



# Treatment of chromium (VI) waste solutions with clay-based adsorbents after reduction with acid mine drainage

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## Synopsis

South Africa has one of the largest Cr-related industries in the world and generates large amounts of Cr-containing smelter dusts. It is normal practice to scrub Cr from the furnace off-gas in water, and to recover the Cr by adsorption, followed by recycling of the loaded adsorbent into the furnace. A study was conducted to find a low-cost adsorbent that is capable of removing sufficient Cr from the solutions generated by scrubbing. The test work focused on the evaluation of different clay minerals (attapulgite, bentonite, and kaolinite) as adsorbents for Cr. Cr(VI) could only be adsorbed after prereduction with e.g. ascorbic acid (ASA). An 80% removal of Cr(VI) could be achieved from a solution containing 20 mg/L Cr using bentonite clay after reduction with ASA. Attapulgite and kaolinite adsorbed less than 55% Cr after reduction with ASA. Both ASA and ferrous salts were found to be suitable reducing agents. The use of acid mine drainage (AMD) was also investigated as a low-cost alternative reducing agent, as AMD usually contains iron. The Cr(VI) reduction potential of the AMD was determined by redox titration against a solution containing 50 mg/L Cr(VI). Studies show that AMD can potentially be used as a reducing agent in a Cr removal process if it is available on site.

## Keywords

acid mine drainage, adsorbent, chromium, clay minerals, reduction.

## Introduction

South Africa possesses approximately 70–80% of the world's viable chromite ( $\text{FeCr}_2\text{O}_4$ ) reserves (Coetzee, Bansal, and Chirwa, 2018). The country is the world's leading producer of ferrochrome (FeCr), which is an important contributor to the gross domestic product. FeCr is a relatively crude iron-chromium alloy, used predominantly in the production of stainless steel. Despite the economic importance of the ferrochrome industry, there are concerns relating to waste generation. According to data from 2015, the South African ferrochrome smelting industry generated over 3.59Mt of ferrochrome with smelter charges containing 48–54% Cr (International Chromium Development Association, 2016). An enormous quantity of chromium-based waste was demonstrated to contain Cr(VI) concentrations above the maximum acceptable risk authorized for dumping (up to 3.5–4.5% chromium as  $\text{Cr}_2\text{O}_3$ ), both in liquid and solid form as chromium sludge (Georgaki and Charalambous, 2023). Certain Cr(VI) species are considered to be highly toxic, mutagenic, and carcinogenic. In particular, exposure to airborne Cr(VI) has been known to cause cancer of the respiratory system (Adhikari *et al.*, 2022). An environmentally friendly, cost-effective treatment for chromium-bearing waste materials is required to reduce the environmental impact.

The main Cr reserves of South Africa are located in the Bushveld Complex, and are strongly associated with platinum group metal (PGM) ores. After PGM extraction, the fine by-product material is physically upgraded to > 40%  $\text{Cr}_2\text{O}_3$  (> 27% Cr) before being used as smelter feed. The non-valuable material is exposed to oxidative conditions in waste dumps with large surface areas. Table I shows the amount of Cr(VI) contained in the smelter dusts from a local ferrochrome plant.

These values represent a high pollution potential and the capture and safe immobilization of chromium therefore deserves attention. It is normal practice to scrub chromium from furnace off-gas in water, and to recover the chromium by adsorption, followed by recycling of the loaded adsorbent into the furnace. This can also add value by improving overall process recoveries.

The dust generated by ferrochrome plants typically consists of coarse dust from the cyclones (which is lower in grade since the feed material is upgraded in the cyclone) and fine (normal) dust from the open, submerged arc furnace, which is captured by the baghouse filters (Sedumedi *et al.*, 2009). The cyclone and furnace dusts are sent to the slimes dam. The dried clay from the slurry is the slimes dust (du Preez *et al.*, 2023). These dusts are classified as hazardous wastes by the US Environmental Protection Agency,

**Table I**  
**Typical total Cr(VI) content (g/t) in ferrochrome smelter dusts from a local ferrochrome plant (Sedumedi *et al.*, 2009)**

| Sample    | Cyclone (coarse) dust | Normal (fine) dust | Slime dust  |
|-----------|-----------------------|--------------------|-------------|
|           | g Cr/t dust           | g Cr/t dust        | g Cr/t dust |
| Furnace 1 | 16.4                  | 2520               | 5632        |
| Furnace 2 | 29.9                  | 1686               | 6658        |
| Furnace 3 | 43.5                  | 2043               | 7277        |
| Furnace 4 | 24.8                  | 2713               | 7856        |

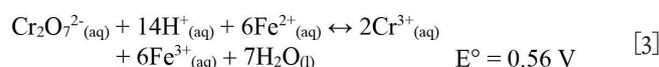
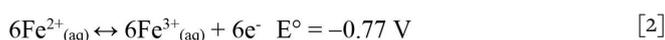
as they contain hexavalent chromium, Cr(VI) (Sedumedi *et al.*, 2009). The fine dust mostly consists of agglomerated particles that comprise oxide, metallic, and carbon-based phases, while the coarse dust mainly consists of oxide and carbon-based particles that are irregular in shape (Sedumedi, 2009).

Furthermore, Cr(VI) is formed in small quantities as an unintended by-product during production of FeCr (Coetzee, Bansal, and Chirwa, 2018; Beukes, Dawson, and van Zyl, 2012; Govender-Ragubeer, 2012). The remediation of Cr(VI) from mining activities is accomplished through landfilling in licensed and managed landfills, recycling, solidification, and stabilization. Prior to post-treatment activities, the pollutants in processed and leachate water are required to be reduced to environmentally safe levels. The commonly used effective reducing agents include ferrous sulphate, sulphur dioxide, or sodium disulphite, to reduce soluble and mobile Cr(VI) to Cr(III), which is deemed less toxic (Minas, Chandravanshi, and Leta, 2017). However, these chemicals are expensive and may contribute to water pollution.

Adsorbents that have been tested include zeolites, as well as clay minerals such as attapulgite, bentonite, and kaolinite. The benefits of using clay minerals such as attapulgite and kaolinite as adsorbents include their large specific surface area, cation exchange capacity, and the fact that they do not swell (Murray, 2000). Mankge and Govender-Ragubeer (2014) evaluated the treatment of Cr(VI) by fresh ferrous salts and ascorbic acid. The results revealed that it would be cheaper to reduce Cr(VI)-containing dust with ferrous salts than ascorbic acid. However, the addition of fresh ferrous-containing salts poses environmental risks. Ascorbic acid (ASA) is more expensive, but is biodegradable and environmentally benign.

In an attempt to find a cheaper reductant with minimal environmental consequences, it was proposed to investigate acid mine drainage (AMD). AMD is particularly problematic in South Africa in gold and coal mining areas in the Witwatersrand Basin and Witbank Coalfield (Sakala *et al.*, 2021; Minnaar, 2020). However, chrome mines and the majority of platinum mines do not generate acid-producing wastes.

The reduction of Cr(VI) by Fe(II) and AMD is described by Equations [1] to [3]. The standard redox potential for the overall reaction (Equation [1]) is 0.56 V against the standard hydrogen electrode (SHE) scale (Mankge and Govender-Ragubeer, 2014).



The aim of this study was to investigate the adsorption of Cr(VI) from oxidized solutions by clays. The pre-reduction of Cr(VI) to Cr(III) with ASA and Fe(II) salt was also investigated. The use of AMD as a novel reductant was subsequently tested.

### Experimental procedure

#### Clay adsorption of Cr(VI) from oxidized solutions

Potassium dichromate ( $\text{K}_2\text{Cr}_2\text{O}_7$ ) was used to produce the synthetic Cr(VI) solutions (20 or 100 mg/L). For comparison, Cr(III) solutions (20 or 100 mg/L) were also prepared using Cr(III) nitrate nonahydrate ( $\text{Cr}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ ). Experimental conditions are summarized in Table II. All tests were carried out at 25°C over a period of 24 hours in Erlenmeyer flasks at an adsorbent dosage of 94 g/L. Samples were periodically collected and analysed by ICP-OES (inductively coupled plasma-optical emission spectroscopy). Redox potential was measured using a Crison ELP 21 Eh meter against a Ag/AgCl reference electrode (3 M KCl). The pH was measured using a Metrohm 713 pH meter. The attapulgite ( $(\text{Mg},\text{Al})_2\text{Si}_4\text{O}_{10}(\text{OH}) \cdot 4\text{H}_2\text{O}$ ) used in this study was sourced from Matutu clay mine and two samples, A (-300 µm) and B (-1400+ 500 µm), were received from Incubex Minerals.

**Table II**  
**Experimental conditions: chromium adsorption with attapulgite from Cr(VI) and Cr(III) solutions**

| Tests | Attapulgite |   | Chromium species |               |
|-------|-------------|---|------------------|---------------|
|       | A           | B | Cr(VI), mg/L     | Cr(III), mg/L |
| 1     | X           |   | 20               |               |
| 2     |             | X | 20               |               |
| 3     | X           |   |                  | 20            |
| 4     |             | X |                  | 20            |
| 5     | X           |   | 100              |               |
| 6     |             | X | 100              |               |
| 7     | X           |   |                  | 100           |
| 8     |             | X |                  | 100           |

# Treatment of chromium (VI) waste solutions with clay-based adsorbents after reduction with acid mine drainage

*Table III*  
**Test matrix for adsorption of reduced Cr(VI) to Cr(III) using attapulgite**

| Test | Ascorbic acid % | Cr(VI) mg/L | Clay mineral |           |               |               | H <sub>2</sub> O soaked |
|------|-----------------|-------------|--------------|-----------|---------------|---------------|-------------------------|
|      |                 |             | Bentonite    | Kaolinite | Attapulgite A | Attapulgite B |                         |
| 1    | 0.03            | 20          | X            |           |               |               |                         |
| 2    | 0.03            | 20          |              | X         |               |               |                         |
| 3    | 0.03            | 20          |              |           | X             |               |                         |
| 4    | 0.03            | 20          |              |           |               | X             |                         |
| 5    | 0.03            | 20          | X            |           |               |               | X                       |
| 6    | 0.03            | 20          |              | X         |               |               | X                       |

## Clay adsorption of Cr(III) from reduced solutions (ASA and Fe(II) salts)

Previous studies (Mankge and Govender-Ragubeer, 2014; Govender-Ragubeer, 2013) indicated that ascorbic acid could reduce Cr(VI) to Cr(III) within 15 minutes, with > 99% reduction. In this study, Cr(VI) solutions were prerduced with ascorbic acid, followed by adsorption in shake flasks with attapulgite, kaolinite, and bentonite. The test matrix is summarized in Table III. Control tests were conducted by soaking kaolinite and bentonite in water, in order to allow for a possible increase in pore size prior to the start of the experiment. The tests were run over a 24-hour period, at an adsorbent dosage of 94 g/L. The powdered bentonite (Al<sub>2</sub>O<sub>3</sub>·4SiO<sub>2</sub>·H<sub>2</sub>O) and kaolinite (Al<sub>2</sub>O<sub>3</sub>·2SiO<sub>2</sub>·2H<sub>2</sub>O) were purchased from Sigma Aldrich South Africa.

Four tests were conducted in order to determine the effect of surface modification on the capability of bentonite to adsorb chromium. During the preparation of the surface-modified bentonite, 9.38 g of bentonite was added to 100 mL solutions containing either 0.5 M and 1 M Fe(II) or ascorbic acid solution.

*Table IV*  
**Test matrix for chromium adsorption using surface-modified bentonite**

| Test | Ascorbic acid M | Fe(II) M | Cr(VI) mg/L |
|------|-----------------|----------|-------------|
| 1    | 0.5             | -        | 20          |
| 2    | 1               | -        | 20          |
| 3    | -               | 0.5      | 20          |
| 4    | -               | 1        | 20          |

Synthetic Fe(II) was prepared from ferrous sulphate (FeSO<sub>4</sub>·7H<sub>2</sub>O). The mixtures were placed in a shaking incubator operated at 110 r/min at room temperature (25°C) for 24 hours. After 24 hours the modified bentonite was dried in an oven (30°C) for 24 hours. The test matrix used in this study is summarized in Table IV.

## Reduction with AMD

Additional tests were carried out to investigate AMD as a novel and unconventional reducing agent. AMD was sourced from a gold mine water purification plant in Randfontein. The pH of the AMD solution was adjusted from 6.52 to 1.71 using sulphuric acid (H<sub>2</sub>SO<sub>4</sub>) to prevent iron precipitation. The pH of AMD close to mining sites is typically highly acidic (pH 2 to 4) (Yuan *et al.*, 2022). The corresponding redox potential was 400 mV (*vs.* AgCl/AgCl, 3 M KCl). A sub-sample was submitted for analysis by ICP-OES for Al, As, Ca, Co, Cr, Cu, Fe, Mg, Mn, Mo, Ni, Pb, S, Si, and Zn, and for Fe(II) by potassium dichromate titration (Table V). The Fe concentration was 460 mg/L, similar to that reported by Moyo (2019) for a Witbank AMD sample. A 25 mL sub-sample was measured out and poured into a 200 mL beaker for redox titration. A 50 mg/L Cr(VI) solution was then added to the AMD sample. Both pH and redox potential were recorded at each Cr(VI) addition. This was continued until the redox potential plateaued. The point at which the redox potential plateaus is considered the end-point.

## Results and discussion

### Clay adsorption of Cr(VI) from oxidized solutions

The results of the clay adsorption tests on the Cr(VI) and Cr(III) synthetic solutions are summarized in Figure 1. Attapulgite could not adsorb Cr(VI) at either 20 or 100 mg/L, hence these curves are not shown. Maximum adsorptions of > 99% were observed in the tests with Cr(III) at both 20 and 100 mg/L concentrations; however, desorption started occurring after 5 hours.

*Table V*  
**Chemical composition of AMD (mg/L)**

| Constituent mg/L | Mg  | Al | Si | Ca  | Cr | Mn | Fe(II) | Fe(T) | Ni | Cu | Zn | Mo | Pb | Co | As | S    |
|------------------|-----|----|----|-----|----|----|--------|-------|----|----|----|----|----|----|----|------|
| Randfontein AMD  | 172 | <2 | <2 | 634 | <2 | 51 | 460    | 476   | <2 | <2 | <2 | <2 | <2 | <2 | <2 | 1722 |

# Treatment of chromium (VI) waste solutions with clay-based adsorbents after reduction with acid mine drainage

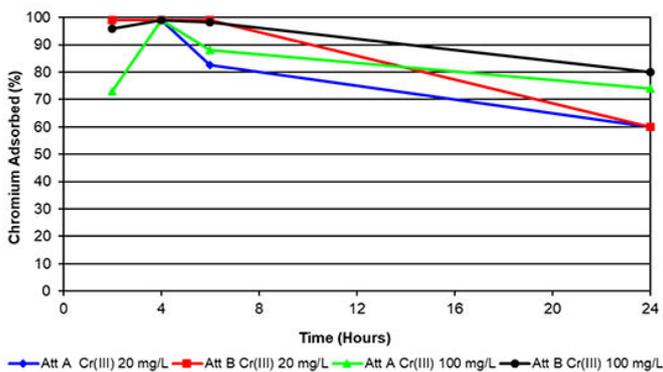


Figure 1—Chromium adsorption using attapulgite

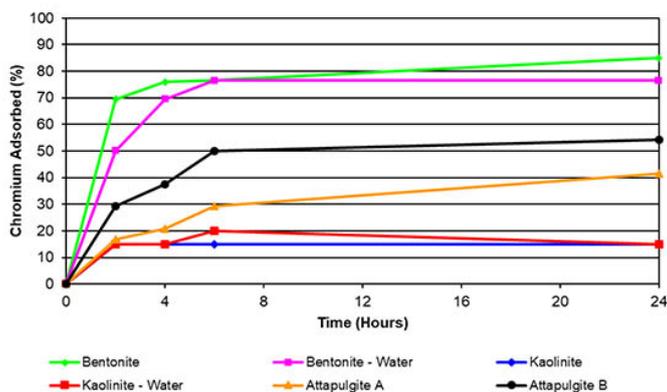


Figure 2—Cr(III) adsorption using attapulgite, kaolinite, and bentonite, after reduction of Cr(VI) with ASA

## Clay adsorption of Cr(III) from solutions reduced using ASA and Fe(II)

The potential use of the clay minerals as adsorbents for reduced Cr(VI) was evaluated (Figure 2). Clay minerals usually possess high surface area (Murray, 2000) which is beneficial for the adsorption of heavy metals such as Cr from solution. In this study, the ability of attapulgite, kaolinite, and bentonite to adsorb Cr(VI) reduced with ASA was tested. The results are illustrated in Figure 2.

Although complete Cr removal was not obtained, bentonite was capable of adsorbing between 76-85% of the reduced Cr(VI). Soaking of kaolinite and bentonite in water prior to the tests did not result in a marked difference (< 5%) in the adsorption capability of the clay minerals.

Cr(VI) reduced to Cr(III) with ASA could be easily adsorbed by bentonite since bentonite has a strong cation exchange capacity and adsorption capacity. Similar results were observed by Zhao (2008) and Muhammad (2004), and bentonite has been shown to be the best adsorbent for metal ions (Tahir and Naseem, 2007; Kaya and Oren, 2005).

It was therefore decided to combine Cr(VI) adsorption with bentonite and reduction in the same process. Four tests were conducted in order to determine the effect of Fe(II) and ASA surface modification on the capability of bentonite to adsorb chromium. The results indicate that the use of ASA during surface modification was more effective than Fe(II) (Figure 3). It is speculated that the ASA was first adsorbed into the bentonite pore structure, where it reduced Cr(VI) to Cr(III).

## Chromium-ferrous ions titration

A 50 mg/L Cr(VI) titrant was added to a 25 mL AMD sample in order to determine the amount of Cr(VI) that can be reduced to Cr(III) ion by the Fe(II) in the AMD.

Figures 4 to 6 show the chromium-ferrous ion titration curves at different pH levels. Plotted on the primary x-axis is the cumulative volume of titrant (50 mg/L Cr(VI)) added. The corresponding amount of Cr(VI) in moles calculated from the titrant volume added is plotted on the secondary y-axis. The redox potential of the solution mixture is plotted on the primary y-axis. The end-point is identified by the plateau of the redox curve. Hence the ratio of Cr(VI) added in moles to the moles of Fe(II) in the original AMD solution can be determined and is plotted on the secondary x-axis.

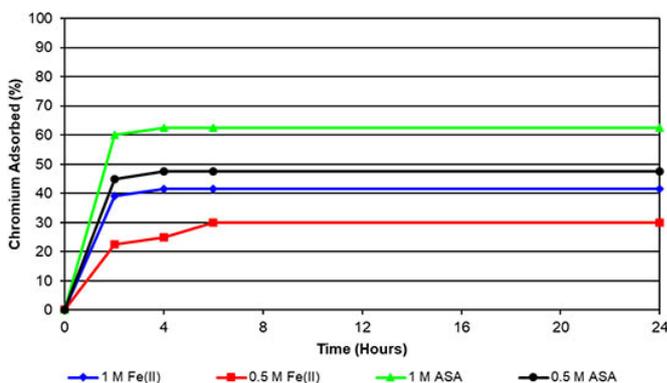


Figure 3—Chromium adsorption using bentonite modified with Fe(II) and ascorbic acid

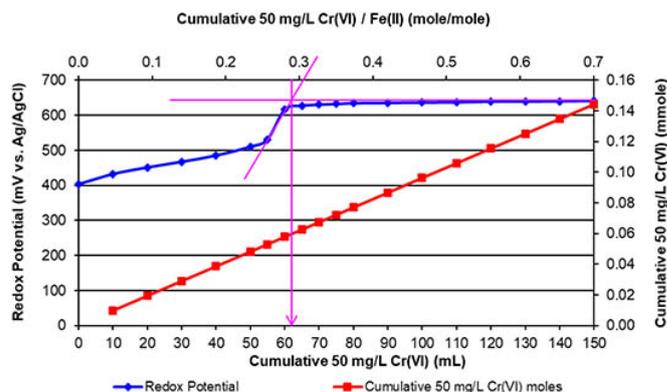


Figure 4—Chromium-ferrous ion titration at 50 mg/L Cr(VI), pH 1.8

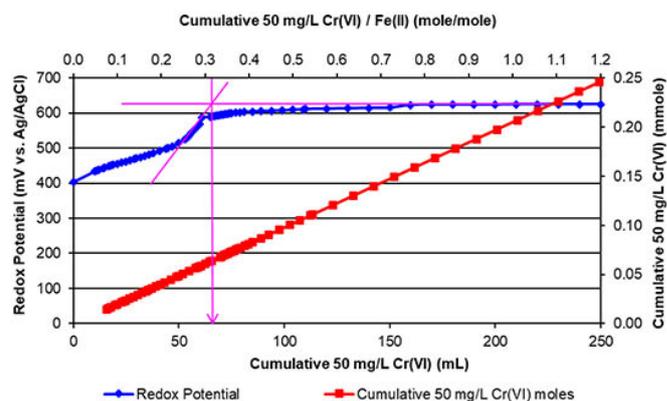


Figure 5—Chromium-ferrous ion titration at 50 mg/L Cr(VI), pH 4.5

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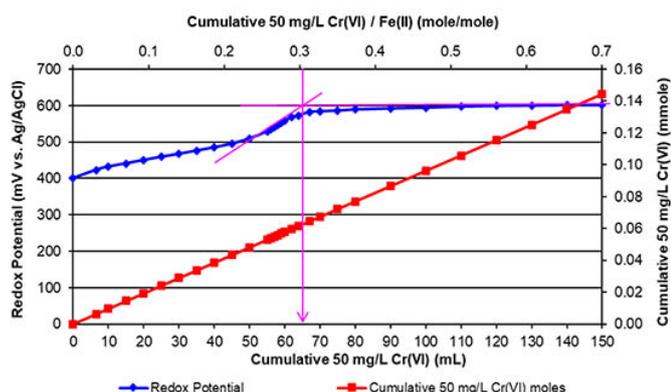


Figure 6—Chromium-ferrous ion titration at 50 mg/L Cr(VI), pH 8

The redox potential was plotted against cumulative 50 mg/L Cr(VI) for pH values of 1.8, 4.5, and 8. The respective end-point reaction ratios (mol Fe(II) : mol Cr(VI)) are 3.45, 3.12, and 3.20. These are the inverse of the ratios shown on the secondary x-axis. The average reaction ratio is 3.26, which is close to the theoretically expected value of 3 as indicated by Equation [3].

## Conclusions,

The results indicate that attapulgite, bentonite, and kaolinite could not adsorb Cr without prerduction of Cr(VI) to Cr(III). Bentonite adsorbed 80% Cr(III), whereas attapulgite and kaolinite achieved less than 55% adsorption after 24 hours. Surface modification of bentonite by 1 M ascorbic acid resulted in 62% Cr(VI) removal.

It was not possible to achieve sufficient loading (of the same order of Cr content as chromite furnace feed) to be able to consider this process for Cr recycle to FeCr furnaces. Although promising results were obtained using bentonite for removal of reduced Cr(VI), the clay-based material presented handling challenges after the adsorption process, which will need to be addressed if the process were to be scaled up.

The local gold mine acid mine drainage contained 460 mg/L ferrous ion (470 mg/L total iron) and less than 2 mg/L chromium. The average equilibrium Fe(II) to Cr(VI) molar ratio required for Cr(VI) reduction was 3.26, which is close to the stoichiometric ratio of 3. It can be concluded that AMD is a potential reducing agent for the treatment of Cr(VI)-containing dust if it is available on site.

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