

Reduction and Stabilization Remediation of Hexavalent Chromium (Cr⁶⁺)-contaminated Soil

Xiaohui Lu ^{1*}, Bing Li ¹, Lin Guo ², Gubin Zhang ², Peifang Wang ¹

¹ School of Earth Science and Engineering, Ministry of Education Key Laboratory of Integrated Regulation and Resource Development on Shallow Lakes, Hohai University, Nanjing 210098, CHINA

² Henan Institute of Geological Survey, Zhengzhou 450001, CHINA

* Corresponding author: luxiaohui945@hhu.edu.cn

Abstract

Hexavalent chromium (Cr⁶⁺)-contaminated soil samples were obtained from an electroplating workshop in Jingjiang, China. Then, the Cr⁶⁺-contaminated soil was processed using reduction and stabilization methods. Six reducing agents with different remediation abilities with respect to Cr⁶⁺ contamination (iron, SSL, FeSO₄, Na₂HSO₃, Na₂S₂O₃, and Na₂S) were tested. The most efficient reducing stabilizing agent was FeSO₄; which efficiency varied with the pH, with the optimum pH being neutral or near neutral. Lastly, the effects of acidified ferrous sulfate as a reducing agent, cement as a curing agent, and the combined use of dyeing were tested. Increases in the cement content and curing age led to a marked improvement in the curing effect on the Cr⁶⁺-contaminated soil.

Keywords: six valence chromium, soil pollution, reducing agent, curing agent

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INTRODUCTION

Chromium (Cr) is a common metal pollutant in the natural environment. It is also in the top 20 poisonous substances under the control of the American superfund (Chrysochoou et al. 2012). There are three common Cr valence states in nature: divalence (Cr²⁺), trivalence (Cr³⁺), and hexavalence (Cr⁶⁺). The degree of harmful chromium poison is associated with its valence. For example, studies have shown that Cr⁶⁺ is 100 times more harmful than Cr³⁺. The valence of chromium and other similarly reactive elements, forming new compounds, exhibit a range of potential harm (Tang 1997). At present, the traditional methods for remediation of soil from Cr⁶⁺ pollution include chemical leaching, electrokinetic remediation, and in-situ reduction. These methods have not been widely used because of their high economic cost and time requirement (Mowla et al. 2013). In this paper, the chemical reduction solidification/stabilization technology of chromium-contaminated soil has been investigated. Stabilization/solidification technology emerged after 1950, primarily for the removal of hazardous wastes with radioactive properties (Barth 1990). Since 1980, the methods of stabilization and solidification have been improved and popularized. Currently, the most widely used curing material at

home and abroad is cement. This technology has evolved into a combination of cement, reduction materials, clay and other technical methods (Vipulanandan and Krishnan 1990).

Regarding stabilization studies, Shao and Jiang studied the purification principle of Cr compounds with montmorillonite as the main mineral material (Shao and Jiang 1999). These studies found that the purification principle mainly depended on the cation exchange adsorption performance of montmorillonite. Langton has shown that the addition of coal ash in a flocculent can make heavy metals more readily available, or lose electrons becoming more stable (Langton 1989). Zhang et al. (2014) investigated 16 chromium-contaminated sites in China, and explored the effects of FeSO₄, CaO, and cement as reducing and solidifying stabilizing agents on the reduction stabilization of chromium-contaminated soil. It was concluded that increasing the water content of the system, adding acid, adjusting the pH value, or increasing the ratio of CaO and cement could improve the efficiency of reduction and solidification stabilization. Chen, Pu and Bi showed that in Nanjing there was a serious Cr⁶⁺ soil overload compromising environmental fitness (Chen et al. 2013).

Table 1. Chemical composition of contaminated soil

Element name	Cr	As	Cd	Cu	Ni	Pb	Zn	Hg	Six valence chromium
Concentration (mg/kg)	2239.59	13.81	0.23	48.09	26.10	43.41	70.63	0.18	1915.41

Table 2. Basic physical properties of contaminated soil

Water content (%)	Bulk density (g/cm ³)	Porosity (%)	pH	Organic matter (%)	Cation exchange capacity (cmol/kg)	Liquid limit (%)	Plastic limit (%)
31.3	1.42	43.6	7.8	0.54	8.6	33.6	21.4

Table 3. Soil particle size composition content

Clay	Powder	Fine sand	Coarse sand
D ≤ 0.002 mm	0.002 mm < D ≤ 0.02 mm	0.02 mm < D ≤ 0.2 mm	0.2 mm < D ≤ 2 mm
10.6	1.7	70.3	17.6

By adding FeSO₄ to the soil, Cr⁶⁺ was reduced to Cr³⁺. NaOH was then added to induce a hydroxide of Cr, such as Cr(OH)₃. After the restoration of the soil to meet the relevant standard guidelines, the tendency of Cr diffusion into the food chain was solved. Test results showed that this technology could be very good for the remediation of chromium-contaminated soil, suitable for the needs of the construction of human settlements. In the field of curing research, Meegoda et al. used silica as the curing material in concert with the solidification/stabilization technology for the treatment of Cr-contaminated soil (Meegoda et al. 2003). On a small scale, silica was incorporated into the Cr polluted soil, and with fully mixing, was able to achieve Cr curing/stabilization. Liu aimed to solve the environmental pollution problem caused by leaching of Cr⁶⁺ into the soil and infiltration into groundwater (Liu 2015). Taking chromium-contaminated soil from a chromium polluted storage yard in Shenyang, the influence of different curing ages of lime and fly ash on leaching toxicity of Cr⁶⁺ was studied. The mechanism of combined repair was also analyzed. Conversely, Danili used natural latex polymers to improve cement solidification of heavy metals (Daniali 1990). It was found that the bonding force between cement particles and contaminants was enhanced by this method. In this manner, the polymer takes up the void in the capillary zone and in bulk soil, and the leaching concentration of heavy metals was reduced. Researchers such as Kalb, Iii and Colombo used modified sulfur cement to solidify the cinder (Kalb et al. 1991), which enhanced the compressive strength and tensile strength of the solidified soil and improved the ability to inhibit sulfate attack. The above technologies have been comprehensively promoted all over the world in the process of pollutant treatment. Therefore, understanding the evolution of this technology can help to popularize and perfect its advancement. From the perspective of management processing of pollutants to

**Fig. 1.** Panorama of electroplating workshop

reduce additional damage to the environment, this technology has positive and practical significance.

MATERIAL AND METHODS

Test Soil

Contaminated soil samples were taken from an electroplating workshop, Measuring Tool Co., Ltd., located in Jingjiang, China. The surface soil layer (0-2 m) was taken from the site as shown in **Fig. 1**. The main chemical composition, physical properties, and particle sizes are shown in **Table 1**, **Table 2**, and **Table 3**, respectively.

Test Equipment

Balance, 60 tons of pressure testing machine, sand sieve size (0.08 mm, 1.25 mm, 5 mm), electric bending testing machine: DKZ type 5000, a unified test mill, high temperature oven Freas645 thermostatic oscillator Innova 43, refrigeration centrifuge CR21GIII, Flame atomic absorption spectrophotometer: AA800, spectrophotometer UV2550, PHS-3C precision acidity meter.

Test Methods

(1) Screening of Cr⁶⁺ reducing agents. The purpose of this experiment was to reduce the amount of reductant. Based on the stability reduction effect of Cr⁶⁺



Fig. 2. An instrument for the determination of Cr⁶⁺



Fig. 3. Instrument and equipment for leaching toxicity

and the engineering cost, the best reducing stabilizer was determined. 200 g of the chromium-contaminated soil sample was added to the mixer. A 5% mass ratio of six reducing agents (iron, SSL, FeSO₄, Na₂HSO₃, Na₂S₂O₃, and Na₂S) was added individually. The mixture was stirred well. In order to maintain consistency among samples, an appropriate amount of water was added during the mixing process to maintain the soil moisture content between 20% and 30%. As shown in **Fig. 2** and **Fig. 3**, the content of Cr⁶⁺ in soil samples and in the leaching solution was determined.

(2) Reduction effect of different reducing doses. From the six reducing agents, it was found that FeSO₄ had a better remediation effect on the contaminated soil. Based on the previous experiments, the effect of reduction, stability, availability, and amount of Cr on the reduction of Cr⁶⁺ by FeSO₄ addition was investigated. 200g chromium-contaminated soil samples were added to the soil mixer followed by a 1%, 3%, and 5% mass ratio of ferrous sulfate. In order to maintain consistency among samples, an appropriate amount of water was added during the mixing process to maintain the soil moisture content between 20% and 30%. As shown in **Fig. 2** and **Fig. 3**, the content of Cr⁶⁺ in soil samples and in the leaching solution was determined.

(3) Reduction effects at different pH values. The stability reduction efficiency of FeSO₄ at varying pH levels was analyzed. 200 g of chromium-contaminated

soil samples were taken separately. Then, a 5% mass ratio of ferrous sulfate was added to the scale. The pH value was adjusted to 3, 5, 7, 9, and 11, respectively. The sample was mixed evenly, then the appropriate amount of water was added to adjust the soil moisture content to between 20% and 30%. The mixture was shaken for 10 min. After centrifugation, the supernatant was filtered into the sample bottle to be tested.

Stabilizing Effect of Reducing Agents on the Cement Solidified Body

(1) Determination of Cr⁶⁺ in the leaching solution of cement with different proportions of cement and reducing dose. The cement curing agent was selected and the quantity of soil was 200 g. The proportion of cement was adjusted to 20% and 40% soil, adding 3% and 5% ferrous sulfate, respectively. The soil was mixed with cement and reducing agent and stirred to ensure uniform mixing. Pure water was added with continued mixing, in order to maintain the moisture content between 20% and 30%. The static pressure method was adopted and made Φ5 cm*H10 cm cylindrical specimens. The sample was placed in a sealed bag and stored in the curing room for maintenance. The leaching toxicity of the solidified body was tested after curing. The experiment was carried out at room temperature.

(2) Determination of Cr⁶⁺ in the leaching solution of the solidified body with different curing time. The cement curing agent was selected and the quantity of soil was 200g as in the previous experiment. The proportion of cement was adjusted to 20% and 40% of soil, adding 3% and 5% ferrous sulfate, respectively. The soil was mixed with cement and reducing agent and stirred to uniform mixing. Water was added with continued mixing in order to maintain the moisture content between 20% and 30%. The static pressure method was adopted and made Φ5 cm*H10 cm cylindrical specimens. The sample was placed in a sealed bag and stored in the curing room for maintenance. The leaching toxicity of the solidified solid was determined 7 d and 28 d after maintenance. The experiment was carried out at room temperature.

Analysis and Test Methods

(1) Chromium standard curve. Chromium standard stock solution was made by adding 0.29 g K₂Cr₂O₇ in 1000 mL water. Standard curves were generated by aliquoting 0, 0.20, 0.50, 1, 2, 4, 6, 8 and 10 mL chromium stock solution and adding water to the liquid mark. Then, 0.5 mL of H₂SO₄ was added, followed by 0.5 mL H₃PO₄ color liquid. The mixture

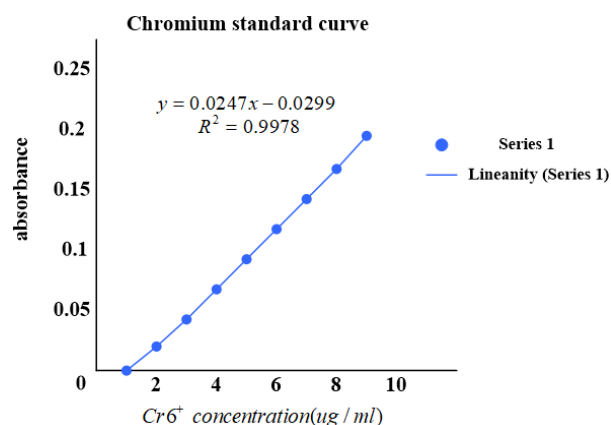


Fig. 4. Chromium standard curve

was shaken 5 times in 10 min. Samples were read at 540 nm using 1 mL or 3 mL water containers as the comparison blank. Blank samples were also used to test light responsiveness and correction to zero balance. The absorbance was set to the Y coordinate, and concentration of Cr⁶⁺ was set to the X coordinate to generate the standard curve, as shown in **Fig. 4**.

(2) Determination of total chromium. Samples of 0.2 to 0.5 g (accurate to 0.0002 g) were placed in a 50 mL Teflon crucible, and 10 mL HCL was added. The solution was placed on an electric heating plate under the hood on the low temperature setting. Decomposition continued until the solution evaporated down to 3 mL. The solution was removed the heat to cool. Subsequently, 5 mL HNO₃, 5 mL HF, and 3 mL of HClO₄ was added and the mixture and placed on an electric heating plate set to 150°C for 1 hour. After 1 h the lid was opened. The mixture was kept under constant heat, with regular shaking, in order to achieve a good effect on silicon removal. Heating was continued to decompose organic matter until the black organic material on the inner wall of the crucible disappeared. The lid was opened, white smoke and steam were visible and the content was sticky. The sample was removed and cooled. Then, 3 mL HCL was added to dissolve the insoluble residue. Finally, the solution was brought to 50 mL in a volumetric flask, 5 mL of NH₄CL aqueous solution was added, plus enough cooling water to the volumetric mark. The final mixture was shaken well.

(3) Five step extraction method. (1) Commutative state: 0.50 g of sample (less than 0.01%) was placed into a 50 mL centrifuge tube. 16 mL 1 mol/L magnesium chloride (MgCl₂·6H₂) was then added to the tube, and the mixture was oscillated at room temperature for 1 h (200 rpm). Subsequently, the mixture was centrifuged for 10 min (4000 rpm). The supernatant was removed,

filtered, and the volume adjusted with a 50 mL volumetric flask.

The residue after treatment with (1) was extracted with 16 mL of 1 mol/L sodium acetate (NaAc) at room temperature. The pH was adjusted to 5.0 with acetic acid (HAc) before extraction. The mixture was shaken for 8 h (200 rpm), then centrifuged for 10 min (4000 rpm). The supernatant was removed, and adjusted to 50 mL with a volumetric flask. Reagent preparation: 82.030 g NaAc/L was equivalent to 1 mol/L sodium acetate.

The iron-manganese oxidation state was extracted by adding 25% (v/v) acetic acid (HAC) solution and 16 mL 0.04 mol/L hydroxylamine hydrochloride (NH₂OH.HCl) to the residue after treatment (2). The extraction was conducted at 96±3°C, for 4 h, then centrifuged for 10 min (4000 rpm). The supernatant was removed filtered, and the volume adjusted to 50 mL with a volumetric flask. Reagent preparation: 2.780 g hydrochloric acid hydroxylamine, plus 25% acetic acid to maintain hydrochloric acid hydroxylamine at 1 L.

The organic binding was carried out on the residue treated by (3). 3 mL of 0.02 mol/L nitric acid (HNO₃), and 5 mL 30% (V/V) hydrogen peroxide (H₂O₂) were added. The pH was then adjusted with nitric acid (HNO₃) to 2.0. The mixture was heated to 85±2°C, incubated for 2 hours, and shaken several times during heating. 5 mL hydrogen peroxide (H₂O₂) was added, adjusted to pH to 2.0, then heated to 85±2°C. The mixture was kept warm for 3 hours with intermittent oscillation. After cooling, 5 mL of 3.2 mol/L ammonium acetate (NH₄Ac) was added and diluted with 20% (V/V) nitric acid to 20 mL, oscillating for 30 min. The mixture was centrifuged for 10 min (4000 rpm), the supernatant was removed and filtered into a 50 mL volumetric flask. Reagent preparation: 30% H₂O₂ was no longer configured. 1.35 mL nitric acid volume in 1 L was 0.02 mol/L nitric acid. 246.656 g ammonium acetate in 1 L was 3.2 mol/L ammonium acetate, or conversely, 123.328 g was set to 500 mL.

The residue was used after treatment by step (4). The dissolving analysis of nitric acid-perchloric acid-perchloric acid was used. The residue in the centrifuge tube was washed with 10% nitric acid in a crucible. Heating digestion was applied until the remaining solution was approximately 3 mL. Next, 15 mL HNO₃, 10 mL HF, and 5 mL HClO₄ were added and shaken gently. The mixture was heated continually until steam and white smoke was observed, and the soil sample appeared as a white or pale yellow gum block. If the material could not be digested completely, 5 mL HF

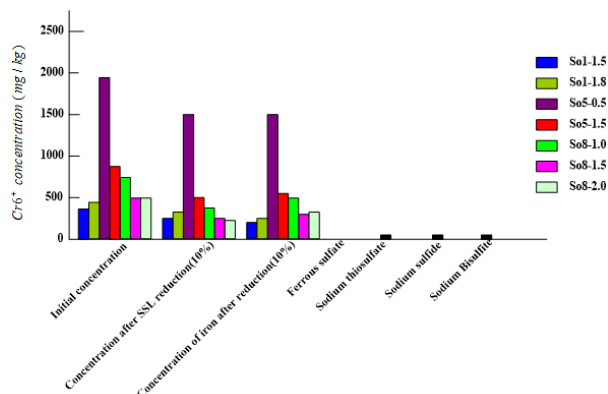


Fig. 5. The reduced hexavalent chromium concentration of six reducing agents

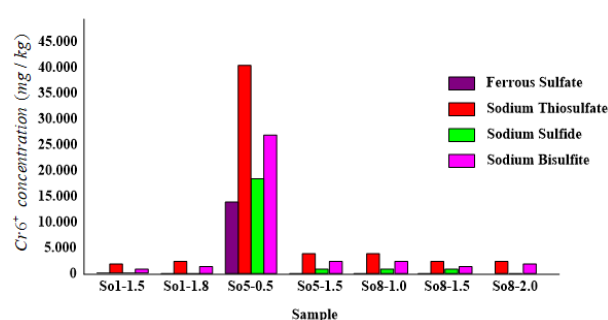


Fig. 6. Reduction of hexavalent chromium concentration by four soluble reducing agents

was added incrementally until complete digestion. The inner wall of the crucible was rinsed with 0.5% dilute nitric acid to dissolve residual material with gentle heating, and then transferred after cooling.

RESULTS

Screening of Cr⁶⁺ Reducing Agents

As can be seen from the relevant literature, the reduction reactions of different reducing agents under the same conditions produced differing effects on Cr⁶⁺ reduction. **Fig. 5** and **Fig. 6**, illustrate the reduction effects of 6 reducing agents on Cr⁶⁺. The same amounts were added of the following reagents: iron, SSL, FeSO₄, Na₂HSO₃ and Na₂S₂O₃, and Na₂S. The reducing effects of soluble agents FeSO₄, Na₂HSO₃ and Na₂S₂O₃, and Na₂S on Cr⁶⁺ were better. As can be seen from **Fig. 5** and **Fig. 6**, the reducing effect of insoluble reducing agents such as iron powder and SSL were more general. The removal rate of Cr⁶⁺ in soil was only approximately 50%. However, FeSO₄, Na₂HSO₃ and Na₂S₂O₃, and Na₂S soluble reductants had a good reduction effect on Cr⁶⁺ in soil. For toxic soil with high initial concentrations, these four chemicals can reduce Cr⁶⁺ in soil to below 5mg/kg, with a removal rate of more than 99%. Therefore, these four chemicals can be used to control Cr⁶⁺-contaminated soil. For the remediation of

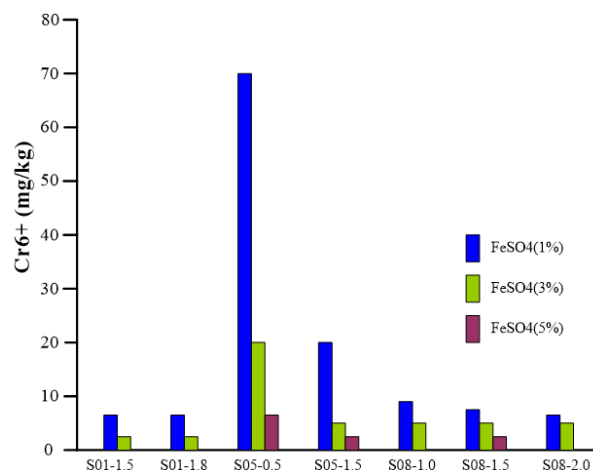


Fig. 7. The concentration of Cr⁶⁺ with different reducing doses

Cr⁶⁺-contaminated soil, the theoretical values required for FeSO₄, Na₂HSO₃, Na₂S, and Na₂S₂O₃ were 1:3, 1:3, 1:1.5, and 1:0.75, respectively. For an economic comparison, FeSO₄, Na₂HSO₃, Na₂S, Na₂S₂O₃ and other reductants, cost 350 yuan/t, 2450 yuan/t, 2650 yuan/t, and 1250 yuan/t, respectively. Based on comprehensive economic and environmental benefits, the use of FeSO₄ was the best treatment agent.

Reduction Effect of Different Reducing Doses

It can be seen from **Fig. 7** that when the percentage of soil added with 1%, 3%, and 5% FeSO₄, the Cr⁶⁺ in the soil can be well reduced, accompanied by an increase in the amount of Cr⁶⁺, and a corresponding reduction in the content of Cr⁶⁺. When the amount is 5%, the reduction rate of Cr⁶⁺ in the soil was more than 99%, and the concentration in the soil was lower than 5mg/kg, which conforms to the national standard. In the process of the reduction of the six valences, many of the ferric and trivalent chromium isoforms produced by the action will affect the hydrolysis of FeSO₄•7H₂O and thus reach equilibrium (Li et al. 2016). If the amount of FeSO₄ input is increased, the content of Cr⁶⁺ has little effect. FeSO₄•7H₂O hydrolyzes to form hydrogen ions and ferric hydroxide gums, accompanied by the development of reactions, the formation of hydrogen ions can make the reaction more prone to adsorption of Cr⁶⁺ in the soil due to the adsorption capacity of ferric hydroxide. In this case, the soil Cr⁶⁺ content was significantly reduced. If the excess addition of FeSO₄, relative to the precipitation of ferric and trivalent chromium, FeSO₄•7H₂O hydrolysis at this time will be affected, achieving a balanced reaction. Conversely, an increase the amount of FeSO₄•7H₂O input will occur in a small area of the reaction where the remaining amount of Cr⁶⁺ will be of little effect (Chen et al. 2013).

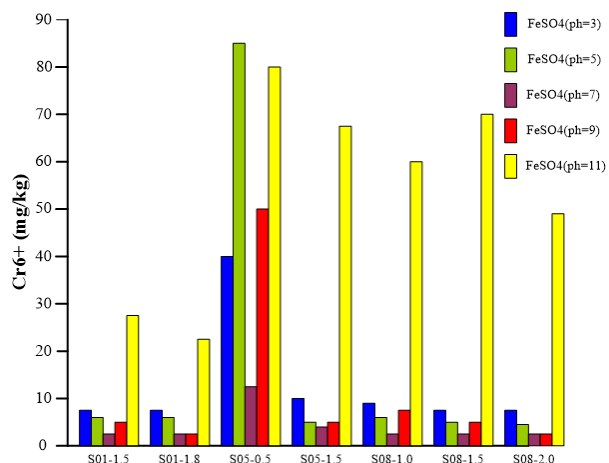


Fig. 8. Reduced Cr^{6+} concentration at different pH values

Ferrous sulfate treatment on chromium-contaminated soil had a good reduction effect, in a relatively short period of time. This treatment was shown to reduce the total chromium and Cr^{6+} leaching concentrations, reducing the content of Cr^{6+} in the soil. Regarding the long-term stability of the process, gradually iron hydroxide will form, and the stability of the stabilization of ferrous sulfate will continue to improve (Chen et al. 2013). However, studies have shown that (Huang et al. 2008), sulfate will erode concrete leading to its structural deterioration. Therefore, in selecting ferrous sulfate as a reducing agent to repair Cr^{6+} -contaminated soil, taking into account that excess ferrous sulfate may cause secondary contamination, the amount of ferrous sulfate added was 5%.

Reduction Effect of Different pH

As can be seen from **Fig. 8**, when the pH is close to neutral or neutral, the concentration of Cr^{6+} in the soil was significantly lower than that in the soil when the pH was acid or alkaline. Therefore, when neutral or near neutral, FeSO_4 -mediated reduction and stabilization had the optimum effect on the Cr^{6+} in the soil. The effect of acidic or alkaline conditions on the FeSO_4 reduction was not significant. The data showed that due to a large amount of hydrogen ion consumed by the reaction, ferrous ions are reduced to Cr^{6+} and ferrous ions are reduced. However, under acidic or alkaline conditions, the removal rate of Cr^{6+} is not obvious. When the soil is acidic, it affects the formation of chromium salt precipitation. When the pH value is high, it will reduce the availability of Cr^{6+} and the reduction of Cr^{6+} by FeSO_4 . When the pH value is neutral, Cr^{6+} can be removed optimally (Zhang et al. 2014). When the pH of the liquid is less than 7, the

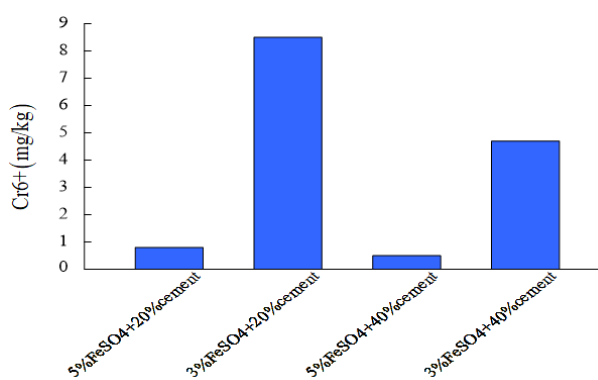


Fig. 9. Determination of Cr^{6+} in leaching solution of solidified body with different curing time

content of hydrogen ions in the chemical formula (1) will be greater, and the reaction will be developed in the positive direction. The reaction takes place completely and the efficiency of the removal of Cr^{6+} is relatively large. These results were consistent with a study by Zhang (Xu et al. 2011). When the pH is greater than 7, the concentration of hydrogen ions will be reduced, and thus will have an impact on the reaction. The greater the pH value, the greater the impact. Maurizio Pettine and others (Pettine et al. 1998) have shown that when the pH exceeds 10, the strong iron oxide in the liquid increases, thus ferrous ions used in the reduction of the available Cr^{6+} is reduced (Chen et al. 2005).

Determination of Cr^{6+} in solidified leaching solution with different proportions of cement and reducing. As can be seen from the **Fig. 9** and **Fig. 10**, when the amount of cement added was 20% and 40%, the amount of ferrous sulfate was 3% and 5%, respectively. The leaching concentration of ferrous sulfate with addition of 5% was lower than that of ferrous sulfate when the amount of ferrous sulfate was 3%. When the amount of ferrous sulfate was 3% and 5%, the cement content was 20% and 40%, and the leaching concentration of cement of 40% was lower than that of cement at 20%. With the passage of curing time, the leaching content of chromium in the chromium-contaminated soil was further reduced. When the cement content was 20% and 40%, curing was seen at 7 days (Mohammadmahdi et al. 2017). The curing agent leaching concentration was very high, and when the curing time was 28 days, the leaching concentration was further reduced. Over time, the solidified body reached steady state, thus its stability can be guaranteed.

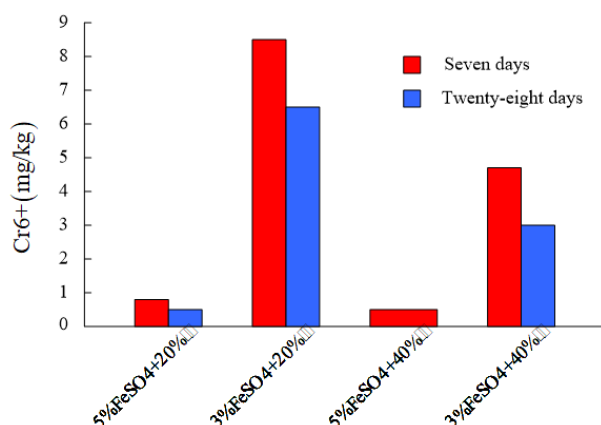


Fig. 10. Leaching concentration of solidified body with different curing time

DISCUSSION AND CONCLUSION

By adding the reducing agents iron powder, SSL, ferrous sulfate, sodium bisulfite, sodium thiosulfate, and sodium sulfide, the leaching toxicity of Cr⁶⁺ in soils decreased in varying degrees. The reduction efficiency of ferrous sulfate, sodium bisulfite, sodium thiosulfate, and sodium sulfide was more than that of an insoluble agent, and the removal rate of Cr⁶⁺ in soil was 99%.

Compared with sodium bisulfite and sodium thiosulfate, ferrous sulfate showed a better stabilizing effect on the reduction of Cr⁶⁺ in soil. In comparison, the stabilization effect of sodium sulfide and sodium sulfite was slightly worse. Previous studies have shown that the stability of the cured body was not good after detoxification with sodium sulfide and sodium sulfite, and the dosage of the two reducing agents increases to a critical point, which has a significant inhibitory effect on the efficiency of reduced chromium. Considering the economic and environmental benefits, ferrous sulfate was selected as the best reducing agent.

The results show that the curing effect of the contaminated soil was improved significantly with the increase of the cement, the amount of cement, and the curing age, such that the leaching toxicity was reduced and the strength of the contaminated soil was significantly improved.

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