Great Britain
Journals Press

RESEARCH FINGERPRINT

IDENTIFIER
LJRS-226330

PEER REVIEW
Double Blind

SIMILARITY CHECK
Perplexity AI and iThenticate

ACCESS
Open Access

LANGUAGE
English

PRINT ISSN
2631-8490

ONLINE ISSN
2631-8504

EDITION

ABBREVIATION
LJRS

VOLUME
26

ISSUE
5

YEAR
2026

KEY DATES

RECEIVED
2026-03-13

ACCEPTED
2026-03-18

CATALOGING

CROSSMARK DOI
10.34257/LJRS226330UK

LCC CLASS
TD878, TD885, GE230

London Journal of Research In Science: Natural and Formal
General Section

Article Record

Hybrid Approach for Arsenic Remediation: Soil Washing Coupled with Hematite Nanoparticles in Cu-As-Au Mining Waste

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ABSTRACT

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Full abstract continues on the metadata continuation sheet.

Index Terms: Arsenic removal • hematite nanoparticles • mining waste management • valuable metal extraction

FUNDING
No external funding was declared for this work.

CONFLICTS
The authors declare no conflict of interest.

AI USAGE
No generative AI was used for analysis or results.

HOW TO CITE

Jameshourani et al. (2026). Hybrid Approach for Arsenic Remediation: Soil Washing Coupled with Hematite Nanoparticles in Cu-As-Au Mining Waste. London Journal of Research In Science: Natural and Formal, 26(5), 21-25. DOI: 10.34257/LJRS226330UK

ACCESS ONLINE

METADATA CONTINUATION

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ARCHIVAL RECORD

LJRS · Vol 26 · Issue 5 · 2026

Article ID LJRS-226330 · DOI 10.34257/LJRS226330UK

Print ISSN 2631-8490 · Online ISSN 2631-8504

CROSSREF DOI 10.34257/LJRS226330UK

RESEARCH ARTICLE

Hybrid Approach for Arsenic Remediation: Soil Washing Coupled with Hematite Nanoparticles in Cu-As-Au Mining Waste

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Abstract

Copper–gold mining in the eastern Alps, Austria, has produced sulfide-rich tailings that continue to release arsenic (As) into the environment. This study examined the effectiveness of removing arsenic from mining waste using various chemical extractants, nanoparticle (NP) dosages, and different sample grain sizes. The goals are to extract arsenic and produce a concentrate for recovering valuable metals, such as copper and gold, through metallurgical processes. This paper presents an integrated experimental framework designed to simulate realistic remediation scenarios using hematite nanoparticles (HMNPs). Multiple extractants—ultrapure water (UPW), nitric acid (HNO_3), monosodium dihydrogen phosphate (NaH_2PO_4), and hydrogen peroxide (H_2O_2)—were used to explore arsenic mobilization under different chemical stress conditions. Among the tested extractants, 0.1 M HNO_3 showed the highest arsenic removal efficiency, significantly outperforming UPW, 0.1 M NaH_2PO_4, and 3% H_2O_2. The addition of NPs improved overall arsenic removal; however, the efficiency did not increase proportionally with higher NP concentrations. A concentration of 0.05 HNPs g/L resulted in slightly higher arsenic removal rates compared to 2.5 HNPs g/L and 5 HNPs g/L, likely due to particle aggregation at higher concentrations, which reduced the available reactive surface area. The texture of the mining waste also influenced removal efficiency. Finer particles promoted greater arsenic release under the H_2O_2 + HNP treatment, whereas coarser particles showed better responses to UPW and NaH_2PO_4. In contrast, HNO_3 consistently delivered high removal efficiencies across all particle sizes by directly dissolving the mineral compositions. The distinct oxyanion behavior of As underscores the necessity for strong extractants or NP-assisted approaches, as conventional methods optimized for cationic metals are less effective. Overall, HNO_3 proved to be the most effective single extractant, achieving removal efficiencies of up to 87.6% for As, 63.8% for Zn, 90.7% for Pb, and 85.97% for Cu. Post-treatment filtrates were analyzed using Inductively Coupled Plasma Mass Spectrometry (ICP-MS) and Instrumental Neutron Activation Analysis (INAA) to quantify As removal.

Keywords: *Arsenic removal, hematite nanoparticles, mining waste management, valuable metal extraction*

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1 Introduction

The reprocessing of polymetallic low-grade ores previously considered economically unfeasible has gained renewed significance due to advances in extraction technologies and the increasing global demand for critical metals, such as copper, zinc, silver, and gold. Historically discarded materials, including tailings and mining residues, represent not only potential secondary metal resources but also persistent environmental hazards; due to the presence of sulfide minerals and associated potentially hazardous elements (PHEs), particularly arsenic [1]. These environmental risks are exacerbated by natural weathering processes that induce sulfide oxidation, resulting in acid mine drainage (AMD) and the subsequent mobilization of toxic elements into surrounding ecosystems [2].

This study examines the effectiveness of HMNPs (α-Fe_2O_3) combined with various chemical extractants to reduce arsenic bioavailability in legacy mining waste from the eastern Alps, Austria, an area historically affected by orogenic copper-gold mining activities. The main objectives are twofold: (1) to evaluate HMNPs' ability to adsorb and remove arsenic under different extraction conditions, and (2) to understand how soil particle size, mineralogical composition, and solution chemistry influence arsenic mobility and remediation success.

The significance of this research lies in addressing the dual challenge of environmental protection and sustainable resource recovery. Conventional mining waste remediation techniques often prove inadequate when dealing with the heterogeneity and chemical complexity of mine-contaminated substrates, emphasizing the need for adaptable and targeted approaches [3, 4]. Nanotechnology offers a promising alternative in this regard, with iron oxide-based nanomaterials demonstrating exceptional surface reactivity, tunable physicochemical properties, and relatively low toxicity, making them highly suitable for environmental remediation applications [5, 6].

Previous studies have explored a range of nanomaterials for arsenic remediation, including zero-valent iron, goethite, magnetite, and various carbon-based composites, highlighting their efficiency under specific environmental conditions [7, 8, 9]. Earlier works have demonstrated the potential of HMNPs to interact with a range of PHEs in complex mining waste matrix; however, their role in real-world remediation scenarios, particularly under field-representative extractant conditions, remains underexplored.

2 Role of Nanotechnology in Arsenic Remediation

Traditional methods for arsenic removal—such as physicochemical adsorption, redox reactions (both photochemical and chemical), and filtration—have shown limitations in efficiency and flexibility. The advent of nanotechnology has significantly advanced the remediation of arsenic from mining waste by enhancing removal methods and improving detection accuracy.

2.1 Nanoscale iron oxides

Iron oxide nanoparticles (IONPs) and iron nanoparticles (FeNPs) have been extensively studied for their high efficiency in removing pollutants, particularly arsenic, from contaminated soils and water. IONPs are especially effective due to their ability to bind arsenic through chemisorption, forming strong and stable chemical bonds with arsenic species. In contrast, carbon-based NPs typically rely on physisorption, which involves weaker, non-specific interactions; however, their adsorption performance can be significantly enhanced through surface modification.

Nanoscale iron oxides (nFeOs) demonstrate exceptional efficiency not only in removing arsenic but also in eliminating other toxic metals such as V, Cr, Co, Mn, Se, Mo, Cd, Pb, Sb, Tl, Th, and U. Their low toxicity, high reactivity, and resistance to desorption make them a promising material for environmental remediation. Among the various forms of iron oxides, goethite $(- \mathrm{FeOOH})$, hematite $(- \mathrm{Fe_2O_3})$, maghemite $(\gamma - \mathrm{Fe_2O_3})$, and magnetite $(\mathrm{Fe_3O_4})$ are recognized as the most efficient for such applications. The adsorption mechanism—whether chemisorption or physisorption—ultimately depends on the NP's composition and surface chemistry, which govern their interaction strength and selectivity toward contaminants.

Studies confirm that nFeOs efficiently remove arsenite $\mathrm{AsO_3^{3-}}$ and arsenate $\mathrm{AsO_4^{3-}}$ while minimizing secondary contamination risks. They also immobilize toxic metals in mining waste. Factors influencing the stability and transport of nFeOs include particle size, concentration, magnetism, solution chemistry, and environmental conditions [9].

NPs in concentrated solutions tend to aggregate due to increased collision rates, reducing stability compared to dilute solutions. For instance, smaller HMNPs exhibit higher aggregation rates due to surface property changes at reduced sizes [8]. Additionally, strong magnetic properties in IONPs contribute to increased aggregation. However, studies suggest that IONPs generally have low or negligible toxicity to living organisms [5].

2.2 Hematite nanoparticles

Hematite α-$\mathrm{Fe_2O_3}$ is a stable, well-crystalline NP commonly found in mining waste and plays a key role in the iron biogeochemical cycle. Its high surface area, thermal stability, and abundant hydroxyl sites make it highly effective for adsorbing metalloids and trace metals [10]. Due to these properties, hematite is widely used in contaminant remediation, catalysis, gas sensing, and battery applications [11]. Studies have shown its ability to adsorb contaminants like copper, nickel, cobalt, cadmium, lead, zinc, and anions such as uranyl, phosphate, sulfate, and selenate. Common synthesis methods include precipitation, hydrothermal, and solvothermal techniques, which are valued for their simplicity and high yield. Preventing precursor coagulation is crucial for producing monodispersed HMNPs [12].

2.3 Factors influencing NP performance

The effectiveness of nanomaterials in arsenic remediation is governed by various environmental and operational factors. Among these, the pH and redox potential of mining waste are particularly important, as they significantly influence the chemical speciation and mobility of arsenic in mining waste. The presence of organic matter and the mineralogical composition of the mining waste also significantly affect how NPs interact within the mining waste matrix. Additionally, the specific surface area of IONPs is a key factor in optimizing their adsorption efficiency, as it determines the number of active sites available for binding arsenic. Particle size is vital to adsorption efficiency because it affects surface area, crystallinity, and site density. Hematite particles range from nanoscale to microns, with smaller sizes generally providing better adsorption due to increased surface reactivity [12].

2.4 Advantages of nanotechnology

Nanomaterials offer several advantages for arsenic remediation. Nanomaterials possess a high surface area that significantly increases their adsorption or catalytic activity. This structural feature also contributes to improved detection capabilities and greater selectivity, allowing for lower detection limits and increased accuracy in identifying arsenic presence.

In the context of mining waste reclamation, various nanomaterials have proven effective. These include zeolites, IONPs, phosphate-based NPs, and carbon nanotubes. Among them, zero-valent iron (nZVI) and IONPs have been the most extensively studied due to their strong chemical affinity for arsenic and their effectiveness across a wide range of mining waste types [5, 6]. IONPs tend to aggregate based on concentration and pH. Brownian motion increases collisions, leading to aggregation, especially at concentrations above 50 mg/L [13].

3 Chemical and mineralogical composition of the mine waste

The tailings waste contains primary minerals like chalcopyrite ($\mathrm{CuFeS_2}$), bornite ($\mathrm{Cu_5FeS_4}$), and covellite (CuS), which were part of the original ore. Secondary minerals formed through weathering and oxidation include malachite ($\mathrm{Cu_2CO_3(OH)_2}$), azurite ($\mathrm{Cu_3(CO_3)_2(OH)_2}$), cuprite ($\mathrm{Cu_2O}$), and brochantite ($\mathrm{Cu_4SO_4(OH)_6}$). These minerals show the transformation of sulfides into oxides, carbonates, and sulfates over time. Various techniques have been developed to treat mining waste, primarily focusing on containing or immobilizing pollutants or on cleaning up and removing them. Each case is different, so methods need to be tailored or combined. However, many remediation options face significant challenges [3, 14].

The mineral composition of historical tailing waste significantly affects arsenic (As) removal efficiency. Iron oxides (e.g., hematite, goethite, magnetite) are the primary arsenic adsorbents, while quartz is inert and does not contribute to removal. Clay minerals moderately aid adsorption, carbonates help stabilize arsenic through precipitation, and sulfides can release arsenic, requiring pre-treatment.

Magnesium minerals enhance stability but may compete with iron for binding, while phosphates reduce arsenic removal by competing for adsorption sites. Silicates (e.g., feldspar, mica) have minimal impact. Understanding these minerals is crucial for effective arsenic remediation

Table 1. Selected components and particle sizes of the mining waste

Sample Name	Particle size (mm)	As (ppm)	Au (ppb)	Ag (ppm)	Cu (ppm)	Mn (ppm)	Mg (%)
WB[a]_12	0.18–2	570	81	0.14	252	895	0.77
WB_12	< 0.18	3180	792	0.72	983	1210	1.45
WB[b]_13	0.18–2	3450	415	0.26	440	1700	0.55
WB_13	< 0.18	5920	858	1.51	1780	1730	0.91

[a]Weißenbach12 (WB) 1.5 m depth from surface; [b]Weißenbach13 (WB) 2.5 m depth from surface

strategies [15, 16, 17]. The chemical composition of the samples before treatment is presented in Table 1, which also indicates the presence of precious and base metals, as well as some hazardous elements such as arsenic.

4 Material and Methods

4.1 Sample collection and preparation

Samples containing sulfide-rich tailings were obtained from one mine waste dump at two different depths (WeiBenbach12: 1.5 m and WeiBenbach13: 2.5 m). The samples were air-dried and sieved to < 0.18 mm and $0.18 - 2$ mm for general characterization and arsenic removal.

4.2 Reagents and pH conditions for removal of arsenic

A 0.1 M phosphate solution prepared from salts such as NaH_2PO_4 was used to facilitate the displacement of arsenate ions from grain size particles, operating effectively at a pH of 6.1. Additionally, 3% hydrogen peroxide (H_2O_2) was employed to oxidize sulfide minerals, promoting the release of arsenic and associated metals into the aqueous phase at a pH of approximately 6.5. UPW served as a blank solution and was also utilized to assess the influence of HMNPs under near-neutral conditions (pH 6–7). Furthermore, concentrated nitric acid (0.1 M HNO_3), a strong oxidizing agent, was applied under highly acidic conditions (pH 1) to ensure the complete oxidation and dissolution of mineral constituents.

4.3 Hematite nanoparticles

HMNPs (α-Fe_2O_3; $20 - 50$ nm; 99.8% purity) were utilized to evaluate their impact on the removal of potentially harmful elements (PHEs) in mining waste. Stock suspensions of HMNPs were prepared at concentrations of 5.0 g/L, 2.5 g/L, and 0.05 g/L using various extractants: UPW, 0.1 M nitric acid (HNO_3), 0.1 M monosodium dihydrogen phosphate (NaH_2PO_4), and 3% hydrogen peroxide (H_2O_2). To prevent NP aggregation, 5 mM sodium citrate was added as a stabilizing agent. The suspensions were stirred for 30 minutes and subsequently sonicated for 15 minutes before soil application.

Samples were introduced into the NP suspensions, and the mixtures were agitated on a rotary shaker at 13 rpm for periods of 5 days and 10 days in the dark. The pH of the solutions was adjusted to between 6.0 and 7.2 using 0.1 M HCl or NaOH except for the HNO_3-based suspensions, which maintained an acidic pH of ~ 1. For the H_2O_2 treatment, 10 g of sample was mixed with 100 ml of 3% hydrogen peroxide and stirred at 77°C and 240 rpm for 2 hours to promote oxidation of sulfide minerals. Subsequently, 0.5 g of HMNPs was added, and the mixture was stirred at 40°C for 24 hours, with pH adjusted to 6.5. Parallel control experiments were conducted under identical conditions but without the addition of NPs.

4.4 Mining waste treatment and metal extraction

For each treatment, 5 g or 10 g of sample was combined with 50 mL or 100 mL of NP suspension and control solution in polypropylene containers (ratio is 1:10). The mixtures were homogenized by shaking for 5 days and 10 days under dark conditions. After treatment, samples were centrifuged at 2400 rpm for 10 minutes, and the supernatants were filtered through 0.45 μm membrane filters. The filtered extracts were then analyzed to determine the concentration of bioavailable PHEs and removal efficiency. The treated samples were dried at room temperature and stored for further analysis by INAA following 4-acid digestion and ICP-MS [6, 18].

5 Results and Conclusions

The study demonstrated that arsenic (As) removal efficiency is heavily dependent on the type of extract, NP dosage, and grain particle size. Among the tested extractants, HNO_3 delivered the highest As removal efficiency, performing significantly better than UPW, NaH_2PO_4, and H_2O_2. However, H_2O_2 had a much shorter reaction time (1 hour) compared to the other methods (5 and 10 days), but still produced promising results, especially with NPs, and could be further optimized for improved performance.

The addition of NPs generally enhanced As removal; however, increasing the mass fraction of NPs does not always result in a proportional increase in contaminant removal. Higher nominal NP loading (5 HNP g/L) can underperform because the extra mass no longer translates into available active surface due to aggregation, passivation, mass-transfer limits, or changes in solution chemistry—or because of experimental artifacts.

Particle size of samples also influenced performance: for $H_2O_2 + NPs$, finer particles (< 0.18 mm) improved As removal, because more reactive surface area and coatings were available for oxidation and release. For UPW and NaH_2PO_4, which are weak extractants, coarser particles ($0.18 - 2$ mm) were more effective because As was more weakly bound and could leach, whereas finer particles held As too strongly for pure water to remove. Strong extractants like HNO_3 dissolve the mineral composition and release As regardless of grain size. Finer particles may still give slightly higher efficiency (more surface area, faster dissolution), but the difference is less pronounced because HNO_3 is powerful enough to attack both coarse and fine fractions. Furthermore, deeper layers (up to 2.5 m depth) contained higher concentrations of hazardous elements as well as Au, Cu, and Ag.

Chemically, arsenic behaves differently from heavy metals due to its oxyanion nature, which makes it harder to remove with methods designed for cationic metals. Thus, if NPs are not used, a strong extract such as HNO_3 is necessary to achieve effective As removal (pH ~ 1). Importantly, the extractant that works best for As may not be the most efficient for other hazardous metals. However, the HNO_3 could remove 63.8% of the Zn, 87.6% of As, 85.97% of the Cu, and 90.7% of Pb. Comparison of the removal efficiency of As with the blank solution, with the HNO_3 solution, and the removal efficiency of different extractants on As removal is shown in Figures 1 and 2.

Table 2. Conditions and descriptions of the suspensions

Symbol	Condition and Description
S1	UPW, sample of 0.18–2 mm, 5 HNPs g/L
S2	UPW, sample of 0.18–2 mm without NPS
S3	NaH_2PO_4, sample of 0.18–2 mm, 5 HNPs g/L
S4	NaH_2PO_4 sample of 0.18–2 mm without NPS
S5	HNO_3 sample of 0.18–2 mm, 5 HNPs g/L
S6	HNO_3 sample of 0.18–2 mm without NPS
S7	UPW, sample of 0.18–2 mm, 0.05 HNPs g/L
S8	UPW, sample of 0.18–2 mm, 2.5 HNPs g/L
S9	UPW, sample of 0.18–2 mm, 5 HNPs g/L
S10	H_2O_2 with 5 HNPs g/L, sample of < 0.18 mm
S11	H_2O_2 with 5 HNPs g/L, sample of 0.18–2 mm

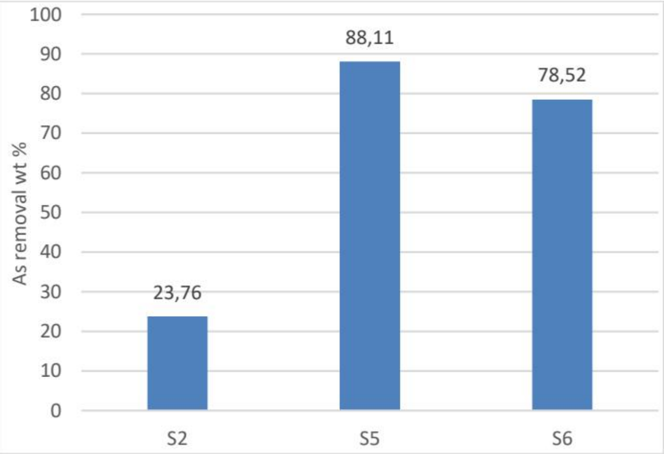

Figure 1. The As removal [wt.%] of the blank suspension vs. the HNO_3 with HNPs and without HNPs

The efficiency of combining chemical extraction with HMNPs for arsenic removal depends on the mineralogical and chemical properties of the mining waste. Nitric acid (HNO_3) proved to be the most effective extractant, achieving up to 87.6% arsenic removal, while also extracting copper (Cu), zinc (Zn), and lead (Pb) in parallel. In cases where the mineralogical and chemical characteristics of the mining waste are compatible with the NPs, these NPs can be partly reused through selective extraction methods, improving cost efficiency and sustainability.

After As removal, both the filtrate and the solid leach residue will be further treated to recover as many different valuables as possible. From the filtrate, copper could be separated from arsenic, for example, by means of cementation, while the solid concentrate or leach residue could be treated using different approaches depending on the amount of valuable elements present. Low-Cu material could be leached with cyanide to obtain Au, whereas Cu-rich material could be treated following pyro- or hydrometallurgical standard procedures, where Au will be enriched in a potential anode slime. Ultimately, the extraction of As is an important part of treating these historical mine wastes; however, As is only one of the many components in focus.

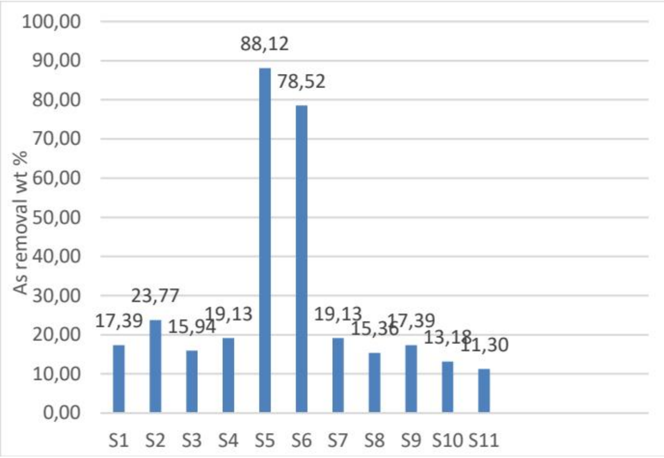

Figure 2. The As removal [wt.%] of different extractants under different conditions

ACKNOWLEDGEMENT

This work was supported by the European Union's Horizon Europe research and innovation programme under grant agreement No.101177746 (SCIMIN-CRM project).

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