



## Mechanochemical treatment of Cr(VI) contaminated soil using a sodium sulfide coupled solidification/stabilization process

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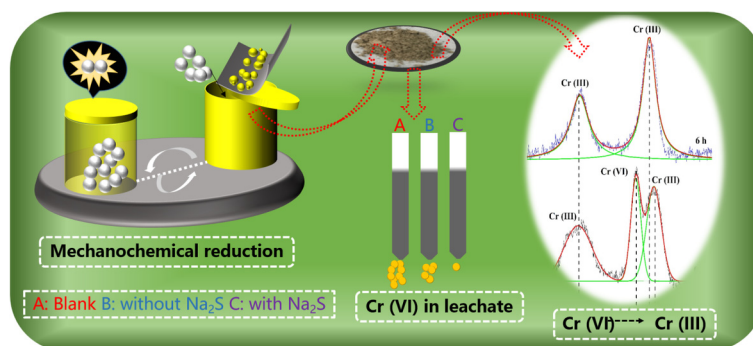
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### HIGHLIGHTS

- Mechanochemical reduction was applied for the remediation of Cr(VI) contaminated soil with Na<sub>2</sub>S.
- Mechanochemical reaction between Cr(VI) in contaminated soil and Na<sub>2</sub>S occurred.
- After treatment, leachable Cr(VI) concentrations reduced and be lower than 5 mg L<sup>-1</sup>.
- Mechanochemical reduction technique is promising for Cr(VI) contaminated soil remediation.

### GRAPHICAL ABSTRACT



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### ABSTRACT

In this research, mechanochemical reduction was carried out to remediate Cr(VI) contaminated soil, and the reduction effectiveness was evaluated by analyzing the corresponding leachable fraction obtained through the toxicity characteristic leaching procedure (TCLP) proposed by the EPA. The results indicated that mechanochemical reduction can efficiently reduce the Cr(VI) concentration in the leachate. Under a milling time of 2 h, milling speed of 500 rpm, ball-to-powder weight ratio of 14 and Na<sub>2</sub>S dosage of 5%, the Cr(VI) leaching concentration significantly decreased from 663.98 mg L<sup>-1</sup> to 0.84 mg L<sup>-1</sup>, much lower than the regulatory limit of 5 mg L<sup>-1</sup>. In addition, the significant decrease in Cr(VI) was mainly due to the reduction of Cr(VI) to Cr(III), as supported by X-ray photoelectron spectroscopy (XPS). The mechanochemical reduction with mechanism proposed in this experiment may involve two major processes: solidification and reduction (stabilization).

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## 1. Introduction

Currently, the country of China is one of the leading producers of

chromate with an annual production of greater than 160,000 tons (Deng, 2016). With increasing urbanization, many chromium contaminated sites have been created (Cao et al., 2016; Sinha et al., 2017; Wang et al., 2017). Soil surrounding a chromate plant was considerably contaminated due to most of the waste chromium residue accumulating in the air and chromium-containing waste water discharging into the environment. Considering the great hazards of hexavalent chromium pollution to both the environment and human health, chromium contamination has attracted widespread attention from the public and government (Gao and Xia, 2011; Homa et al., 2016; Wu et al., 2017a, b). In China, chromium pollution in soil has become an urgent environmental issue, and the remediation of chromium contaminated soil is a critical issue (Costa and Klein, 2006; Plugaru et al., 2009; Rumpa et al., 2011).

Typical remediation techniques of chromium contaminated soil include solidification/stabilization (S/S), chemical reduction, ex-situ chemical washing, electrokinetic remediation and bioremediation (Graham et al., 2006; Franco et al., 2010; Zhang et al., 2010; Achal et al., 2011; Yang et al., 2011; Lyu et al., 2018). Recently, bioremediation has attracted particular attention and observed rapid development; however, due to its long remediation cycle, the use of this technique has been confined to laboratory (Chao et al., 2016; Qu et al., 2016). S/S treatment of Cr(VI) contaminated soil can reduce the mobility and bioavailability of Cr(VI). Commonly used S/S agents in remediating Cr(VI) contaminated soil include cement, lime, asphalt, fly ash, gypsum, iron based agents, organic agents and sulfur based agents (Wang et al., 2015; Cerbo et al., 2017; Li et al., 2017). Efficient Cr(VI) removal performance was obtained by Velasco et al. (2012) using sodium sulfide for the treatment of chromite ore processing residue. A comparative study on the Cr(VI) reduction efficiency using different sulfur based agents was conducted by Mahdiah et al. (2016), indicating that sodium sulfur delivers the best performance.

Previous studies have demonstrated that heavy metal solidification can be obtained by simple mechanical ball milling (Concas et al., 2007; Montinaro et al., 2007, 2008, 2009; Chen et al., 2016). In particular, Montinaro and Concas et al. investigated dry ball milling for the treatment of synthetic kaolinite, sandy and bentonite type soils (Concas et al., 2007; Montinaro et al., 2007, 2008, 2009) and indicated that a leaching concentration lower than the regulatory threshold was achieved after mechanical treatment. Concas et al. (2007) made three hypotheses to explain the phenomenon induced by mechanical ball milling: (1) entrapment of heavy metals into aggregates; (2) solid diffusion into the crystalline reticulum; (3) irreversibly absorbed onto fresh surfaces. Similarly, Chen et al. (2016) found that the heavy metal concentration in leachate significantly decreased after ball milling. In addition, Zhang (2008) works indicated that metal oxides and hydroxides could be sequestered with quartz to form structures that bind heavy metals even in the presence of boiling aqueous acids.

In the present study, Cr(VI) contaminated soil was treated with sodium sulfide by ball milling, and the degree of treatment was evaluated by analyzing the leachable fraction of Cr(VI) obtained through the “toxicity characteristic leaching procedure” (EPA TCLP). To elucidate the mechanism induced by the mechanical treatment, particle size, SEM analysis and XPS analyses were carried out. This study indicates the great future potential of mechanochemical reduction applied to Cr(VI) contaminated soil remediation.

## 2. Materials and methods

### 2.1. Soil samples and reagents

In this study, Cr(VI) contaminated soil samples were collected from an abandoned chromic salt factory in Ji'nan, Shandong, China.

Before use, the soil samples were air-dried and sieved with a 0.15 mm standard mesh. Next, the total Cr(VI) contents were determined following hexavalent chromium extraction by alkaline digestion (EPA METHOD 3060A) (USEPA, 1992a, b). After filtration through a 0.45  $\mu\text{m}$  nylon syringe filter, the Cr(VI) contents of the filtrates were analyzed. The Cr(VI) concentration was measured by a spectrophotometric method using 1,5-diphenylcarbohydrazide with a UV–Vis spectrophotometer (TU-1810D, Purkinje, China) at a wavelength of 540 nm. The Cr(VI) content in the original soil was 13,806.4 mg kg<sup>-1</sup>. Additionally, the pH values of the soil samples were measured using the “Method NY/T 1377–2007” (NY/T 1377, 2007) in a 2.5:1 soil/H<sub>2</sub>O suspension using a pH meter (ST3100, OHAUS, China), and the investigated soil was determined to be alkaline with a pH value of 11.25. Sodium sulfide (anhydrous, 90% purity, J&K Scientific Ltd., Beijing, China) was used as the reductant in this study, and the extraction solution was prepared by diluting 5.7 mL of glacial acetic acid (Sinopharm Chemical Reagent Co., Ltd) with distilled deionized water (DDIW) (Milli-Q water, 18.2 M $\Omega$ -cm resistivity, Millipore Corp., Milford, MA).

### 2.2. Experimental procedures

The Cr(VI) contaminated soil samples were mixed with sodium sulfide in a planetary ball mill (FRITSCH P7, Germany) using 50 mL of zirconia vials and 70 g of 20.0 mm diameter balls. The vials were then sealed under normal atmospheric conditions and fixed on the mill. The soil samples were mechanically treated under air with different milling speeds, milling times, and ball-to-powder weight ratios (BPRs). The operating conditions of the ball milling trials are summarized in Table 1. In addition, 5 g of each treated sample was mixed with 100 mL of an extraction solution at a liquid-to-solid ratio of 20 (mL/g) in a capped polypropylene wide-mouth bottle on a rotary oscillator at 30  $\pm$  2 rpm for 18  $\pm$  2 h (23  $\pm$  2 °C) according to the “EPA TCLP” (Method 1311) (USEPA, 1992a, b). After extraction, the final liquid Cr(VI) concentrations of the leachates were measured by a spectrophotometric method using 1,5-diphenylcarbohydrazide with a UV–Vis spectrophotometer (TU-1810D, Purkinje, China) at a wavelength of 540 nm, and the Cr(VI) leaching concentration of the original soil was 663.98 mg L<sup>-1</sup>, which exceeded the EPA regulatory limit (TCLP Cr (VI)  $\leq$  5 mg L<sup>-1</sup>) by over 120 times. Based on the preliminary experiments, a mass fraction of 5% was chosen as the optimal dosage for the remediation of the Cr(VI) contaminated soil, as the Cr(VI) concentration in the leachate decreased to 0.84 mg L<sup>-1</sup>, which is much lower than the EPA regulatory threshold (data is shown in Fig. 1). All sample analyses in this study were carried out in triplicates, and the average values are reported.

### 2.3. Analytical methods

The particle size distribution was determined using a laser particle size analyzer on a Mastersizer2000 (CHUANG YU TECHNOLOGY CO., LTD, Zhengzhou, China), which covers a particle size range of 0.01–10000  $\mu\text{m}$ . The morphology of soil samples before and after treatment was investigated by scanning electron microscopy (SEM, Phenom Prox, Netherlands). X-ray photoelectron spectroscopy (XPS, ESCALAB 250Xi, Thermo Fisher, U.S.) was carried out to determine the valence variation of the elements (Cr, S) before and after mechanochemical treatment.

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