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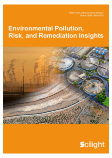

Article

Influence of Immobilisation Agents on the Leaching Behaviour of Copper, Arsenic, and Chromium from Chromated Copper Arsenate-Treated Timber

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Abstract: Chromated copper arsenate (CCA) is widely used globally as a waterborne inorganic wood preservative, acting as a fungicide and insecticide to prevent timber damage. Toxic metals (Cr, As, and Cu) can leach into the environment, posing risks, and a thorough understanding of the immobilisation agents used to prevent leaching is lacking. This study systematically investigated the leaching behaviour of As, Cr, and Cu from fresh and weathered CCA-treated timber in block and mulch forms under simulated rainfall conditions. The effectiveness of selected immobilisation agents (iron sulfate, bentonite, iron powder, and steel wool) was evaluated for their ability to lock up and immobilise toxic metal(loid)s from leaching out via a column study. Column experiments were conducted over 15 weeks to quantify metal(loid)s release and treatment performance. Compared with fresh timber, weathered timber exhibited sustained and greater metal(loid)s leaching, with arsenic showing the most significant mobility. Among the immobilisation agents tested, iron powder and steel wool markedly reduced metal(loid)s leaching, resulting in a >99% reduction in As and substantial reductions in Cr and Cu. Speciation, SEM–EDS, and XRD analyses revealed that zero-valent iron promoted the reduction of Cr(VI) to Cr(III) and facilitated adsorption, co-precipitation, and the incorporation of As and Cr into newly formed iron (oxyhydr)oxide and mixed Fe–As–Cr mineral phases, thereby decreasing metal mobility. Notably, steel wool treatment increased both bacterial and fungal diversity in the timber mulch, indicating reduced toxicity following immobilisation. Overall, this study demonstrates that iron-based amendments, particularly steel wool, provide a practical, mechanistically robust approach for immobilising toxic metals in CCA-treated timber, supporting safer, risk-based management and reuse strategies.

Keywords: CCA-treated timber; leaching of trace elements; immobilisation agent; iron; arsenic; chromium

1. Introduction

Chromated copper arsenate (CCA) is a water-borne inorganic wood preservative used to prolong the lifetime of timber, such as radiata pine (*Pinus radiata*). The copper (23–25%), 38–45% chromium, and arsenic (30–37%)

in CCA act as fungicides and insecticides, whereas the chromium fixes both chemicals into the wood to prevent damage to timber by insects (termites, borers, beetles), wood rot, and wood fungus [1]. As a result, CCA-treated wood is less susceptible to natural degradation, and the service time can last approximately 30–40 years [2]. Therefore, CCA-treated timber has been commonly used for outdoor structures, such as telegraph poles, decking and fencing, landscaping, building structures, playground equipment, and marine structures since the 1930s [3–5]. Two-thirds of the 500,000 tons of wood preservatives used annually are CCA [6].

Metal(loid)s, including copper chromate ($CuCrO_4$), chromium(III) arsenate ($CrAsO_4$), copper chrome arsenate ($Cu(OH)CrAsO_4$), copper(II) arsenate ($Cu_3(AsO_4)_2$), and a variety of metal complexes with lignin and cellulose, are found in CCA-treated timber [4,7]. There has been growing concern among the public about the toxicity of Cu, Cr, and As in treated timber [7]. Cr(VI) is classified as a carcinogen by the ITRC, implicating enough evidence to conclude that it can cause cancer, and various toxic effects are associated with exposure to Cr, e.g., respiratory toxicity, renal toxicity, immunotoxicity, and reproductive and developmental toxicity [8]. Similarly, exposure to As contamination can cause cancer, neurotoxicity, hepatotoxicity, nephrotoxicity, and various diseases associated with As [9].

The environmental receptors and humans could be exposed to toxic metal(loid)s by direct contact through wood cutting, drilling, touching, or disposal of used treated timber waste. Recent studies have shown that toxic metal(loid)s can leach into the environment during their application in the field and cause adverse risks to human health and the environment [10,11]. High levels of total As ranging from 101.3–166.6 mg/kg were found in proximity to and underneath CCA-treated structures after 16 years of installation, and these levels increased to 143.4–213.5 mg/kg after 26 years of installation [12]. Mercer and Frostick reported that the peak concentrations of As, Cr, and Cu were 1885 μg/L, 1243 μg/L, and 1261 μg/L, respectively, in their lysimeter study [13,14]. The concentrations of Cr, Cu, and As in soil samples collected from CCA-contaminated sites in Korea were reported to be 50.56–94.13 mg kg^{-1}, 27.78–120.83 mg kg^{-1}, and 0.13–9.43 mg kg^{-1}, respectively [15]. Overall, soil heavy metal concentrations decrease with increasing distance from the CCA-treated wood structure. Studies have indicated that As concentrations in precinct soils can reach up to 500 mm horizontally from stacked posts [16]. Several studies have investigated the leaching properties of this waste and the potential risk to environmental receptors [7,11]. Significant levels of As, Cr, and Cu can leach from CCA-treated timber into the environment, especially over long-term applications [5,17].

As a result, regulators have restricted or phased out the use of CCA-treated timber in some domestic and industrial applications. For example, a partial ban on the use of CCA as a preservative in the UK was introduced in 2004 and in several other countries, including Japan, Indonesia, Sweden, and Germany [13,17]. Concerns about arsenic residues in CCA-treated timber led CCA manufacturers in the US to voluntarily phase out CCA timber for non-industrial applications in 2003 [18]. In Australia, several restrictions on the CCA issued by the Australian Pesticides and Veterinary Medicines Authority (APVMA) have been enacted since March 2006. CCA is also prohibited for the treatment of timber for structural uses, where frequent and intimate contact occurs, such as playground equipment, picnic tables, handrails, decking boards, garden furniture, and exterior seating [5]. In New South Wales (NSW), Australia, wood preservation treatment plants must hold an environmental protection license and are regulated under the NSW Protection of the Environment (Operations) Act 1997 [19,20].

Although the use of CCA has recently decreased and been limited, the disposal of CCA-treated wood is likely to remain a significant concern because of its long service life and the large volume accumulated annually. In South Australia, life disposal volumes are predicted to reach 160,000 m^3 per year by 2040 [2]. Different physical and chemical treatment processes have been utilised to remediate CCA-treated timber waste [21]. Currently, several potential options for the recycling and reuse of CCA-treated timber, including landfill disposal, energy recovery by burning, mulching, particleboard production, and metal extraction, are under investigation [7,22,23]. However, few of these methods have been technically or practically applied at any significant scale due to concerns of the release of toxic metal(loid)s into the environment during utilisation or disposal processes [21,24]. An increasing number of reports have demonstrated this concern under both laboratory and field conditions [3,13,25,26]. Moreover, the pyrolysis of CCA wood was investigated for its ability to generate bio-oil and char [27], with all the metals identified in the biogas, bio-oil, and char. A monofil is proposed for the management of CCA timber to separate the leaching of metals from normal landfills, potentially providing temporary storage of CCA timber prior to sustainable technologies for the removal of metals for recycling [28]. Several decontamination methods, including pyrolysis and solvent/chelate extraction [29,30], chemical leaching/extraction [31,32], electro-dialytic remediation [33], are used to remediate CCA-treated timber waste.

Despite extensive studies on CCA-treated timber leaching and disposal, limited attention has been given to immobilisation approaches that directly reduce metal(loid) leaching during reuse or interim management [5]. In particular, the systematic evaluation of low-cost iron-, steel-, and clay-based immobilisation agents under simulated rainfall conditions and their influence on metal speciation and microbial responses remain largely unexplored. The leaching behaviour of metals from CCA-treated timber is critical for the development of utilisation or management strategies that either minimise or maximise leaching [5,17]. This study investigated the leaching behaviours of Cu, Cr, and As from fresh and used CCA-treated timber in either block or mulch forms under simulated rainfall conditions. Moreover, several materials were tested to investigate their effects on locking and immobilising heavy metals to prevent leaching. The immobilisation of heavy metals in soils has been intensively investigated, and reduced bioavailability and risks have been demonstrated after immobilisation [34–36]. Similar research is also applicable to agricultural soils [37,38]. However, no such efforts have been made to examine toxic metal leaching behaviour in CCA timber. Thus, the primary aim of this study was to assess the effectiveness of selected immobilisation agents in reducing the leaching of Cu, Cr, and As from fresh and weathered CCA-treated timber under controlled rainfall simulation. The specific objectives are to (i) compare the leaching behaviours of metal(loids) from timber in block and mulch forms; (ii) evaluate the performance of iron sulfate, iron powder, steel wool, and bentonite in immobilising metal(loid)s; and (iii) investigate changes in metal speciation and microbial diversity following treatment. This study evaluated the ecological response of CCA-treated timber following immobilisation by assessing shifts in the microbial community structure. Microbial analyses were undertaken to determine whether reductions in metal bioavailability translate into measurable biological recovery, using bacterial (16S rRNA) and fungal (ITS) diversity as indicators of residual toxicity. Microbial assessment focuses on community diversity and richness as proxies for alleviating ecological stress and ensuring environmental safety. This approach links physicochemical immobilisation performance to biological risk reduction, supporting the evaluation of CCA-treated timber reuse and management options. This study represents a trial of CCA treated timber management. This study explores a potential remediation platform for CCA-treated timber, thereby expanding its safe utilisation.

2. Materials and Methods

2.1. Sources and Characterisation of the CCA-Treated Timber

The used CCA-treated timber was collected from City Works Depot and processed at the Newcastle City Council's Summerhill Waste Management Centre (Newcastle, NSW, Australia). All the soil and metal parts were removed before the timber was shredded into mulch. The used timber was treated with H5 grade CCA oxide type C (01), containing 34.0% As2O5, 47.5% CrO_3, and 18.5% CuO. Fresh CCA-treated radiata pine (*Pinus radiata*) timber (Iron Wood Landscaping 134 01 H4) was purchased from Bunnings Warehouse, Wallsend, NSW, Australia, for use as a reference control.

The samples were shredded by a commercial jaw shredder into small pieces (<150 mm) prior to further shredding to coarse size A (<30 mm) and finer size B (<15 mm) (Figure S1 in Supplementary Materials). Both sizes were used for a column study to investigate the leaching behaviours of toxic metal(loid)s from CCA-treated timber mulch. Both fresh and used blocks were shredded and pulverised with a coffee grinder to <2 mm. The powder sample was dried in an oven at 60 °C for 24 h before analysis for As, Cu, Cr, and Fe concentrations and speciation (As/Cr) via ICP-MS. Block CCA timber was also used for the leaching study. For comparison, fresh CCA timber purchased from Bunnings (grade H4) was used in the block leaching studies.

The characterisation of the CCA timber waste was conducted by analysis of toxic metal(loid)s (total), the morphology of the metal distribution in the timber, and laboratory leaching tests. In particular, <2 mm mulch was used to analyse the total metal concentration (As, Cr, and Cu) via ICP-OES or ICP-MS, followed by acid digestion [39,40]. A small piece of CCA timber H5 post was minced to a length of 65 mm, carbon glued onto the sample stage of SEM (scanning electron microscopy), and carbon coated, as shown in Figure S2. The sample was then analysed via a Philips XL30 SEM (FEI Company, Eindhoven, The Netherlands) equipped with an Oxford ISIS electron dispersive spectroscopy (EDS) system (Oxford Instruments, High Wycombe, UK) for morphological analysis and via a Philips XL30 SEM equipped with an Oxford ISIS electron dispersive spectroscopy (EDS) system for elemental composition analysis.

Shredded CCA timber mulch <15 mm in size was further reduced to less than 2 mm or 8 mm in size via an electronic coffee blender for leaching tests. Bench leaching tests, including the Toxicity Characteristic Leaching Procedure (TCLP, US EPA1311), Synthetic Precipitation Leaching Procedure (SPLP, US EPA1312), and Australia Standard Leaching Procedure (ASLP, AS4439), were conducted to determine the leaching characteristics of toxic metal(loid)s from timber. The TCLP was designed to assess the mobility of heavy metals present in CCA-treated wood under simulated landfill conditions. The SPLP was intended to evaluate the mobility of heavy metals in stockpile waste, where the leaching potential due to natural rainfall/weathering could be determined. The ASLP employed a much finer-sized timber powder than did the TCLP and SPLP (2 mm for the ASLP and 8 mm for the TCLP and SPLP).

2.2. Characterisation of Remediation Agents

Iron sulfate ($FeSO_4$), bentonite clay, iron powder, and steel wool were used as immobilisation agents and tested for their ability to reduce the leachability of toxic metal(loid)s. Iron sulfate was selected as the reducing agent and purchased from Sigma-Aldrich (Sydney, NSW, Australia). Similarly, iron powder and steel wool were used as reducing agents. Iron powder (C100.29) was purchased from Australian Metal Powders Supplies (Melbourne, VIC, Australia). The chemical composition is 98.5% Fe, 0.8% O, 0.2% C, and trace amounts of S and P. The density is between 2.4–2.6 g/cm^3, and all the iron particles are less than 180 µm, with 57% falling into the 75–150 µm range. Fine-grade 00–0 (SCA brand) steel wool was obtained from Supercheap Auto (Newcastle, NSW, Australia). Bentonite clay was selected because of its high surface area and cation exchange capacity (CEC). Sibelco provided bentonite clay under the commercial name Trubond MW, a sodium bentonite mined near Miles in Queensland. The Trubond MW contains a high proportion of montmorillonite with a CEC of 82 (meq/100 mL). The mineral compositions of the iron powder, bentonite, and steel wool were characterised via X-ray diffraction (XRD) (Philips X'Pert MPD XRD (PANalytical B.V., Almelo, The Netherlands)).

2.3. Column Leaching Study

A schematic of the rain simulator column setup is shown in Figure S3a. The control columns consisted of 2 kg of CCA-treated timber mulch or blocks (fresh or used) in a 10 L plastic bucket with holes drilled in the bottom. Two meters of water weeper, curved into an Archimedean spiral, was fixed to the column cover as a rainfall simulator (Figure S3c). For the mulch columns, a layer of polyethylene-based geotextile was placed at the bottom of each column; the geotextile was tested to prevent adsorption of the metal(loid)s of interest during the leaching test. A total of 1.5 L of potable water was dripped onto the whole surface of the column in the rainfall simulator weekly during the test. The leachate was collected and analysed for elemental concentration via ICP-MS. Blocks of fresh and waste CCA timber (2 kg) were submerged in 7.5 L of water in a bucket to investigate the leaching of toxic metals.

For the columns with remediation agents, 44.3 g of iron powder or 2 kg of bentonite were mixed with shredded mulch, and 44.3 g of steel wool was placed at the bottom of the column as a layer of filtering and reducing agent. The $FeSO_4$ (120 g) was dissolved in 500 mL of Milli-Q water and sprayed onto 2 kg of mulch.

The fresh CCA samples were tested for As, Cr, and Cu concentrations by ICP, which were 2320, 2590, and 1460 mg/kg, respectively. The block samples were denoted "Block Fresh" (fresh CCA timber purchased from Bunnings) and "Block waste" (block waste CCA timber sampled from the Summerhill Waste Management Centre). The leaching test lasted for 15 weeks. Tap water was sprayed from the top of the column, and leachate samples were collected periodically for analysis of toxic metals. Details for the column setup are shown in Table S1.

2.4. Analytical Methods for Heavy Metals and Microbial Analysis

All liquid samples were filtered through 0.45 µm filters prior to ICP–MS analysis. As species (arsenite and arsenate) were separated via high-performance liquid chromatography (HPLC) via an anion-exchange column (Hamilton PRP-X100). Each species was quantified by ICPMS using a single ion at *m/z* 75. Hexavalent chromium is determined directly on a water sample by a discrete analyser as received by pH adjustment and colour development via diphenylcarbazide. Each sample run was measured against a five-point calibration curve. The Australia Laboratory Services (ALS), a NATA-accredited laboratory, was used for the analysis of metal(loid)s and speciation. QA/QC protocols were followed in accordance with ALS internal QA/QC procedures, including the analysis of reference samples, surrogates, laboratory duplicates, and spikes.

Upon completion of the leaching study, timber mulch samples from columns L3, L7, and L9 were collected for microbial diversity analysis via 16S rRNA sequencing using the Illumina HiSeq platform (AGRF, Adelaide). The samples were stored at −80 °C for examination. CCTAYGGGRBGCASCAG was the forward primer sequence (341F), and 806R was the reverse primer for sequencing the bacterial 16S rRNA gene with 16SV3V4. CCTAYGGGRBGCASCAG was the forward primer (341F), whereas GGACTACNNGGGTATCTAAT was the reverse primer (806R). ITS gene targets were employed for fungal analysis using the ITS1F-ITS2 primer pair CTTGGTCATTTAGAGGAAGTAA and GCTGCGTTCTTCATCGATGC. Microbial biodiversity was measured to determine deterioration. Microbial biodiversity was measured to estimate CCA timber degradation after fixation.

3. Results and Discussion

3.1. Characterisation of CCA Timber Waste

3.1.1. Toxic Metal Concentrations in the CCA Timber

The waste mulch samples (<2 mm) showed very high concentrations of all three elements, with average concentrations of As, 2410 mg/kg; Cr, 2996 mg/kg; and Cu, 1723 mg/kg. These are above CT4 and below CL4 Class IV in landfills, which require leaching tests [41]. Table S2 in the SI summarises the concentrations of elements for waste and fresh CCA timber, indicating the wide range of metal(loid)s concentrations in various waste timbers. Fresh CCA timber contains fewer toxic metal(loid)s than waste timber because the level of timber treatment for fresh timber (H4) is one level less than that for waste timber (H5). The higher the level, the stronger the timber protection and the higher the preservative level. The variations in metal concentrations depend on the year of service, the initial loading of preservatives, and the types of preservatives used. The highest concentrations of Cr and As identified are greater than 10,000 mg/kg and 7000 mg/kg, respectively, for timber waste [13,42].

3.1.2. Surface Morphology and Elemental Analysis of the CCA Timber

An energy dispersive spectroscopy (EDS) line scan was executed from the outer edge to the center core of the CCA-treated post to investigate the distribution of CCA heavy metal(oid)s in the lateral direction (Figure S2). In total, 23 scans were conducted to cover the entire length of the sample, from the edge to the center. In each scan, the photo width was 3 mm, with 100 detection points in the lateral direction. A total of 69 mm of sample length was covered from the outer edge to the central point. The EDS patterns of all 23 scans were consistent, as were the edge, middle, and core of the CCA (Figure 1).

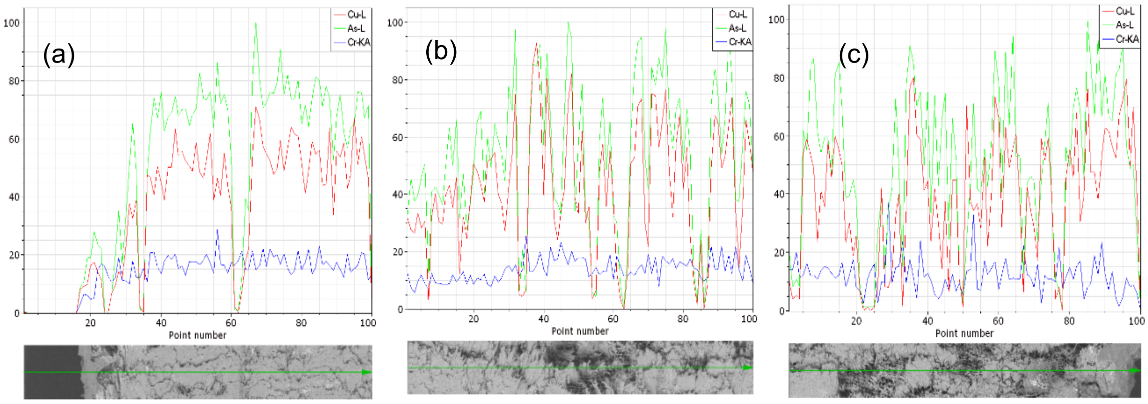


Figure 1. Lateral distributions of Cu, As, and Cr in the CCA-treated samples from the outer perimeter (**a**); middle (**b**); and core (**c**) by EDS and SEM. The length of each section was 30 µm on the horizontal axis, and the vertical axis was the EDS count, which was directly proportional to the CCA metal concentration.

The EDS line-scan spectra showed that CCA concentrations along the lateral direction were largely consistent. There was no noticeable decrease in the CCA concentration from the core of the CCA-treated post (Figure 1c) to the outer perimeter (a), apart from 1 mm from the outer edge (a). The CCA post's outer edge presented a decrease in the CCA concentration (Figure 2a), which indicated that the leaching of the CCA heavy metal(loid)s first started from the outer surface during their service. The roughness in the spectra occurred when the electron beam hit the honeycomb-shaped void in the timber texture (Figure S2). The presence of metal(loid)s in the cross-section of the timber pole helps determine their potential for treatment/utilisation. The relatively even distribution of metal(loid)s is due to the fixation process when the CCA solution is loaded into timber products, which ensures that virtually all the CCA becomes chemically bonded within the wood structure. This poses challenges for timber treatment/utilisation. Chemical and biological extraction of metal(loid)s alone rarely achieves a high level of removal of metal(loid)s [43–46], and pyrolysis of CCA timber leads to the occurrence of metal(loid)s in all fractions for biogas, bio-oil, and char residue [47].

3.1.3. Leaching Characterisation

Lab leaching studies using TCLP, SPLP, and ASLP were conducted to determine the amount of toxic metals leached. Table 1 shows the concentrations of the leached metals. These concentrations are within the range of toxic metals leached, as reported in the literature for CCA timber [4,26]. TCLP revealed greater Cu leaching than As and Cr leaching, whereas SPLP and ASLP showed that As leached at a higher rate. The higher rate of Cu leaching by the TCLP was due to the more readily complex formation of Cu with citric acid. The leaching percentages were 4.3–12.1% for Cu, 5.7–7.6% for As, and 1.2–2.3% for Cr in the tests. ASLP resulted in the highest extraction of As and Cr. The leaching solution of As is above ASLP3 and below ASLP4, and the total concentration of As is above CT4 and below CL4 [41]. This suggests that the ASLP is required for a Class IV landfill site and that the leachate solution must be specified in the site licence.

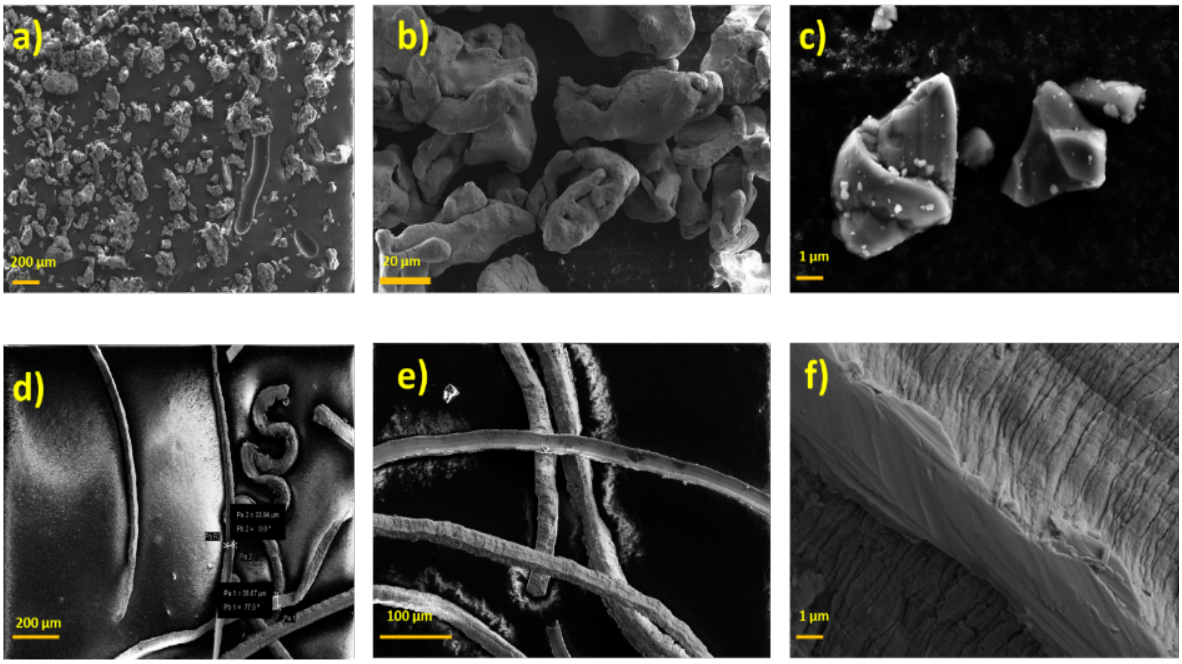


Figure 2. SEM images of iron powder (**a**–**c**) and steel wool (**d**–**f**) from lower to higher magnification.

Table 1. Leaching of toxic metals via standard leaching methods.

Leaching Method	**Leaching Concentration and Percentage**		
	Cu (mg/L)/%	**As (mg/L)/%**	**Cr (mg/L)/%**
SPLP	3.7/4.3	6.9/5.7	1.8/1.2
TCLP	10.4/12.1	7.8/6.5	2.4/1.6
ASLP	5.9/6.8	9.1/7.6	3.5/2.3

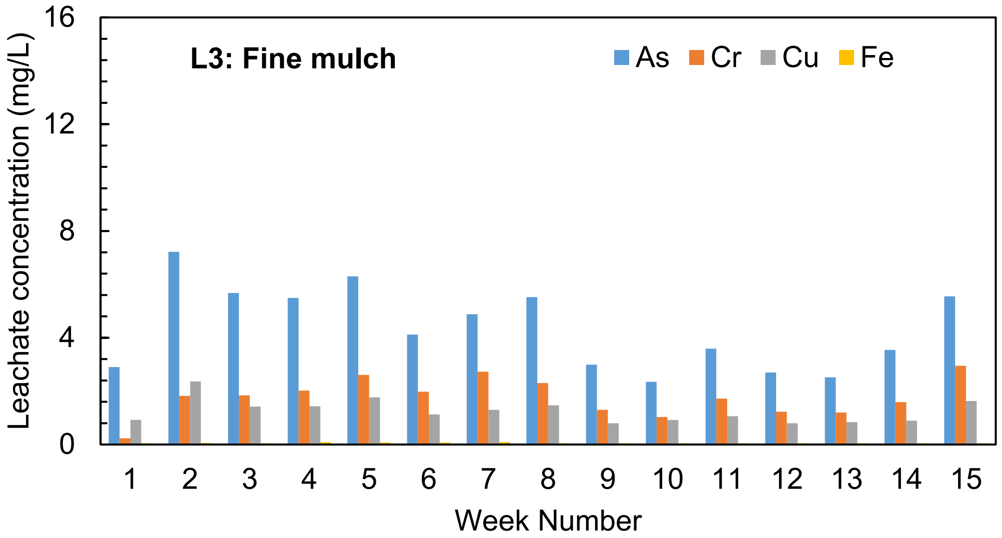
3.2. Surface Morphology Analysis of the Immobilisation Agent

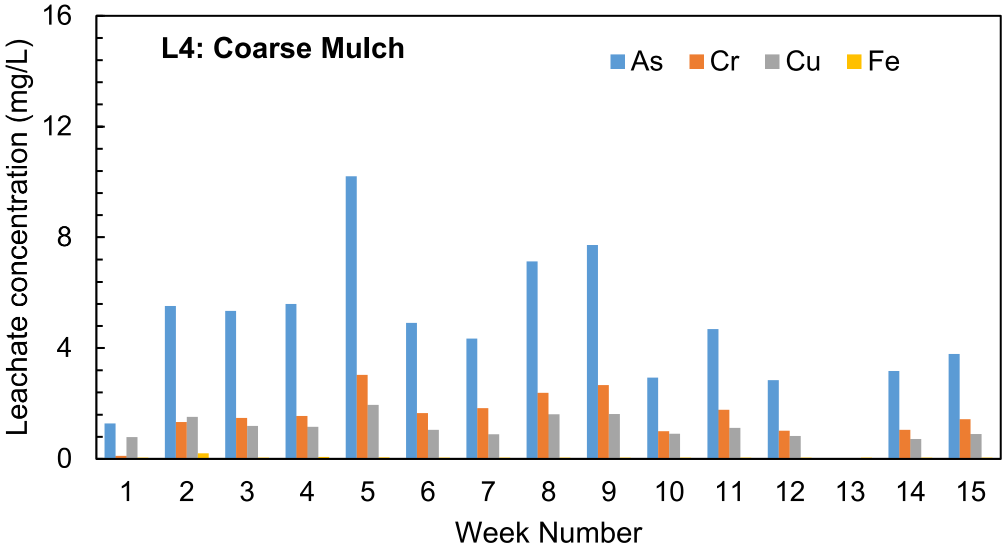
SEM images and EDS spectra of the iron powder and steel wool samples are presented in Figures 2 and S4. The iron powder had a rough surface covered with fine scales with diameter ranging from 12.0 to 134.5 µm. The steel wool has a hair-like structure, straight or serpentine, with a relatively uniform diameter of 33 µm and various lengths (Figure 2). Iron powder contains mainly iron with a tiny amount of oxygen. Similarly, steel wool also contains iron as the dominant elemental component. The EDS spectra of both steel wool and iron powder indicated that they were of high iron purity, with minor impurities detected (Figure S4).

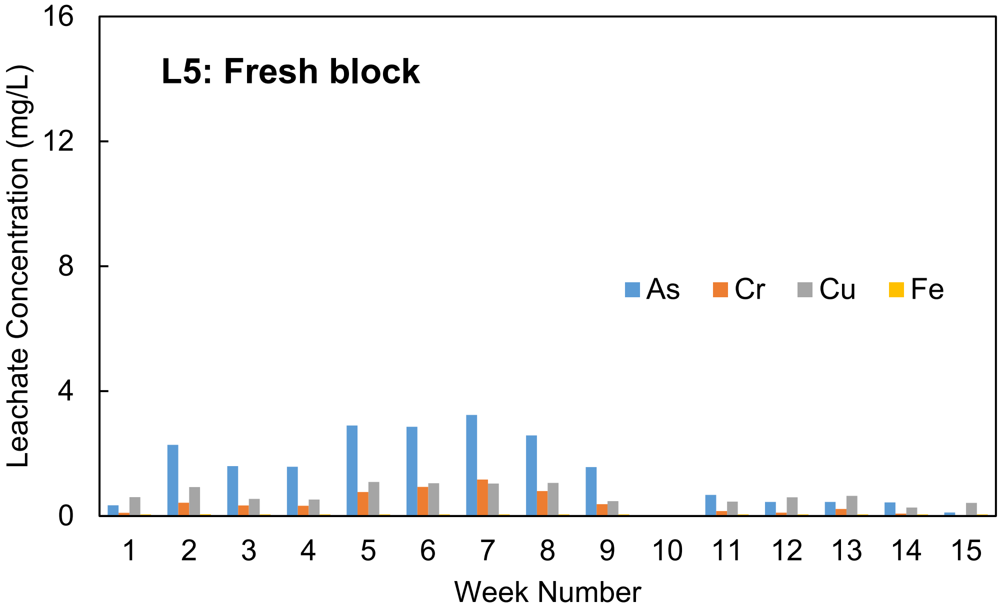
3.3. Leaching and Immobilisation Behaviours of CCA Metals in the Presence and Absence of Immobilisation Agents

3.3.1. Column Leaching Characteristics (Control)

The leaching test was designed to assess the leaching rate of metal(loid)s from CCA-treated timber, either in the original unshredded form or in shredded mulch form, to determine whether shredding CCA-treated wood accelerates metal(loid)s leaching. Figure 3 shows the leaching concentrations of As, Cr, Cu, and Fe from the mulch and block control columns.


L3: Fine mulch
As
Cr
Cu
Fe
Leachate concentration (mg/L)
0
4
8
12
16
1
2
3
4
5
6
7
8
9
10
11
12
13
14
15
Week Number


L4: Coarse Mulch
As
Cr
Cu
Fe
Leachate concentration (mg/L)
0
4
8
12
16
1
2
3
4
5
6
7
8
9
10
11
12
13
14
15
Week Number


L5: Fresh block
As
Cr
Cu
Fe
Leachate Concentration (mg/L)
0
4
8
12
16
1
2
3
4
5
6
7
8
9
10
11
12
13
14
15
Week Number

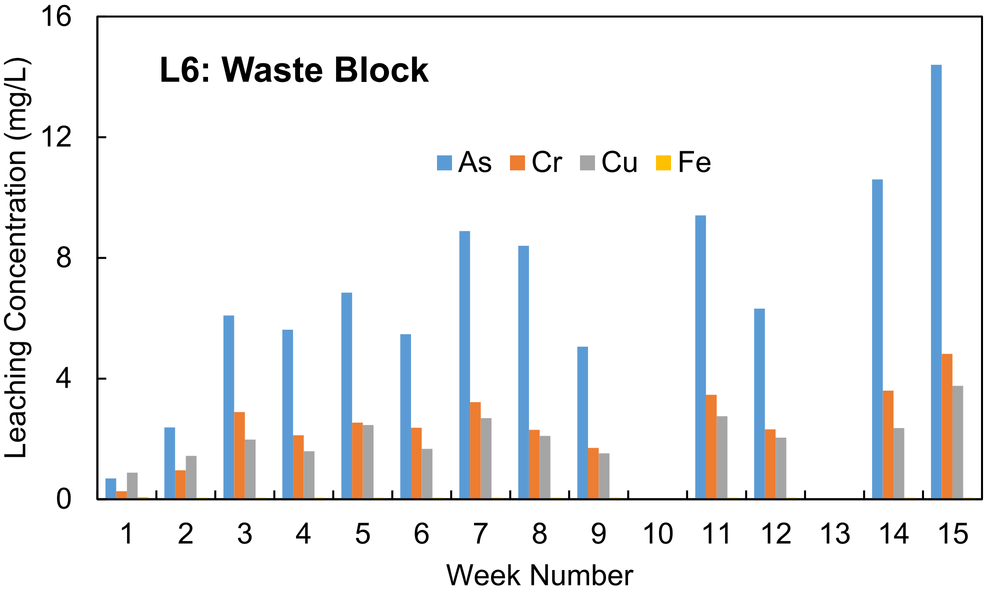


Figure 3. Leaching profile of CCA metals in fine (L3) and coarse mulch (L4) from waste timber and block dripping samples (L5 and L6) without fixing agents.

As shown in Figure 3, leaching of As, Cr, Cu, and Fe from the fine- and coarse-mulch columns that were not remediated began immediately after tap water was sprayed. Leaching fluctuated throughout the experiment. Arsenic had a higher leaching concentration than did Cr and Cu. Negligible amounts of Fe were detected in the leachate due to the limited amount of Fe in the original CCA timber waste. No evident trend in leaching over time emerged, while the concentrations of As, Cr, and Cu ranged from 1.28–10.20 mg/L, 0.11–3.04 mg/L, and 0.72–2.36 mg/L for fine mulch and coarse mulch, respectively.

In week one, the finer mulch leached 2.90 mg/L As, 0.23 mg/L Cr, and 0.92 mg/L Cu, which was twice the concentration of CCA in leachate from the coarse mulch and almost four times the concentration in leachate from the original CCA-treated post (block waste) for As. However, over the subsequent 11 weeks, there were no significant differences in the leachate concentrations of all three heavy metals among the three samples (i.e., fine, coarse mulch, and original block samples). Despite the large differences in sample size and surface area, no significant differences were detected in the average CCA concentrations in the leachate from the fine, coarse mulch, and original (waste) block samples. However, the amount leached each week (leaching rate) varied for different samples. The leaching rates for the mulch samples were fairly consistent throughout the leaching period, whereas block leaching rates increased. Boxplots comparing the toxic metals leached from block timber, fine mulch, and coarse mulch are shown in Figure S5. Comparable leaching of As, Cr, and Cu was found for the coarse mulch and fine mulch. However, leaching of toxic metals from the timber block resulted in a wider range of concentrations, with median values higher than those in the coarse and fine mulches. These results suggested that the size and surface area did not significantly increase or decrease the CCA leaching rate from either mulch or the original block. Therefore, only fine mulch was selected for the fixation study, which is described in the following sections.

Compared with the waste block samples, the fresh block samples presented much lower leaching concentrations of toxic elements (Figure 3). A slight decrease in the leaching rate was observed for the fresh block sample, whereas the block waste samples showed an increase. For comparison, leaching percentages were calculated from the concentrations and total amounts of toxic metals, as shown in Figure 4a,b. The fresh block samples exhibited leaching of 0.02–0.43% As, 0.00–0.17% Cr, and 0.06–0.24% Cu, whereas the waste block samples exhibited leaching of 0.10–2.02% As, 0.03–0.54% Cr, and 0.18–0.75% Cu during the experiment. The boxplots for the leaching range (percentages) of toxic metals from fresh and waste block samples (L5 and L6) are shown in Figure S6. A higher leaching percentage was observed in the waste block samples than in the fresh block samples.

The timber blocks (5 kg each for fresh and waste blocks) were submerged in 7.5 L of water to leach the toxic metals. Leachate samples were collected weekly, and the water was changed at weeks 4, 7, 11, 13, and 15. The leaching concentrations of toxic metals from waste and fresh timber are shown in Figure 5. The concentration ranges leached from waste timber for As, Cr, and Cu are 6.54–45.5 mg/L, 5.17–22.5 mg/L, and 3.96–14.2 mg/L, respectively, which are much higher than those derived from the block-dripping samples. Arsenic is leached at a higher rate than Cr and Cu from waste timber. The concentration increased steadily for weeks 4–6 and weeks 7–10, which may be attributed to the lack of change in water during those periods. This indicated the continuous leaching of toxic metals, and a longer equilibration time may be required to achieve leaching equilibrium.

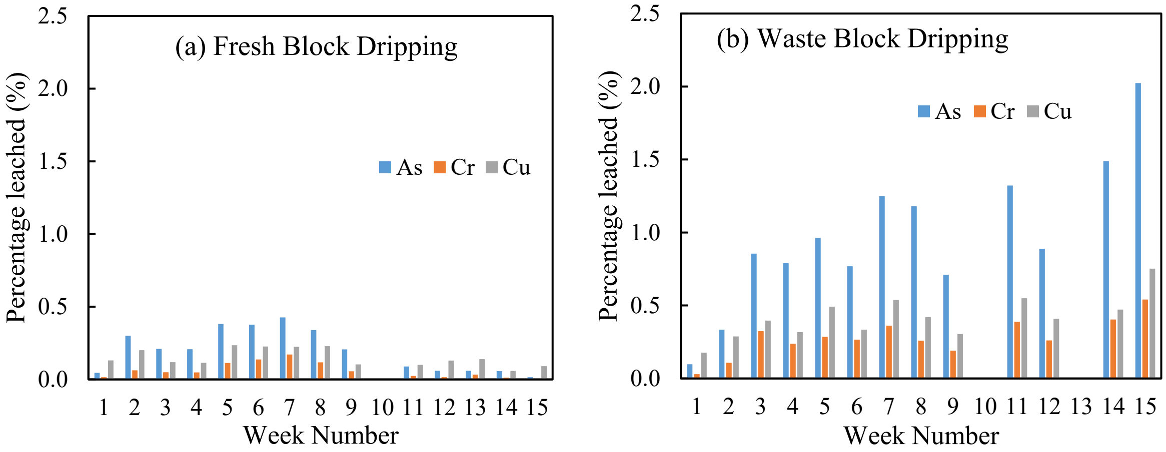


Figure 4. The percentage of toxic metals leached from fresh and waste block timber during dripping.

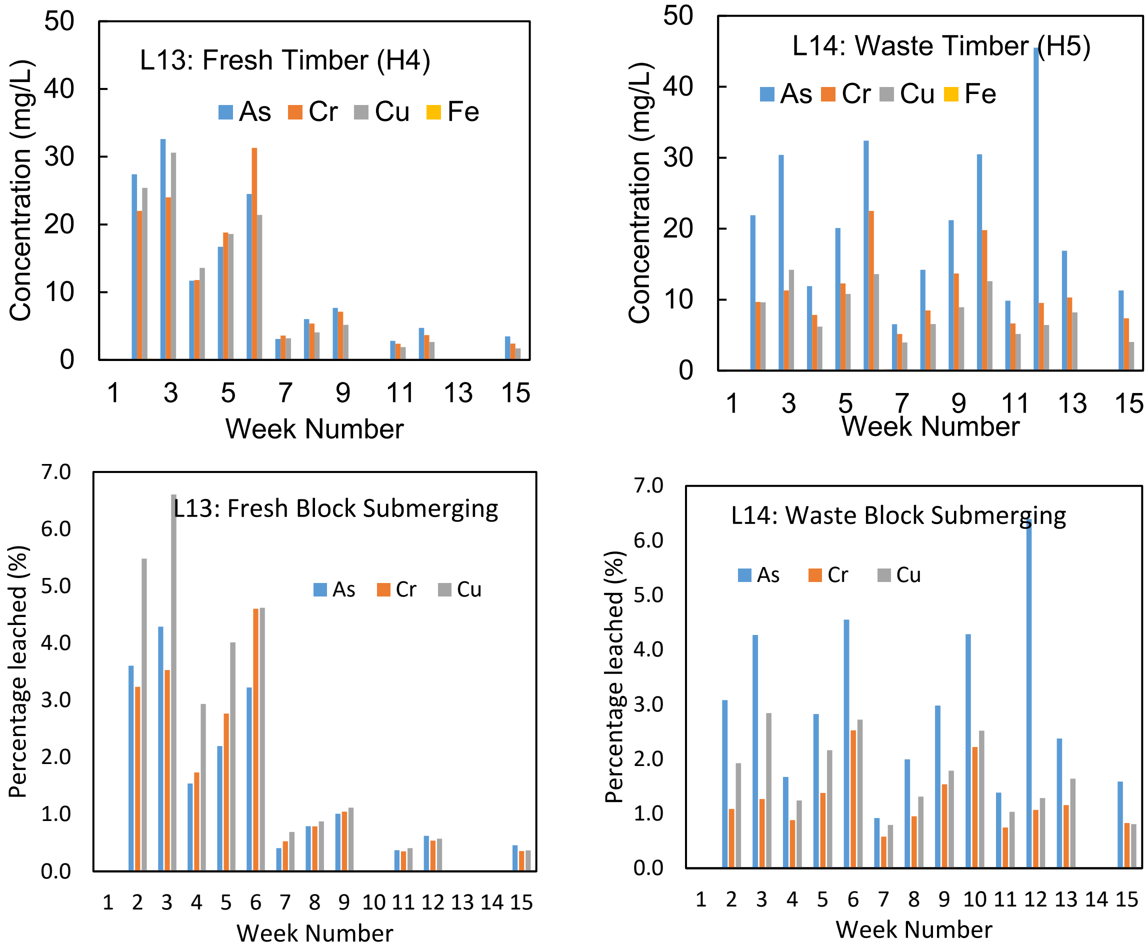


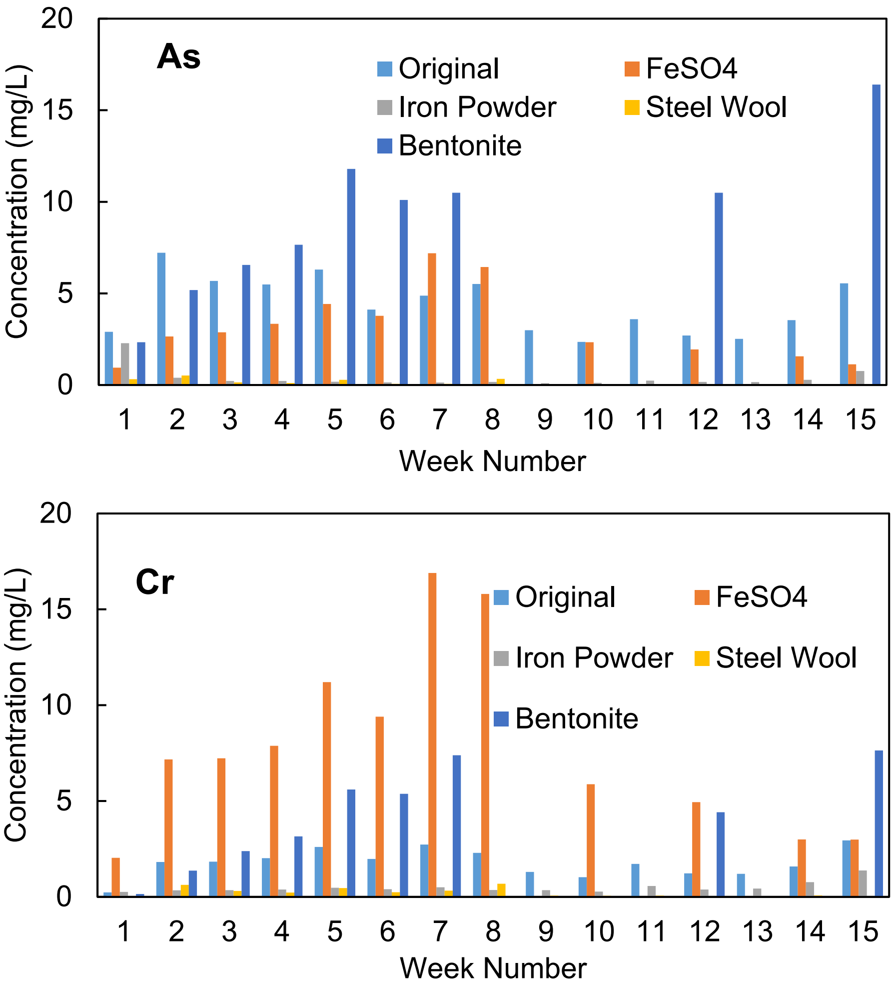
Figure 5. Leaching profiles and percentages of toxic metals in the original CCA timber block submerged samples (Fresh L13 and Waste L14).

The concentrations of the toxic metals leached from the fresh block ranged from 2.82–32.6, 2.38–31.3, and 1.71–30.6 mg/L for As, Cr, and Cu, respectively. A steady, increasing trend was observed at weeks 4–6 and 7–10. However, unlike the overall increase in concentration, the leachable concentration decreased when the water was changed. Similar results were obtained when the percentages of leached toxic metals were plotted (Figure 5). The leaching percentages ranged from 0.37–4.29% As, 0.35–4.60% Cr, and 0.37–6.60% Cu for the fresh block (L13) and 0.92–6.39% As, 0.58–2.52% Cr, and 0.79–2.84% for the waste block (L14). A lower concentration and percentage of leaching for the metal(loid)s are observed for the fresh timber, suggesting that the weathered wood could continuously leach toxic metal(loid)s, which implies that a stronger complexation or bond is involved in fresh wood after the initial leaching of a portion of the free metal(ions). A longer-term leaching study would help understand the weathering effect.

The control column experiments demonstrate that CCA-derived metal(loid) leaching occurs rapidly upon water exposure, regardless of timber form, confirming the inherent mobility of these preservatives under rainfall conditions. Arsenic consistently exhibited higher leachate concentrations than chromium and copper, reflecting its weaker fixation within the wood matrix and greater environmental mobility. Although finer mulch initially released higher metal concentrations due to increased surface area, long-term leaching profiles converged across the fine mulch, coarse mulch, and block samples, indicating that particle size alone does not govern cumulative metal release. The increasing leaching trends observed in weathered timber blocks compared with fresh timber further highlight the role of aging and prior environmental exposure in destabilising CCA complexes. These findings establish a robust baseline against which the effectiveness of immobilisation treatments can be evaluated and confirm that untreated CCA timber represents a sustained source of metal(loid) release over time.

3.3.2. Effect of Remediation Agents on Leaching

Iron powder, ferrous sulfate, and steel wool act as reducing agents when reacting with CCA metals, whereas bentonite clay, with its high cation exchange capacity, is expected to adsorb and retain the leached CCA heavy metals. Figure 6 shows the leaching profiles of As, Cr, and Cu when fixing agents are added to the columns. When either steel wool or iron powder was added to the CCA mulch, the leachate concentrations of As, Cr, and Cu decreased dramatically. On average, greater than 99% decreases in As concentrations were recorded for both iron powder and steel wool; 76% for Cr in iron powder, 49% for steel wool, and 73% for Cu in iron powder and 86% for steel wool. However, the addition of ferrous sulfate increased the leaching of Cr and Cu and resulted in the leaching of a large amount of Fe. The As leached from the column, which was remediated with ferrous sulfate, was initially lower than that from the untreated sample, but the levels were similar at later stages. A boxplot comparison (Figure S7) indicated much lower leaching of As and Cr after treatment with iron powder and steel wool.



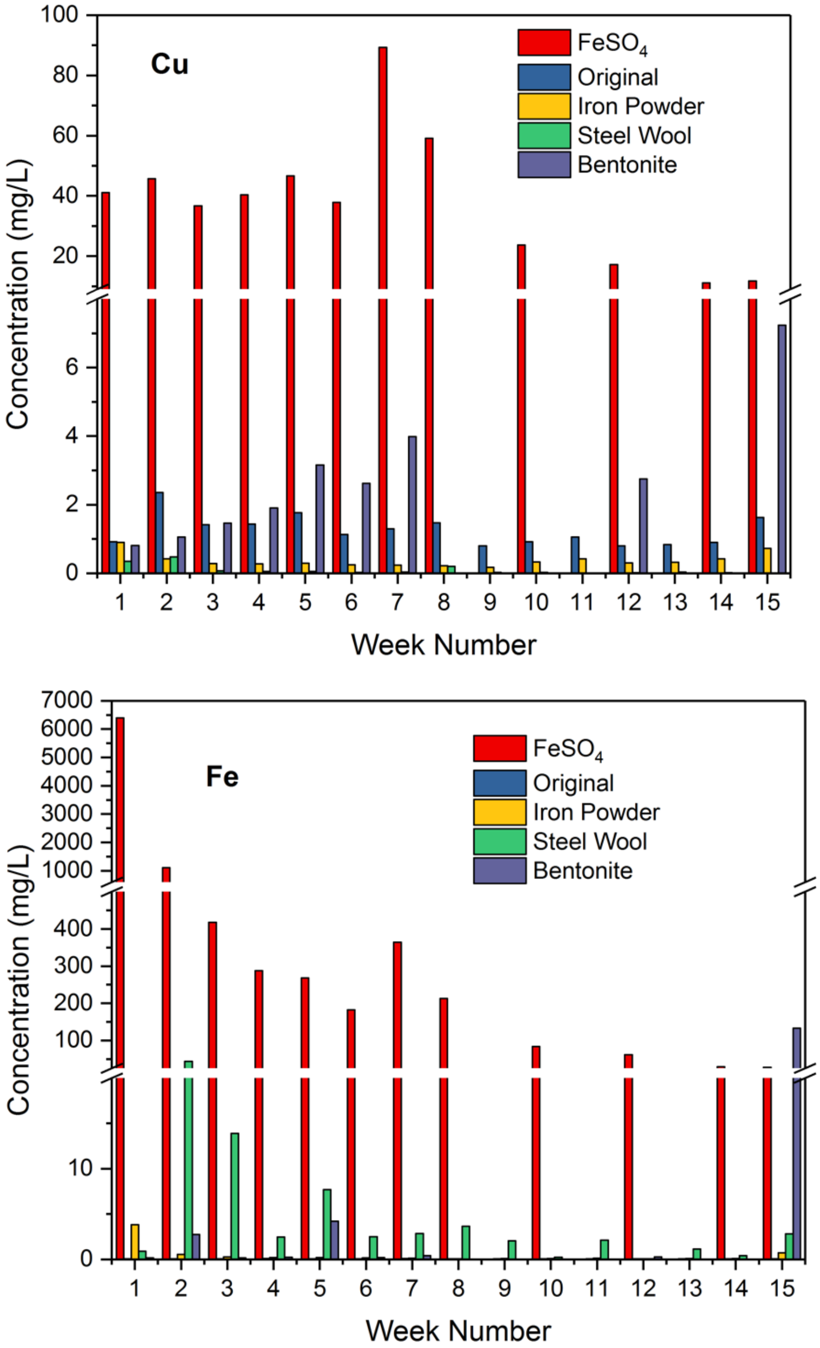


Figure 6. The leaching profiles of CCA metals in fine CCA mulch, mulch mixed with iron powder, steel wool, ferrous sulfate ($FeSO_4$), or bentonite as a fixing agent.

Figure 6 also shows the amount of Fe leached from the column, and according to speciation tests carried out at weeks 3, 8, and 15, ferrous ions were the dominant species leached when iron powder and steel wool were used. Notably, none of the As, Cr, or Cu concentrations decreased when bentonite was added to the CCA fine mulch; instead, the leachate concentrations steadily increased. This was probably due to bentonite adsorbing water, creating moisture in the CCA mulch and, as a result, dissolving more CCA metals in the leachate. These results clearly demonstrate that both steel wool and iron powder could significantly reduce the leaching rate of CCA heavy metals. Ferrous sulfate increased chromium and copper release, likely due to enhanced dissolution and mobilisation under acidic and oxidising conditions. Bentonite addition did not reduce leaching; instead, it promoted metal release, potentially due to moisture retention within the mulch matrix. These results suggested that reductive iron-based amendments are more effective than adsorption-based clays or soluble iron salts. The superior performance of steel wool, in particular, reflects its sustained reactivity and capacity to promote metal sequestration under dynamic leaching conditions.

3.3.3. Effect of Remediation Agent on the Speciation of Toxic Metals

The CCA preservative originally consisted of 34.0% As_2O_5, 47.5% CrO_3, and 18.5% CuO. The As was present as As(V), and the Cr existed as Cr(VI) in the CCA preservative. Speciation tests were conducted on leachate samples collected from columns L3, L7, and L9, which represent the fine mulch control (L3), fine mulch with iron powder (L7), and fine mulch with steel wool (L9), respectively. The speciation of leachate samples from columns L1 and L13 was not determined because the treatment using $FeSO_4$ salts (L1) and bentonite (L13) resulted in higher leaching concentrations during the initial testing. The speciation of As and Fe determined for weeks 3, 8, and 15 during leaching is shown in Figure 7. Arsenic exhibits the highest mobility due to its weaker fixation as an oxyanionic species, predominantly As(V), which readily desorbs and migrates under oxic, near-neutral conditions. Chromium, although originally present as reduced Cr(III) bound to lignocellulosic components, becomes progressively mobilised with aging and weathering, and wood degradation disrupts metal-wood complexes. Copper, although strongly complexed by organic functional groups, is gradually released through ligand exchange and dissolution processes. The introduction of iron-based immobilisation agents fundamentally alters these leaching pathways through coupled redox and sorption mechanisms. Chromium immobilisation is governed by reductive transformation and structural incorporation. Copper is immobilised through surface complexation, coprecipitation with iron oxides, and potential incorporation into secondary mineral phases. The superior performance of steel wool compared with iron powder reflects its slower corrosion rate and sustained generation of reactive iron surfaces, thereby maintaining immobilisation capacity throughout the column experiments. Acidification, increased ionic strength, and competitive dissolution promote the release of Cu, Cr, and Fe into solution, undermining stabilisation. Similarly, bentonite fails to immobilise CCA metals effectively, likely because of its limited affinity for oxyanionic arsenic species and enhanced moisture retention, which prolongs wood-water contact and facilitates leaching.

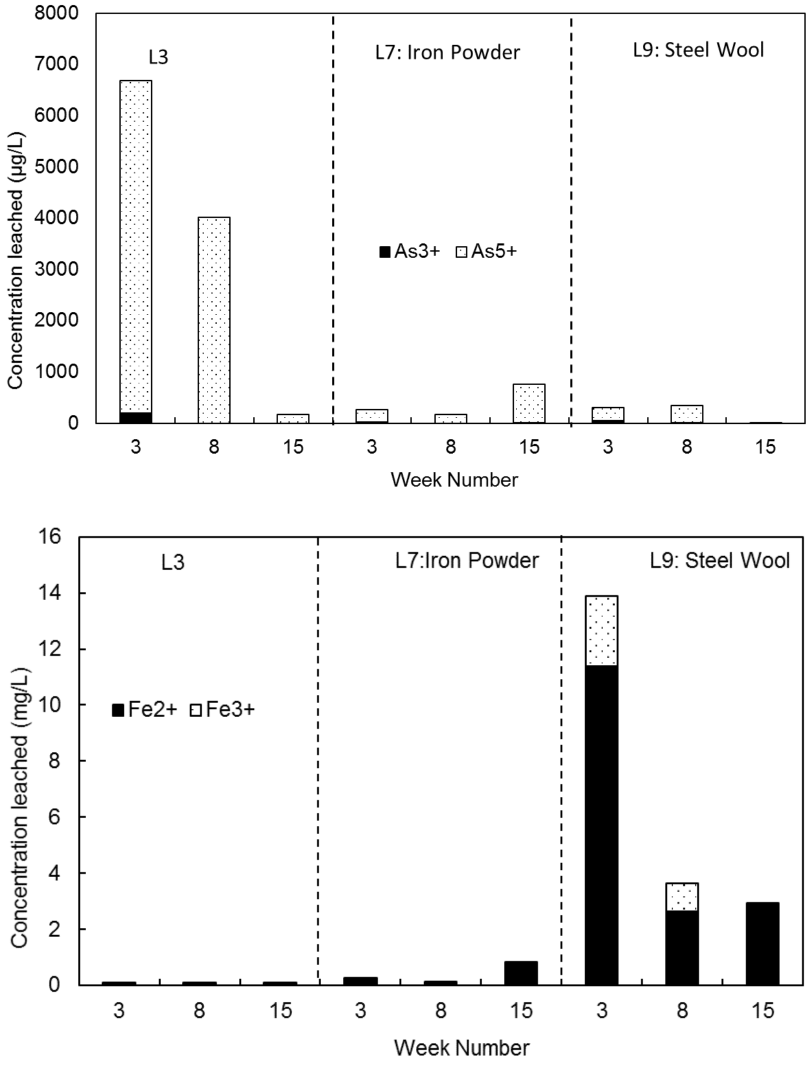


Figure 7. The speciation of As and Fe for columns L3, L7, and L9 during leaching.

A higher concentration of As was detected in the leachate from L3 than in that from the treated columns (L7 and L9), demonstrating the ability of the treatment agents to reduce the leaching of toxic metals. Similarly, all the leachate speciation analysis results revealed that As(V) dominated in the leachates. In week 3, As(III) was detected, and approximately 10% of the total As leached out as As(III) when iron powder or steel wool was used. However, As(III) decreased to a negligible level at weeks 8 and 15.

Cr was detected mainly as Cr(III) in L3, L7, and L9, as no Cr(VI) was detected in these samples. A negligible amount of Fe was detected in the control sample (L3), which was due to the limited amount of Fe in the CCA-treated timber. Fe(II) was the dominant species in the leachate of L7 (treated with iron powder), with the Fe content increasing from 0.07 to 0.74 mg/L from week 3 to week 15. Fe(II) was also detected mainly in L9 (treated with steel wool), with Fe(II) being the dominant species. The total amount of leached Fe decreased with increasing leaching time. L9 contained much more Fe than L7 did, indicating the easier leaching of iron from steel wool than from iron powder.

The results suggested that iron in iron powder or steel wool could react with toxic Cr(VI) and reduce it to less toxic Cr(III) when iron was oxidised. However, in the early stages of leaching, iron reduced As(V) to the more toxic form As(III), but this effect quickly diminished thereafter. Iron-based materials are widely used for the removal of As from water because they are nontoxic, inexpensive and highly abundant [48]. Zero-valent Fe materials enable the electrochemical reduction of metal ions to lower oxidation states and less soluble forms. It has been reported that highly soluble chromate can be rapidly reduced to Cr^{3+} by Fe(0), and precipitation and coprecipitation can occur subsequently [49]. Similarly, As(III) can form under anoxic conditions when it reacts with Fe(0). Adsorption of As(III) and As(V) to iron hydroxides could occur on the Fe(0) surface under anoxic conditions. Both As(III) and As(V) are adsorbed on iron oxides, forming an inner-sphere surface complex [50,51]. Moreover, the presence of Fe(II) could also increase sorption and promote the structural incorporation of As(V) into nonstoichiometric magnetite and maghemite structures [52]. The leached metals (loid)s may react with the iron materials mixed within the timber to form nontoxic forms, which are sorbed by surface hydroxides and precipitated/coprecipitated within the structure.

3.3.4. Effect of Remediation Agents on Microbial Diversity

A significant reduction in the leaching of toxic metals was achieved by adding iron powder to column L7 and steel wool to column L9. Samples of the timber mulch from these columns were collected for analysis of microbial diversity using 16S rRNA sequencing on the Illumina HiSeq platform (AGRF, Adelaide). Microbial biodiversity was assessed to evaluate the level of degradation of the CCA timber following the fixation treatment.

A summary of the bacterial and fungal taxonomy profiles at the phylum level is shown in Figure 8 and Table S3. The phylum-level taxonomy summary for bacteria (16SV3V4) and fungi (ITS) in chromated copper arsenate (CCA)-treated timber under various immobilisation agents (mulch, iron powder, and steel wool) revealed distinct microbial community structures. The bacterial community was predominantly composed of Proteobacteria, the most abundant phylum across all treatments, followed by Firmicutes and Bacteroidetes, which had relatively low abundances. Other phyla, such as Acidobacteria, Armatimonadetes, and Chloroflexi, are present in relatively small proportions, contributing to overall microbial diversity. In contrast, the fungal community was overwhelmingly dominated by Ascomycota, with significant contributions from Basidiomycota, particularly under the steel wool treatment. Other fungal phyla, including Chytridiomycota and Zygomycota, are minimally represented. These findings underscore the influence of different immobilisation agents on microbial dynamics, which may impact the leaching behaviour of copper, arsenic, and chromium from CCA-treated timber. The bacterial diversity changed after treatment with iron powder and steel wool. Interestingly, a major change was observed in Proteobacteria, which decreased from 72% in the control sample to 67.2% and 53.8% for L7 (iron powder) and L9 (steel wool), respectively. In contrast, increases in actinobacteria (such as Firmicutes and Verrucomicrobia), cyanobacteria, and FBP (fructose-1,6-bisphosphate) were observed. The reduced dominance of Proteobacteria indicates that this group is sensitive to the treatments. The principal constituent of the fungus in the samples was Ascomycota, at 98.5%, 99.3%, and 97.0% for L3, L7, and L9, respectively. A decrease in the levels of some minor constituents, such as the “other” group, was observed in L7. Basidiomycota decreased in L7, whereas it increased in L9 compared with L3. A new component, chytridiomycota and zygomygota, was present in L9 but was absent in L3 and L7.

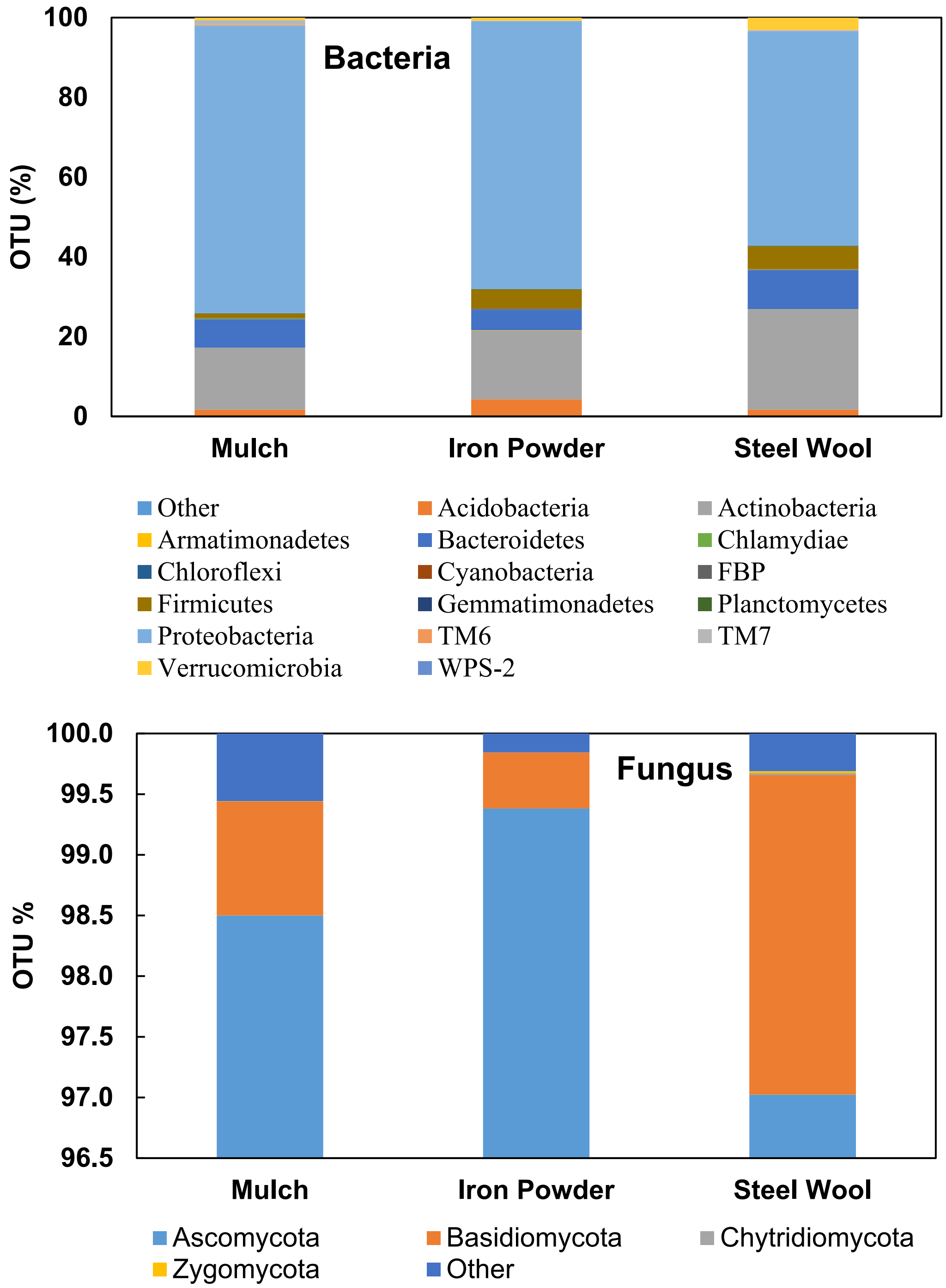


Figure 8. The taxonomy summary (Phylum) for bacteria (16SV3V4) and fungi (ITS).

The Shannon index, an indicator of biodiversity, was calculated via the following equation:

$$Shannon\ index = -\sum P_i \times Ln\ (P_i) \qquad (1)$$

where P_i is the proportion of species i, and the Shannon index values at the genus level for bacteria calculated for mulch (L3), mulch with iron powder (L7), and steel wool (L9) are 2.94, 3.31, and 3.63, respectively, demonstrating that a more diverse system was obtained after remediation using iron powder or steel wool. Similarly, the richness numbers (number of species) at the genus level were 194, 219, and 231 for mulch, mulch with iron powder, and steel wool, respectively.

The fungal richness (genus level) was 56, 36, and 64 for mulch (L3), mulch with iron powder (L7), and steel wool (L9), respectively. Fungal richness was reduced in mulch with iron powder, whereas it increased following steel wool treatment. The Shannon index values for fungi from mulch, mulch with iron powder, and steel wool were 1.88, 1.35, and 1.97, respectively. A detailed examination of the differences in biological diversity and major differences at the genus level are shown in Figure S8. As it is designed to protect wood from microbial decay (Cu) and insect infestation (As), changes in the microbial community may be associated with treatment effects in CCA-treated timber. The higher Shannon diversity indices and richness values in the columns amended with iron powder and steel wool suggest that the reduction in bioavailable As, Cr, and Cu alleviated the metal-induced stress on the microbial communities. In particular, steel wool treatment created a more favourable microenvironment for microbial colonisation, likely because of sustained metal immobilisation and the formation of iron (oxyhydr)oxide phases that reduce toxicity. The partial recovery and diversification of microbial taxa, including the emergence of previously suppressed fungal groups, indicate a shift towards a more functionally resilient community. These findings demonstrate that effective immobilisation of CCA-derived metal(loid)s not only limits leaching but also supports microbial ecosystem recovery, which is a critical consideration for the safe reuse or long-term management of CCA-treated timber mulch.

3.4. Mechanism of CCA-Timber Remediation

Samples from column L9 (steel wool) were obtained for SEM and XRD analysis, as they reduced the leaching of As and Cr. The SEM images are shown in Figure 9, and the XRD patterns are shown in Figure S9. The surface morphology of steel wool is quite different before and after the leaching test, where the reduction process occurs. A relatively smooth, uniform surface was observed on the steel wool, whereas different mineral morphologies were observed after the leaching tests. At least four distinct morphological shapes of reduced steel wool were observed, confirming the presence of crystalline and noncrystalline moieties (Figure 9). The elemental mapping indicated that the sample surface was composed mainly of Fe and O (normalised atom ratio of approximately 2:3) with the presence of other elements, including Si, Na, S, Mn, and As (Table S4). The results indicated the formation of iron oxide minerals on the surface of steel wool during the leaching study, whereas As incorporation or coprecipitation occurred in some areas.

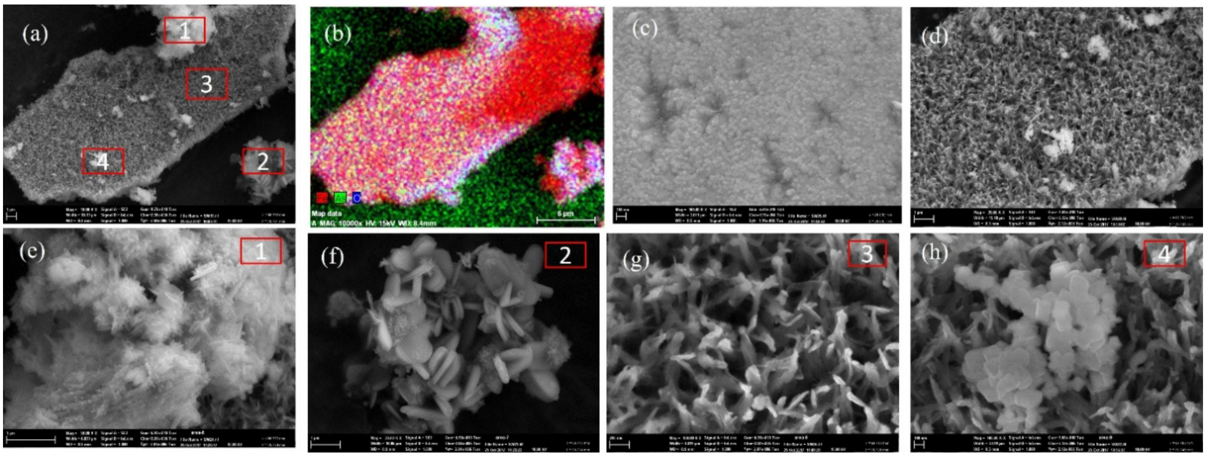


Figure 9. SEM image of steel wool before and after column study (**a**) and EDX mapping (**b**) of steel wool after the column study; morphology comparison of steel wool before (**c**) and after (**d**) the column study; and detailed morphology of steel wool after the column (**e**–**h**).

Similarly, Figure S9 shows the presence of mineral phases (magnetite, goethite, arsenolite, and chromium iron arsenide) on the steel wool surface after column leaching. The XRD pattern of the steel wool showed that it transformed from a crystalline to a noncrystalline phase during the reduction process due to metal complexation. The reduced iron enhances the reactivity of the heavy-metal complexation process, helping bind heavy metals and potentially immobilising them. This phenomenon suggested the formation of complex mineral phases involving Fe, As, and Cr, thereby reducing the leachability of the metal ions.

The As, Cr, and Cu leached from timber could interact with the iron on the surface of the iron powder or steel wool. Iron materials are widely used for the reduction remediation of various contaminants, including As, Cr, VOCs, halogenated organic compounds, and nitrate [53,54]. It could act as a reductant for Cr(VI) and subsequently precipitate in situ as Cr(III) oxyhydroxide or chromium-iron hydroxide [55,56]. The processes involved in As removal are rather complex, but they generally involve adsorption, reduction, surface precipitation, and coprecipitation with various iron corrosion products (e.g., ferrous/ferric (hydro)oxides) [57]. As(V) ions in solution can be adsorbed onto the surface of Fe-based materials to facilitate the reduction reaction, in which As(V) is converted to As(III). The As(III) species can undergo further interactions with the Fe^0 surface or Fe(II) ions, leading to the precipitation of iron-arsenic compounds, which are typically less soluble and mobile than As(V).

The leaching of Cr and As was reduced after treatment with iron powder and steel wool, with only a small percentage of As(III) detected, suggesting a reduction reaction and the removal of soluble ions from the leachate by Fe-based materials (the formation of As-containing minerals). Figure 10 illustrates the processes that could be involved in remediation, e.g., corrosion of the Fe surface and reduction and adsorption interactions.

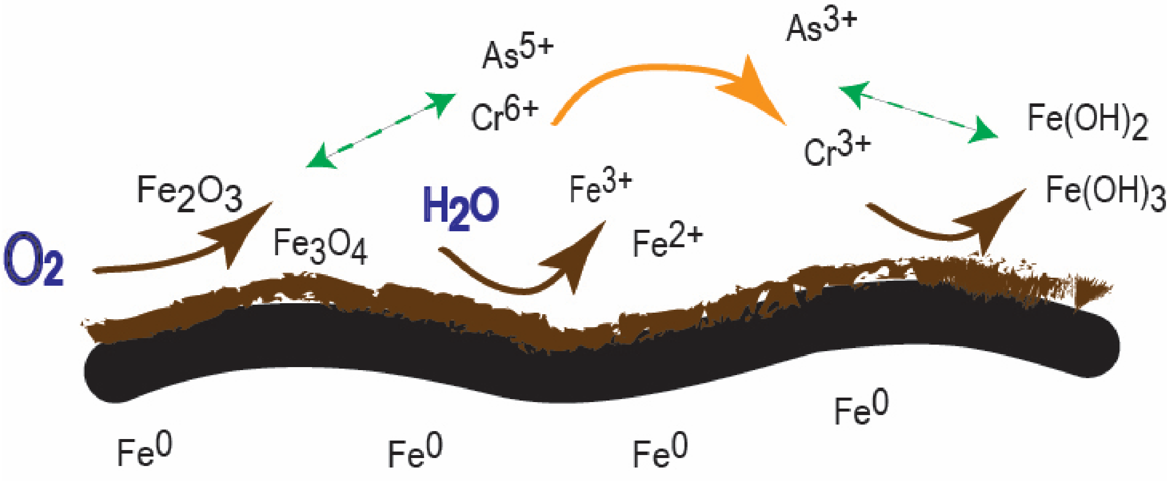


Figure 10. Diagram illustrating the interaction between As/Cr and Fe surfaces; brown arrow—transformation of Fe^0; orange arrow—reduction reaction for As and Cr; green arrow—adsorption, complexation, and coprecipitation of As/Cr with Fe oxides/hydroxides formed on the surface.

Beyond chemical stabilisation, reduced metal bioavailability in iron-amended systems alleviates microbial toxicity, enabling recovery of bacterial and fungal communities. This biological response further supports immobilisation by promoting stable microenvironments and limiting secondary mobilisation pathways. Collectively, these mechanisms demonstrate that zero-valent iron amendments achieve robust, multiprocess immobilisation of CCA metals, integrating redox control, sorption, and ecological stabilisation to substantially reduce environmental risk. The observed increase in microbial diversity following iron-based remediation has direct implications for reducing ecological risk and for the potential reuse of CCA-treated timber. Increased bacterial and fungal richness, particularly in the steel wool-amended system, indicates reduced metal(loid)s bioavailability and toxicity, creating conditions more conducive to microbial colonisation and ecological stability. Such microbial recovery is widely recognised as an indicator of reduced environmental stress and improved material safety. Consequently, the restored and diversified microbial communities suggest that immobilised CCA-treated timber mulch poses a lower ecological risk during reuse scenarios such as landscaping or interim storage. These findings highlight that iron-based immobilisation not only suppresses metal leaching but also supports biological recovery, strengthening its suitability for risk-based management and reuse strategies.

4. Conclusions

Column leaching tests for CCA-treated timber were performed to assess the effect of the immobilisation treatment. Different mulch sizes did not result in significant changes in the leachability of metal(loid)s. Higher mean concentrations of toxic metals (As, Cr, and Cu) were leached from the block timber dripping columns than from the fine and coarse mulch columns. The block-submerged sample leached the most toxic metals. Remediation via ferrous sulfate, iron powder, steel wool, or bentonite had different effects on the leaching of toxic metals. The utilisation of iron powder and steel wool significantly reduced the leaching of toxic metals. The speciation of As and Fe changed during remediation when iron powder and steel wool were used. The use of steel wool increased the biodiversity of bacteria and fungi in the timber mulch, indicating reduced toxicity associated with reduced leaching of toxic metal(oid)s. The use of iron powder increased bacterial biodiversity but decreased fungal biodiversity. The formation of As/Cr and Fe complex minerals helped reduce the leaching of As and Cr from the CCA timber mulch remediated with steel wool. The coupled reduction in metal mobility and improvement in microbial diversity highlight that physicochemical stabilisation can translate into meaningful reductions in biological risk. These findings provide a strong scientific basis for risk-based management strategies, supporting the safer reuse or controlled handling of CCA-treated timber mulch under field-relevant conditions.

From an environmental and practical perspective, the findings of this study provide a basis for estimating remediation requirements for CCA-contaminated timber waste under field conditions. The column experiments demonstrate that relatively low mass loadings of iron materials, particularly steel wool, can achieve near-complete immobilisation of arsenic and substantial reductions in the leaching of chromium and copper over extended rainfall simulations. The extrapolation of the observed iron consumption and sustained immobilisation capacity suggests that remediation can be achieved with modest amendment volumes, thereby significantly reducing metal mobility. Importantly, steel wool offers volumetric efficiency owing to its high reactivity and extended corrosion lifespan, reducing the total amendment volume required for large-scale applications. For stockpiled mulch or treatment cells, iron-based amendments can be blended directly into the material or deployed as permeable reactive layers, minimising excavation and secondary waste generation. While site-specific factors such as rainfall intensity, timber particle size, and contaminant loading influence amendment demand, the results indicate that iron-mediated immobilisation represents a scalable, cost-effective strategy for mitigating CCA-derived metal risks.

Supplementary Materials

The additional data and information can be downloaded at: https://media.sciltp.com/articles/others/2601191621170891/EPRRI-25100078-Supplementary-Materials.pdf. Table S1: Set up information for the column study. Table S2: Concentrations of metal(loid)s in the CCA timber. Table S3: Taxonomy summary (phylum) for bacteria and fungus. Table S4: The normalised atom ratio for steel wool after the column study via SEM-EDX analysis. Figure S1: CCA-treated timber waste log and shredded mulch. Figure S2: A slice of carbon-coated CCA-treated timber ready for SEM (a) and a back scatter image for SEM (b). Figure S3: The setup of the column leaching study for heavy metal ions. Figure S4: EDS spectra of the iron powder and steel wool samples. Figure S5: Boxplots of toxic metals leached by block timber, coarse mulch and fine mulch. Figure S6: Percentage of metals leached from timber blocks with dripping. Figure S7: Leaching concentrations of As and Cr from original and treated timber mulch. Figure S8: Major genera for (a) bacteria (16SV3V4) and (b) fungi (ITS) in mulch, mulch with iron powder, and steel wool samples. Figure S9: XRD patterns of steel wool before and after the column study.

Author Contributions

J.D.: conceptualization, investigation, data analysis, writing—original drafting; Y.L.: conceptualization, investigation, data analysis, writing—original drafting, reviewing and editing; M.H.: data analysis, visualization, writing—reviewing and editing; M.M.R.: data analysis, validation, writing—reviewing and editing; R.N.: conceptualization, validation, writing—reviewing and editing, supervision. All authors have read and agreed to the published version of the manuscript.

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Institutional Review Board Statement

Not applicable.

Informed Consent Statement

Not applicable.

Data Availability Statement

The research results are presented in the article. The original research data can be accessed upon request from Yanju Liu (Yanju.liu@newcastle.edu.au), Jason Du (jason.du@slrconsulting.com), Ravi Naidu (ravi.naidu@crccare.com).

Conflicts of Interest

The authors declare no conflict of interest. Given the role as Editor-in-Chief, Ravi Naidu, and Editorial board members, Yanju Liu, had no involvement in the peer review of this paper and had no access to information regarding its Peer-Review process. Full responsibility for the editorial process of this paper was delegated to another editor of the journal.

Use of AI and AI-Assisted Technologies

No AI tools were utilized for this paper.

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