

Speciation and spatial distribution of Cr in chromite ore processing residue site, Yunnan, China

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Abstract Remediation of COPR sites requires the key information including chromium oxidation, speciation and spatial distribution. Samples were gathered from a COPR site in Luliang County in Qujing, Yunnan Province of China. The total Cr, Cr(VI) and chromium species were investigated. Results indicated the concentration of total Cr was between 110.5 and 21,774 mg/kg, and the concentration of Cr(VI) was between 0.1 and 1075 mg/kg. The map of total-Cr and ratio of Cr(VI)/total-Cr (%) showed that the maximum of total-Cr and Cr(VI) appeared in the layers near the surface. In the horizontal direction, the pollution was more serious in the middle and southeast part than that in the west. Additionally, acid extractable chromium increased in the layers at depth from −0.3 to −2.0 m, and it decreased in the deeper layers. There was a trend that the movable Cr(VI) migrated to the deeper layers, and then it turned into Cr(III). Water played an important role for the Cr distribution. Cr(VI) in COPR released to the soil solution after rainfall, and then gravity led the solution down to the deeper layers. After repeated rainfall and leaching, Cr(VI) moved to the deeper soil layers. Due to capillarity and evaporation, Cr(VI) migrated and was enriched at the

surface layer. Therefore, measures on controlling water movement should be taken in the remediation of the COPR site.

Keywords Chromite ore processing residue · Drilling · Spatial distribution · Chromium species · Migration

1 Introduction

Naturally occurring chromium occurs at an average concentration of 100 ppm in Earth's crust (Taylor 1964). The most frequently encountered valences are +III and +VI. Hexavalent chromium (Cr(VI)) is toxic to microorganisms, plants, animals and is believed to be carcinogenic to human beings (Langard 1990). Trivalent chromium (Cr(III)) is unreactive compared with hexavalent chromium and is considered to be an essential nutrient for human beings. In the natural soil, Cr(VI) is thermodynamically stable while Cr(III) is dynamically stable (Avudainayagam et al. 2003; Schmidt 1984). Cr(III) is the predominant species in soil and can be oxidized to Cr(VI) when oxidants such as oxygen, MnO₂ exist. Cr(VI) can be reduced to Cr(III) in reductive soil containing S^{2−}, Fe²⁺, Mn²⁺ or organic matter.

Chromite ore processing residue (COPR) is a hazardous solid waste yielded in the chromate and dichromate production with about 1.15–2.1 tons COPR being generated for every ton of dichromate manufactured by lime process (Farmer et al. 1999). This has caused public concern due to its high concentrations of hexavalent chromium and some potentially harmful elements. The vast majority of COPR over the latest century had been land filled at disposal sites (Farmer et al. 1999; Villalobos-Aragón et al. 2012), which can result in environmental hazards, such as

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soil contamination and ground water pollution. Many cases of chromium pollutions have been reported in the USA, England, Japan, India, Mexico (Burke et al. 1991; Villalobos-Aragón et al. 2012), and China (Yang 2012).

When Cr pollution aroused public and academic concerns, studies were conducted on the COPR landfill sites' remediation. Risk assessment of COPR sites can provide an important reference for the remediation. After a panel of experts evaluated the health hazards posed by COPR in Hudson County, New Jersey (Paustenbach et al. 1991), they concluded that site-specific health risk assessments should be conducted and remediation be considered if the Cr(VI) concentration exceeded 5000 ppm in the crystals. It became an important reference for risk assessment and remediation at COPR sites. Before remediation design, chromium chemical speciation and ability to release, and mineral composition of COPR were studied. The leaching behavior of chromium in COPR and in Cr contaminated soil can be investigate during batch leaching tests (Weng et al. 1994). Significant amounts of Cr(VI) were leached between pH 4.5 and 12, and Cr(III) was found in solution at pH < 5. The amount of Cr(III) leached was controlled by the solubility of Cr(III) precipitates, the extent of Cr(VI) reduction, and the magnitude of Cr(III) adsorption onto the soil surface. The remaining Cr(VI) in COPR resides mainly in hydrogarnet, hydrocalumite and ettringite and can be anionic exchanged (Hillier et al. 2003). Ettringite formation is a thermodynamically powerful reaction and also a viable expansion and failure mechanism in COPR (Moon et al. 2006). Sulfide is undoubtedly the most common method for COPR detoxification (Chrysochoou and Johnston 2015; Graham et al. 2006); however, new achievements in microbiology and nanomaterials have led recent research to focus on COPR remediation. Zhu et al. (2008) isolated *Leucobacter sp. CRB1* from the soil of the COPR site in China and found *Leucobacter sp. CRB1* had potential application for remediation of high concentrations of Cr(VI) contamination. Watts et al. (2015a) synthesized a highly reactive nano-scale biogenic magnetite and tested its performance for the remediation of COPR leachates. Another study (Watts et al. 2015b) introduced a Pd-bionanocatalyst which was effective for the treatment of alkaline Cr(VI) contaminated leachate. Efficiency, cost, and management varied from program to program of contaminated site remediation. Program screening was a very important step in the process of cleaning contaminated sites. Based on TOPSIS, Bai et al. (2015) established a screening system and applied this system in Cr contaminated soil remediation. The result has provided scientific support for the supervision of contaminated sites in China.

Remediation of COPR sites requires knowledge of chromium oxidation, speciation, and spatial distribution.

However, it was difficult to get the spatial distribution information of the chromium species because of the difficulty of collecting COPR samples from deep burial sites. Especially in China, the chromate chemical industry developed quickly, starting in the 1950s, thus leading to huge quantities of COPR being generated and deposited. Therefore, investigations and remediation at the COPR sites are urgent work for COPR pollution prevention and controlling. The purposes of this study are twofold: 1) to investigate the chromium species spatial distribution at a typical COPR site and 2) to find the ways of controlling pollution as well as give suggestions for remediation at COPR sites.

2 Materials and methods

2.1 Study area

The COPR site lies at about 1 km to the south-west of the chromate company in Luliang, a small county of Qujing city in Yunnan Province of China (latitude 24°59'50"N, longitude 103°36'37"E), neighboring the headstream of the Pearl River, Nanpan River (Fig. 1). The COPR site had been there for decades but is now closed. Before the COPR site closure, a small amount of household garbage had been

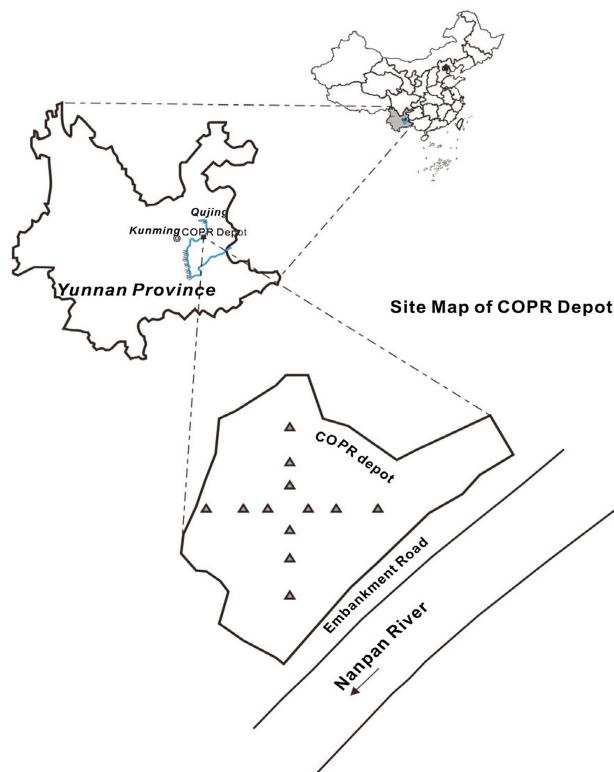


Fig. 1 Map of the COPR Site

discarded there, and soil had been delivered there as top cover.

2.2 Sampling

This work was carried out in early 2012. Samples were taken by drilling and were collected at depths varying from -0.3 to -6 m to the surface by intervals of 0.3 or 0.5 m. 12 boreholes formed, and a total of 105 samples were collected and stored in plastic bags. The samples were air-dried, crushed, and passed through a 100-mesh sieve for analysis.

2.3 Analytical methods

Total Cr, Cr(VI), and chromium species were determined in this work through microwave digestion. The procedure was as follows: 0.2 g sample, 8.0 mL nitric acid and 5.0 mL hydrofluoric acid were mixed and then processed in the microwave digest tank. After acid driven processing, the solutions were washed with 2% (v/v) nitric acid and settled in a 50 mL volumetric flask. The China standard “Determination of total chromium—Flame atomic absorption spectrometry (FAAS)” (HJ 491-2009) was referenced on determining total chromium. As to Cr(VI), the samples were processed with the alkaline digestion for hexavalent chromium, referring to U.S. EPA standard 3060A. As for the Cr(VI), it was determined by using the 1,5-diphenyl-carbohydrazide spectrophotometric method (referring to China national standard GB/T15555.4-1995). The quality for analytical accuracy was guaranteed by setting reagent blanks and inserting standard soil samples (China Standard Soil ESS-3).

In order to analyze the chemical species in COPR, the BCR sequential extraction procedure (Liu et al. 2013; Zheng et al. 2016) was taken in following steps.

1. Acid extractable fraction: 20 mL of acetic acid was added to 0.5 g sample and shaken 30 min with the use of ultrasonic cleaners. The mixture was centrifuged to separate the supernatant from the residue.
2. Reducible fraction: the residual from (1) was treated with 20 mL hydroxylamine hydrochloride, and the extraction performed as above.
3. Oxidizable fraction: 5 mL hydrogen peroxide was added to the sink fraction from (2) twice, evaporated to near dryness, and then treated with 25 mL of ammonium acetate, which was adjusted to pH 2 with nitric acid, and then the extraction performed as above.
4. Residual fraction: The materials remaining were digested with the acid mixture ($\text{HNO}_3:\text{HF} = 8:5$) with microwave assistance, same as above.

5. The method classified the modes of the trace element into four fractions: acid extractable, reducible, oxidizable, and residual. The fractions were analyzed for Cr by FAAS (HJ 491-2009).

2.4 Statistical analysis and spatial mapping

In order to identify the spatial distribution characteristics, the total Cr and the Cr(VI) in soil were first statistically analyzed by layer using MATLAB7.1. The statistical parameters included sample counts, minimum, maximum, median, mean, standard deviation and kurtosis. Secondly, total Cr and Cr(VI) were mapped by layers using the software Surfer10, and then these maps were cascaded to visualize the vertical trends.

3 Results

3.1 Spacial distribution

The data are listed in Table 1. The concentration of total Cr was between 110.5 and $21,774$ mg/kg, with the standard deviation varying from 231 to 5839 at different depths. The concentration of Cr(VI) was between 0.1 and 1075 mg/kg, with the standard deviation varying from 1.1 to 312 . The median was smaller than the mean, and the kurtosis was <3 , which showed that the content of chromium in the sample was mostly low, and the data was very discrete. There was a vertical trend of total Cr and/or Cr(VI) among different layers. The maximum of total-Cr appeared in the layers at depths ranging from -0.3 to -1.5 m. At the depths ranging from -1.5 to -6 m, total Cr decreased but it fluctuated with the change of depth. Cr(VI) was higher in the layers at the depth of -0.3 to -0.9 m than that in the deeper layers. The maximum of Cr(VI) mostly appeared in layers at these depths. Between -0.9 and -2.5 m in some borehole Cr(VI) was also very high. In the deeper layers (<-2.5 m) Cr(VI) was very low.

Some spatial distribution characteristics of total Cr and Cr(VI) are illustrated through the maps (Fig. 2). The distribution of total chromium is not uniform. At -0.3 m layer, the total Cr is more than 1000 mg/kg, and the maximum of total Cr is found in the middle and the northern area. At -0.6 and -0.9 m, the maximum appears in the middle and the southeastern part, and at the deeper layers a similar distribution appeared, but the total Cr decreased. In the northwestern and western areas of the COPR site, total Cr is low. Total Cr should be lower than 300 mg/kg in soil, referring to Environmental quality standard for soils (GB 15618-1995). Therefore, it can be concluded that the pollution is more serious in the middle

Table 1 Statistical list of total Cr and Cr(VI) in COPR

	Depth (m)	Sample counts	Minimum	Maximum	Median	Mean	SD	Kurtosis	
Total Cr	−0.3	12	131.8	4621	770.3	1371	1396	1.41	
	−0.6	12	110.5	21,774	461.1	2668	5839	3.31	
	−0.9	12	170.4	8153	492.8	1467	2314	2.45	
	−1.5	11	144.7	3692	391.7	965.2	1064	1.74	
	−2	11	167.9	4585	407.1	1178	1426	1.64	
	−2.5	10	143.6	3313	348	753.1	893.4	2.69	
	−3	9	234.9	2926	625.4	1120	964.6	0.89	
	−3.5	6	204.3	2254	934.5	1065	778.6	0.36	
	−4	6	306.6	3209	694.8	1142	1015	1.67	
	−4.5	6	243.7	896.9	514.8	522.3	246.4	0.28	
	−5	4	195.5	835.2	436.4	475.9	231.7	0.83	
	−6	2	586.9	1097	841.8	841.8	254.9	–	
	Cr(VI)	−0.3	12	0.3	408.9	9.1	45.8	110.4	3.37
	−0.6	12	1.2	518.5	4.3	49.1	141.7	3.45	
	−0.9	12	0.3	679.4	2.8	115.4	247.1	2.05	
	−1.5	11	0.7	287.4	4.6	47.5	86.28	2.34	
	−2	11	0.1	1075	3.4	135.5	309.5	2.92	
	−2.5	10	0.8	1050	4.5	116.1	312.1	3.14	
	−3	9	0.5	743.8	2	88.5	231.9	2.99	
	−3.5	6	0.6	579.2	2	107.6	211.9	2.4	
	−4	6	1.3	342.8	1.9	73.4	124.8	2.12	
	−4.5	6	1.3	4.7	2.4	2.6	1.14	1.04	
	−5	4	0.9	4.4	1.9	2.3	1.3	1.46	
	−6	2	1.2	4.6	2.9	2.9	1.71	–	

area than in the western part. At all layers, Cr(VI) concentration is high in the middle and eastern areas of the site. Although Cr(VI) is high at the layers in the −0.3 to −0.9 m depths, the ratio of Cr(VI)/total-Cr (%) is very noteworthy for it increased as the layers grew deeper. In other words, the Cr(VI) accounted for a greater proportion of the total Cr in the deeper layer of the site.

3.2 Chemical species

Samples were sequentially extracted according to European Community Bureau of Reference (BCR) (Fig. 3). The reducible and the oxidizable species of chromium were the major proportion at the depths from −0.3 to −4.0 m, but at this depth, the oxidizable and residual speciation were the major proportion. The oxidizable chromium and the residual species should be in the form of Cr(III), which was difficult to migrate; therefore, in the deeper layers, the Cr(VI) transforms into Cr(III). Additionally, the acid extractable chromium increased in the layers at depths ranging from −0.3 to −2.0 m but decreased in the deeper layers. It means that the movable Cr(VI) had a trend of migration to the deeper layers and then turned into Cr(III).

4 Discussion

The behaviors of Cr(III) and Cr(VI) strongly depend on pH and redox potential. Redox potential, pH, and moisture have been discussed in an effort to evaluate the role they played in the environment (Barnhart 1997; Rai et al. 1989; Sparks 2003). Theoretically, chromium exists in a number of oxidation states. However, in soil–water systems Cr(III) is the most thermodynamically stable specie because of the considerable energy required to convert it to either lower or higher oxidation states (Avudainayagam et al. 2003). In the presence of electron donors such as Fe²⁺, organic molecules with oxidizable groups, Cr(VI) is unstable and is easily reduced to Cr(III) (Rai et al. 1989). According to moisture in soils, soils can be divided into three categories, normal (oxidized), wet (seasonally saturated) and waterlogged (semi permanently saturated) (Sparks 2003). In acidic media, the high redox potential of the Cr(VI)/Cr(III) couple favors Cr(III) stabilization, while under alkaline conditions, Cr(VI) is relatively stable (Unceta et al. 2010) (Fig. 4).

In this work, the COPR was produced by high lime process and the buried COPR was under alkaline

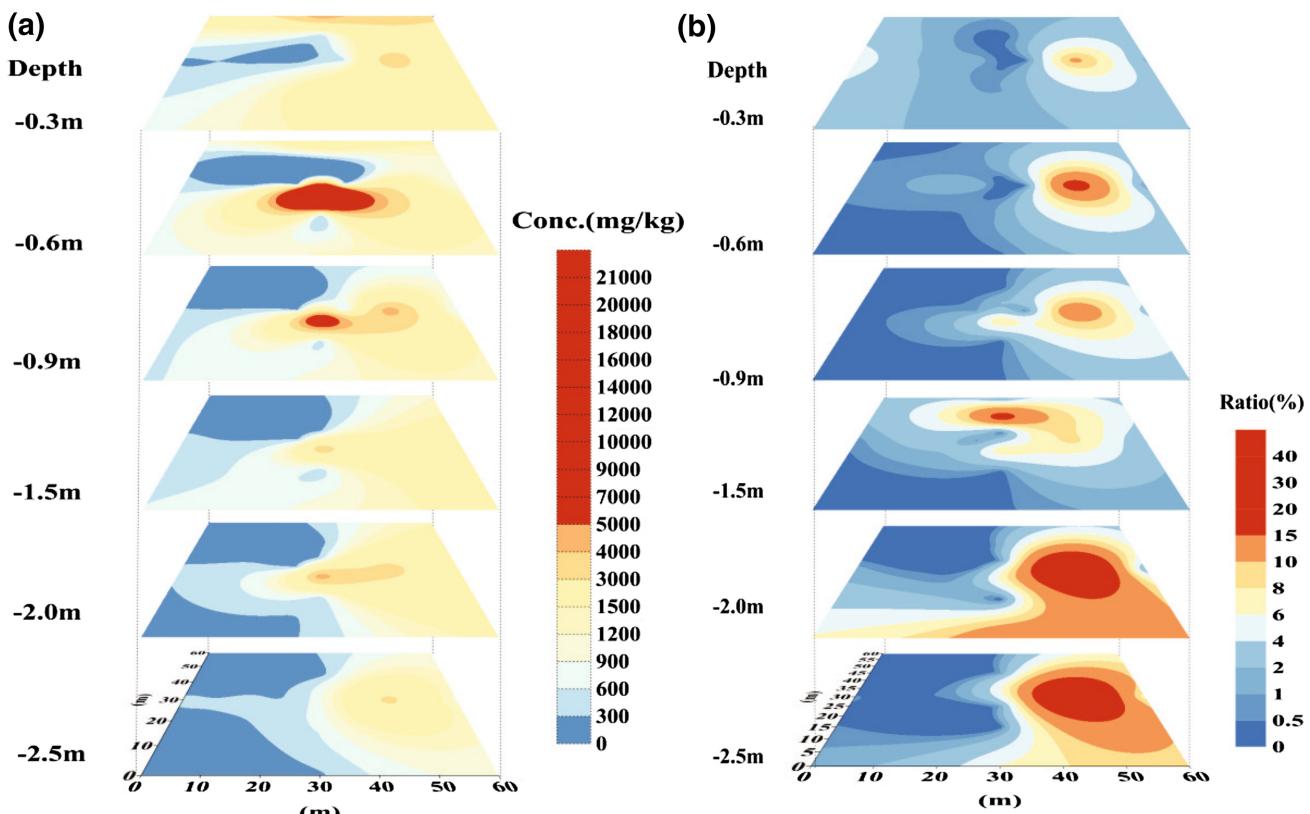


Fig. 2 Map of **a** Total-Cr(ppm) **b** Cr(VI)/Total-Cr (%) at the COPR site

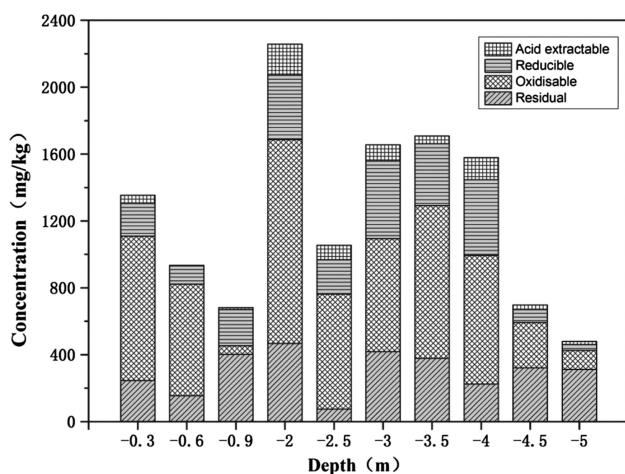


Fig. 3 Chromium species in the soil from the same drilling hole

conditions so that the Cr(VI) was relatively stable in the beginning. However, as time increased, the pH decreased and caused Cr(VI)(aq.) to be easily reduced to Cr(III)(s) and adsorbed by the solids. Due to gravity, the moisture was less in the surface layers than that in deeper layers. Therefore, the redox potential is higher in the surface layers (Sparks 2003).

The process of chromium migration should be as follows (Fig. 5). The remaining Cr(VI) in COPR mainly

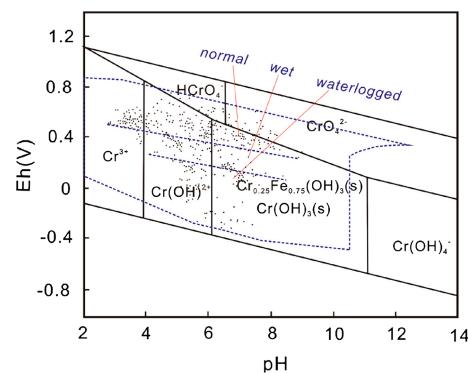


Fig. 4 An Eh–pH diagram of Cr in soils, which was adapted from Barnhart (1997), and the Eh–pH range from Sparks (Sparks 2003)

resides in hydrogarnet, hydrocalumite and ettringite and can be anionic exchanged (Hillier et al. 2003). Under natural conditions, Cr(VI) in COPR can be easily released into the soil solution after rainfall, and then gravity could drive the solution down to the deeper layers (Rhoades et al. 2015), and subsequently, a series of reactions would happen, including the redox reaction and the adsorption (Avudainayagam et al. 2003). Due to capillarity and evaporation of the soil moisture, Cr(VI) can migrate to and be enriched at the surface layer appearing yellow (Rhoades et al. 2015). After repeated rainfall and leaching, Cr(VI)

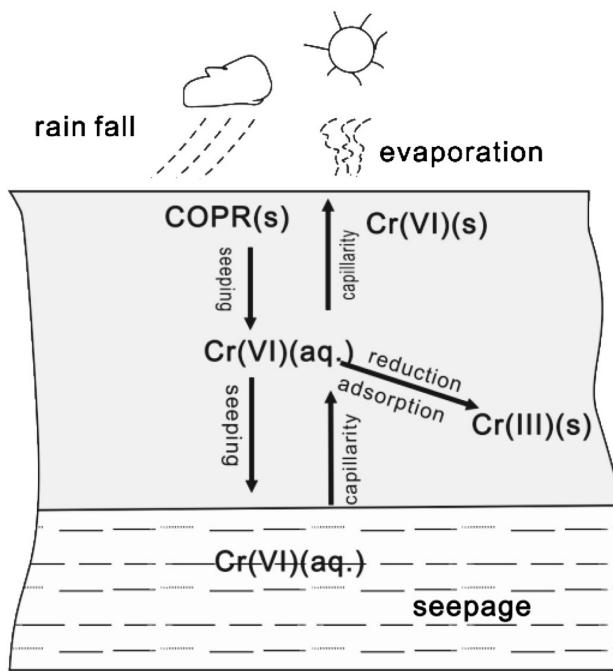


Fig. 5 Diagram of behaviors between Cr(VI) and Cr(III) in the COPR site

seeped into the deeper layers. Due to high positive redox potential of Cr(VI)/Cr(III), Cr(VI) is easily reduced to Cr(III) (Unceta et al. 2010). Soil colloid has a strong adsorption with Cr(III) (Azizian 1993). When the soil pH increased, the adsorption was enhanced so that more than 90% Cr(III) was quickly adsorbed by and fixed in the soil (Rai et al. 1989). In addition, the permeability of the soil was worse in the deeper layers, so the total Cr was highest at −0.3 to 0.9 m, while the ratio Cr(VI)/total-Cr (%) was greatest from −2 to −2.5 m. Therefore, water played an important role in Cr(VI) migration.

We have some suggestions for preventing Cr(VI) pollution. First, by coating the surface of the COPR pile, rainwater can be prevented from seeping into the COPR site. Also, the construction of a flood-control ditch at the western and northern boundaries can assist in the prevention of flood-waters from flowing into the site. Similarly, the source of groundwater can also be cut thus preventing it from flowing into the site. Furthermore, through constructing a concrete curtain at the boundary, the leachate can be halted from seeping into the neighboring Nanpan River. Finally, the seepage can be gathered and removed for special treatment.

5 Conclusions

Speciation and spatial distribution of Cr were studied in the chromite ore processing residue site, in Yunnan, China. The map of total-Cr and ratio of Cr(VI)/total-Cr (%)

showed that the maximum of total-Cr and Cr(VI) appeared in the layers near the surface. In the horizontal direction, the pollution was more serious in the middle and southeast part than that in the western part. In the layers above −4.0 m, reducible chromium and oxidizable chromium were the major proportion, but below this layer, the chromium was the major proportion in oxidizable and residual fractions. There was a trend that movable Cr(VI) migrated to the deeper layers and then turned into Cr(III). Water played an important role in Cr(VI) migration. Therefore, measures on controlling water movement should be adopted in there mediation of the COPR site.

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Author contributions Guangzhu Zhou designed the experiments and collected the samples. Jing Zhou and Weiyu Cheng performed the experiments. Xin Yin developed the figures and tables. Guangzhu Zhou and Xin Yin wrote the paper.

Compliance with ethical standards

Conflicts of interest The authors declare no conflicts of interest.

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