, the organic matter content of the soil decreased less after 15 and 25 days of passivation.

Conclusion

In situ passivation experiments were conducted on contaminated and background soils around the rare earth tailings in Mianning County to investigate the in situ passivation effect on soil Pb and F content with passivation solutions of different pH values, from which the following conclusions were drawn.

(1) As the passivation time increased from 0 to 15 and 25 days, the pH of the contaminated soils passivated with pH 4 and pH 5 solutions increased to 5.07 and 5.27, respectively. The pH of the contaminated soils treated with pH 6–9 passivation solutions did not differ substantially, falling in the range of 5.51–5.74, approaching neutral pH. This indicates that the soil had an adequate acid–base buffering capacity, showing relatively high stability.
(2) According to the analysis of the leaching content of background and contaminated soils, it can be tentatively concluded that the passivation solutions with various pH values had different treatment effects on Pb and F in soils, among which the available Pb and F in soils passivated with pH 5 solution displayed the most noticeable reduction, and the residual Pb and F increased to 391.63 and 1589.07 mg/kg, respectively.
(3) The use of fertilizer as a passivation solution to remediate Pb-F contaminated soil and the preparation of an efficient and environmentally friendly soil passivation solution not only reduces the harm from Pb-F contaminated soil to the environment and humans but also improves soil fertility and eliminates secondary contamination in the soil. In this experiment, the water-soluble F and Fe-Mn bound F were significantly reduced and the residual F content was significantly increased after the application of the fertilizer passivation solutions, which significantly improved the ecological conditions and mitigated the potentially harmful effects on human health.

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Copyright

Data availability

The data that support the findings of this study are available from the corresponding author upon reasonable request.

Author information

Competing interests

The authors declare no conflict of interest.

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