Contents lists available at ScienceDirect

Science of the Total Environment

journal homepage: www.elsevier.com/locate/scitotenv

Characterization of medium and small-scale gold processing operations, wastewaters, and tailings in the Arequipa region of Peru

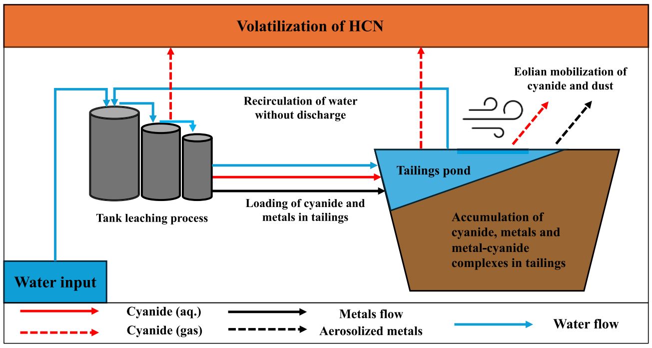
Vincent Hammer [a], Johan Vanneste [a], Francisco D. Alejo-Zapata [b], Julia Zea [b], Héctor G. Bolaños-Sosa [b], Carlos A. Zevallos Rojas [b], Linda A. Figueroa [a], Aaron Malone [c], Christopher Bellona [a], David C. Vuono [a,*]

[a] *Colorado School of Mines, Department of Civil and Environmental Engineering, Golden, CO 80401, USA*
[b] *Universidad Nacional de San Agustín de Arequipa, Centro de Minería Sostenible, Arequipa 04000, Peru*
[c] *Colorado School of Mines, Department of Mining Engineering, Golden, CO 80401, USA*

HIGHLIGHTS

- Gold cyanidation is a major industry in the Arequipa region but is largely uncharacterized.
- Water reuse without discharge leads to buildup of components in tailings and waters.
- Mass balance of cyanide revealed a cyanide deficit during process operation.
- Tailings were found to contain more cyanide and metals than previously thought.
- Exposure risk through volatilization/dust warrant further research or policy response.

GRAPHICAL ABSTRACT



ARTICLE INFO

Editor: Philiswa Nomngongo

Keywords:
Cyanide
Tailings
Gold mining
Arequipa-Peru
Cyanide complexes
Cyanidation

ABSTRACT

Gold cyanidation facilities in the Arequipa Region of Peru are challenged by the availability and quality of water for processing in an arid environment. The facilities reuse decant water which recycles residual cyanide but also undesirable constituents. To understand the impact of intensive water recycling on cyanide and metals concentrations, we collected barren water, decant water, and tailings samples from six gold cyanidation facilities with ore capacities of 10–430 tons per day. Processing facilities in Arequipa recycle all effluents, with decant waters making up 58 ± 11 % of process waters. Decant water contained non-target metals: copper (394 ± 161 mg/L), iron (59 ± 34 mg/L), and zinc (74 ± 42 mg/L). In addition, decant water mean free and complexed cyanide concentrations were 534 ± 129 mg/L and 805 ± 297 mg/L, respectively. Complexed cyanide concentrations remained more constant than free cyanide concentrations with 786 ± 299 mg/L for barren water and 805 ± 297 mg/L for decant water. Cyanide mass balances showed between 21 % and 42 % of unaccounted free cyanide from the start of gold cyanidation and discharge to the tailings storage facility (TSF). Free cyanide estimated losses due to volatilization were 0.8 kg and 2.5 kg of hydrogen cyanide per ton of ore processed at barren water pH of 10.1 and 9.7. Together these results indicate two acute hazards: 1) volatilization of free

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* Corresponding author.
E-mail address: dvuono@mines.edu (D.C. Vuono).

https://doi.org/10.1016/j.scitotenv.2024.174034
Received 2 April 2024; Received in revised form 13 June 2024; Accepted 14 June 2024

cyanide during processing and 2) loading and retention of cyanides and metals into TSFs. This study elucidates the extent of uncontrolled vapor phase cyanide release during gold processing operation and contaminant concentrations in the tailings storage facilities. The data highlights the need for improvement oversight, accountability, and regulation of gold processing facilities practicing intensive recycling and zero discharge.

1. Introduction

The Arequipa region has the third largest mining industry in Peru, producing 19.5 % of the country's annual gold in 2022 (Vera Gargurevich et al., 2023). Small-scale gold processing facilities (ore capacities up to 350 metric tons per day) play a large role in the industry, accounting for 40 % of the Arequipa region's gold production, with medium- (ore capacities of 350–5000 metric tons per day) and large-scale processing facilities (>5000 metric tons of ore capacity) responsible for the remaining 60 % (Dammert Lira and Molinelli Aristondo, 2007; MINEM, 2022). The process of gold extraction has changed substantially over the past several centuries evolving from placer, or alluvial-based, mining, to gold amalgamation with mercury, and starting in the 19th century, to the use of gold cyanidation (Verbrugge et al., 2021). Adoption of gold cyanidation has increased the efficiency of gold extraction, enabling the extraction of gold from low-grade ores, further expanding the gold mining industry (Verbrugge et al., 2021). Today, gold cyanidation is the preferred method of gold extraction in Peru (Vera Gargurevich et al., 2023) as efforts have been undertaken to reduce mercury pollution from artisanal and small-scale gold mining (Veiga et al., 2015). However, the growing gold mining industry in Arequipa raises concerns about the environmental impact of gold cyanidation (Roca Servat, 2012), because the processing facilities in the region face significant water challenges, resulting in conflicts on water rights, usage, and pollution (Triscritti, 2013; Bebbington and Williams, 2008). Contamination of scarce water sources through mining is a major point of concern for agriculture, fish and crayfish populations in gold processing locations, where the gold industry and communities coexist in proximity (Orellana Mendoza et al., 2021; Santi et al., 2021; Malone and Smith, 2021). Although previous literature has focused on the environmental impacts of mercury usage in artisanal and small-scale gold processing facilities, the potential impacts of the cyanidation process used at small-scale and medium scale gold cyanidation facilities have largely remained unstudied.

Gold cyanidation comes with its own challenges: cyanide is acutely toxic, volatile under low pH conditions, and complexes with heavy metals (Dzombak et al., 2006). Wildlife can be exposed to free cyanide through various means, including inhaling HCN, consuming contaminated water and animals (Eisler and Wiemeyer, 2004). Freshwater fish are especially sensitive to cyanide, experiencing adverse effects after continuous exposure to 5 μg/L of free cyanide and high mortality occurring at 20 μg/L of free cyanide (Eisler and Wiemeyer, 2004). Mammals and birds have similar oral LD_{50}, ranging from 1.4 mg CN^-/kg body weight to 14.8 mg CN^-/kg body weight depending on species (Eisler and Wiemeyer, 2004). Metal cyanide complexes, with the exception of mercury cyanide complexes, are considered less toxic, as the primary toxic agent, free cyanide, is only released through dissociation, however they still pose considerable hazards (Eisler and Wiemeyer, 2004; Alencar Meira da Silva and Davée Guimarães, 2024; Manar et al., 2011). These complexes are more persistent than free cyanide and pose long-term environmental risks as they can release free cyanide over longer periods of time as they naturally degrade (Eisler and Wiemeyer, 2004). In sites where mercury contaminated tailings are reprocessed with cyanide to recover additional gold, as is common in Arequipa's small and medium scale processing plants, formation of mercury cyanide complexes exacerbates environmental harm (Malone et al., 2023).

In gold cyanidation facilities, heavy metals are leached from the ore through the formation of metal cyanide complexes and retained in processing solutions and the tailings storage facility (TSF) (Karimi et al., 2010). The creation of these complexes increases the mobility of metals such as Fe, Cd, Zn, Ni, Cu, Co, and Hg and creates additional hazards in the event of TSF failure (Meeussen et al., 1995; Cánovas et al., 2023). This was exemplified in the 2000 Baia Mare cyanide spill, which catalyzed the founding of the International Cyanide Management Code (ICMC) (Akcil, 2010). While the aim of this code is to improve the best practice for the use of cyanide in gold mining, processing, and waste management, this is still a voluntary program that is not adopted by all processing facilities. Many small- and medium-scale gold processing facilities in Peru have little incentive to participate in the cyanide code. Furthermore, while there are strict regulations for cyanide discharge to surface and groundwater (MINAM, 2017), there are no cyanide limits for tailings. Considering many of Arequipa's processing facilities do not discharge water (e.g., all the decanted TSF pond water is recycled to reduce water consumption), this management practice impacts cyanide use and accumulation of undesirable constituents in process waters and tailings (Bahrami et al., 2007).

The main objective of this study was to characterize the process water and tailings at a range of facilities to identify cyanide and cyanide metal complex concentrations in process water and tailings under intensive recycling and zero discharge practices. For this study, six gold cyanidation facilities, from 10 metric tons/day to 430 metric tons/day, were evaluated based on interviews for operations information and grab samples process water and tailings composition.

2. Materials and methods

2.1. Facilities evaluated and operational characteristics

Operating parameters of visited facilities were obtained through interviews with the lead process engineers of the sites (Table 1). Sites 1, 2 and 5 are located within 50 km of Arequipa city. Sites 3, 4, and 6 are located within 45 km of Chala, a town that houses a large gold processing industry with 34 registered processing facilities in the surrounding area. All analyzed facilities utilized gold cyanidation in a carbon-in-pulp/electrowinning process. Operators and process engineers were interviewed to obtain operational parameters such as NaCN and NaOH consumption, water flows, water sources, and processing capacity. This information was used to calculate water flow balance, cyanide usage, and cyanide fate mass balances.

2.2. Water and tailing samples

Three types of water samples were collected at the studied cyanidation facilities: source water, barren water, and decant water. Source water refers to well-water used to cover water consumption during the cyanidation process and storage in the TSF. Barren water refers to samples taken from the effluent of the last carbon adsorption tanks prior to being pumped into the TSF. Decant water refers to water from the active TSF reclaim pond that is reused in the cyanidation process and was sampled from a smaller storage pond prior to being returned to the processing facility. Sampling locations are depicted in Fig. A1 of the supplementary information. pH and conductivity were measured immediately, after which grab samples were split into triplicates of 15 mL filtered (0.45 μm) and unfiltered water and stored in falcon tubes. To preserve cyanide concentrations of barren water and decant water samples, 1 mL of 1 M NaOH water was added to the falcon tube. The cyanide concentration was measured on the day of collection using a field spectrometer and Hach 862 test tubes and ICP, IC, and TOC analysis were carried out within 2 days. Overall storage times for samples ranged from 3 days to 10 days under the mentioned conditions.

Tailing samples were collected from inactive TSFs at sites 3 and 4, and from the beach of active TSFs at sites 1, 2, and 6. Each sample was taken in triplicates from surface and subsurface layers in random patterns over 12 m^2 and stored in 50 mL polyethylene vials for further analysis. A list of collected samples is summarized in Table A2.

2.3. *Sample analysis*

Conductivity and pH were measured using an Apera Instruments SX823-B multi parameter meter and probe. Free cyanide was measured manually with Hach Test 'N Tube (TNT) 862 using the colorimetric pyridine barbituric acid method. The tubes were then analyzed using a Hach DR 1900 field spectrometer. Total cyanide and weak acid dissociable cyanide complex (WAD) concentrations were determined using an OI Analytical Flow Water FS3700 Automated Chemistry Analyzer with 330092CT and 330090CT cartridges. The methods used for the analysis were ASTM D 7286–08 for total cyanide and ASTM 7511 for WAD. Elemental analysis of the waters was performed using inductively coupled plasma-optical emission spectroscopy (ICP-OES) (Avio-500, PerkinElmer, Fremont, CA). Inorganic anions in the water samples were measured by ion chromatography (IC) (Dionex 1500, Sunnyvale, CA). Total organic carbon (TOC) was measured using a TOC analyzer (Shimadzu, Columbia, MD).

The tailings samples were analyzed for pH by adding deionized water and a 0.01 M $CaCl_2$ water in a ratio of 1:2 as described in ASTM D4972–19. Water content and dry weight were determined using the ASTM D2216–92 standard methods by heating the soil sample to 110 °C for 24 h. If applicable, the supernatant of the tailings samples was decanted and analyzed using Hach Test 'N Tube (TNT) 862 test tubes, as well as ICP-OES, IC, and TOC. Metals within the tailing samples were extracted by US EPA method 3051 A and the extract analyzed using ICP-OES (Clesceri et al., 1998). The amount of water-soluble cyanide in the dry tailings was determined by leaching the tailings sample in a 10 % NaOH water over a time period of 48 h (Zagury et al., 2004). The leached water was then analyzed for free cyanide using Hach Test 'N Tube (TNT) 862 test kits.

3. Results and discussion

3.1. *Summary of the gold cyanidation process in the Arequipa region*

Most commonly, gold processing facilities in the Arequipa region operate independently from mines, purchasing ore from multiple sources. Ores of varying grades are mixed to form a consistent gold grade and are crushed in ball mills (Fig. A2A). Water, NaCN and NaOH are added in the appropriate quantity to the ball mills and conveyed to lixiviation tanks (Fig. A2B). The heap leach process was not observed for any of the visited sites. Pregnant water from the lixiviation tanks is then pumped into a series of adsorption tanks containing activated carbon to extract the gold-cyanide complexes (Fig. A2C). Gold loaded carbon is periodically removed from the adsorption tanks and eluted from the carbon with a concentrated sodium cyanide solution at 95–120 °C for subsequent electrowinning. The remaining barren water is then pumped into the TSF which is lined with a high-density polyethylene (HDPE) liner to contain the tailings material and avoid seepage (Fig. A2D). No thickening process was observed as water recycling from the TSF pond is common practice.

3.2. *Barren and decant water characterization*

Gold cyanidation facilities target a pH of at least 10.5 to maximize cyanide leaching efficiency and balance cyanide addition with free cyanide losses through volatilization (Perky et al., 1999). A pH of 11.5 is required to limit the fraction of volatile HCN to 1 % of free cyanide. Cyanide speciation based on a pK_a of 9.48 at 15 °C (Visual MINTEQ 3.1) and measured pH (Table A2), suggests that HCN/total free cyanide ranged from 5 to 40 % in barren solutions and 10 to 60 % in decant waters (Fig. 1). Volatilization is responsible for 90 % of natural cyanide removal from gold cyanidation effluents and tailings and has been the most common method of reducing cyanide concentrations (Kuyucak and Akcil, 2013). However, recent research indicates that volatilized HCN does not undergo significant natural degradation and contaminates air in the vicinity of gold cyanidation facilities, presenting considerable health and environment hazards (Brüger et al., 2018). Studies have even documented HCN emissions from gold processing facilities operating at high pH values (5 air/ppm of HCN at pH > 12 with ~1 g/L aqueous cyanide), suggesting our study locations likely have higher HCN releases due to lower operating pHs (Brüger et al., 2018). While HCN was detected at several sites through personal HCN detectors, precise air HCN concentrations were not directly measured.

The surface areas of TSF ponds were estimated to range from 20 m^2 at site 1 to 11,600 m^2 at site 6. Sites with larger TSF pond surface areas had lower measured pH values in the decant water. This observation is supported through a Pearson correlation with $r = -0.804$, and $p = 0.05$ values, suggesting a strong negative correlation between decant water pH and TSF pool sizes; as TSF surface area increases, pH in decant water decreases. CO_2 uptake from the atmosphere could be the main driver in pH reduction of gold cyanidation decant waters, which is expected to increase with higher TSF surface areas (Johnson, 2015). The pH of barren solutions also decreased at sites with larger TSF pond surface areas, this suggests the reuse of low pH decant water decreases the overall processing pH without adequate correction. Ore characteristics, water buffering capacity, and individual dosage control also impact the pH of the processing sites and are not captured by this correlation.

The speciation of cyanide and its concentrations greatly influence toxicological risks: high concentrations of free cyanide indicate high acute toxicity while high concentrations of metal-cyanide complexes suggest long term releases of cyanide and metal mobilization (Johnson, 2015). Samples obtained from the examined gold processing facilities were analyzed for cyanide speciation in both barren and decant waters (Fig. 2). WAD and strong acid dissociable complex (SAD) concentrations were determined by subtracting measured free cyanide concentrations from measured WAD concentrations and measured WAD concentrations from measured total cyanide concentrations respectively. Previous

Table 1
Summary of gold processing facilities in this study. NA = not available.

Site #	1	2	3	4	5	6
Location	Inland	Inland	Inland	Coastal	Inland	Coastal
Site type	Training	Processing	Mine and Processing	Processing	Processing	Processing
Estimated ore processing (metric tons/day)	10	15	200	250	350	430
NaCN consumption (kg/metric ton of ore)	2	4	2.4–2.5	NA	6	6.5
NaOH consumption (kg/metric ton of ore)	3	NA	2.2	NA	2–3.5	2–3
Process water flow (m3/day)	12	18	230–240	85*	420	462
Source water consumption (m^3/day)	NA	NA	150	30	NA	120
TSF status	Active	Active	Active and inactive	Active and inactive	Active and inactive	Active

NA = not available. * = uncertain information.

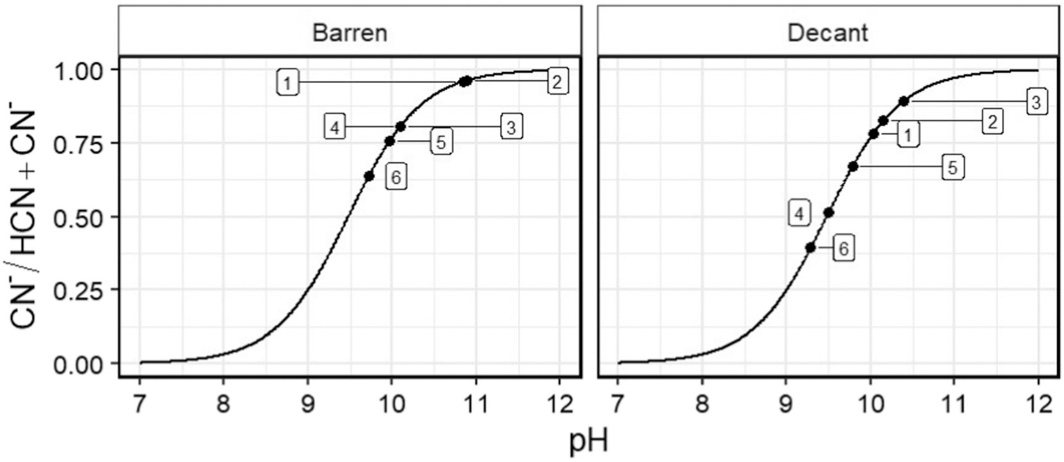

Fig. 1. Distribution of CN^- as a function of pH at 15 °C (pKa = 9.48) with corresponding measured pH from each site. The distribution curve of CN^-/HCN was generated using Visual MINTEQ 3.1.

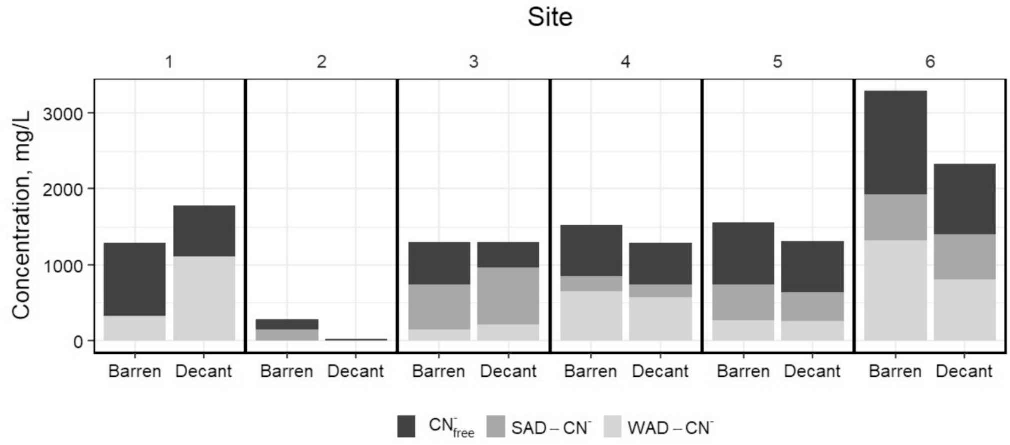

Fig. 2. Concentration of free cyanide, WAD, and SAD cyanide forms in the barren and decant waters. Total cyanide concentrations are the sum of free cyanide, WAD, and SAD.

studies have reported free cyanide concentrations in gold processing effluent between 11.2 and 530 mg/L, with most concentrations reported at <100 mg/L (Zagury et al., 2004; Johnson, 2015; Bakatula and Tutu, 2016; Khamar et al., 2015; Acheampong et al., 2013a; Vick et al., 1995; Acheampong et al., 2013b). Measured free cyanide concentrations at the six studied sites varied widely but were generally higher than literature

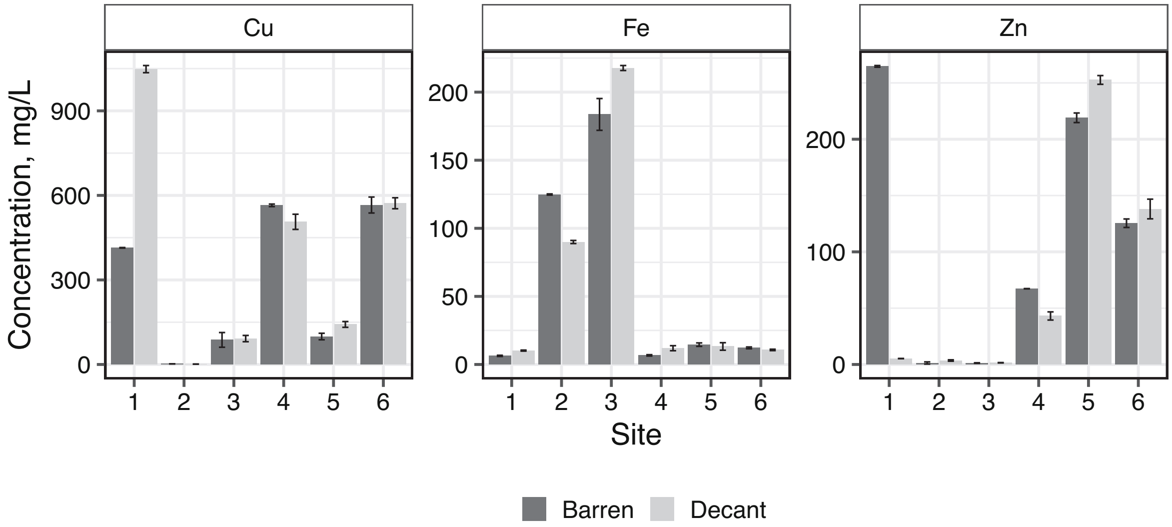

Fig. 3. Concentrations of copper, iron, and zinc in barren waters and decant waters.

reported values, ranging from 19.5 mg/L for the decant water of site 2, to 1375 mg/L for barren water of site 6 (average among the six sites was 753.8 mg/L). Low pH operating conditions require facilities to continuously add more cyanide to offset losses due to HCN volatilization.

High free cyanide concentrations are needed in gold cyanidation operations to overcome the demand created by presence of cyanide complexing metals (e.g., copper, iron, zinc) and to promote selective adsorption of gold-cyanide complexes onto activated carbon by shifting copper and other cyanide complexes to less adsorbable species (Dai et al., 2012; Larrabure and Rodríguez-Reyes, 2021). Copper specifically reduces the process efficiency by leaching faster than the targeted precious metals (Sceresini, 2005). Due to the elevated copper, iron and zinc levels reportedly observed in gold-bearing ores in the Arequipa region (De Haller and Fontbote, 2009), as well as the retention of metals in recycled process waters, we also observed high free cyanide concentrations at the six sites (Fig. 2). Results of WAD and SAD cyanide complex analysis of barren and decant water samples are presented in Fig. 2 and concentrations of copper, iron and zinc in Fig. 3. The presence copper- and zinc-cyanide complexes at sites 1, 4, 5, and 6 is indicated through relatively high concentrations of copper and zinc coinciding with high WAD concentrations. Sites 3, 5, and 6 had high SAD concentrations, with site 3 having the highest concentration of iron. However, barren and decant water from Sites 5 and 6 did not have high measured iron concentrations which may be due to complexed iron not being measured through ICP analysis. Positive correlations were found between free cyanide and sum of complexed cyanide ($r = 0.787$ and a $p = 0.063$ for the barren water and $r = 0.844$ with a $p = 0.035$ for the decant water), and between copper and zinc concentrations and WAD cyanide ($r = 0.751$ with a $p = 0.085$ for decant waters and $r = 0.978$ with a $p = 0.001$) in both barren and decant waters indicating increased free cyanide dosing to address cyanide complexation to non-target metals. Additional metals of interest including As, Cd, Ni and Pb also occur naturally in the ores and soil of the Arequipa region (Huerta Alata et al., 2023) and were measured in barren and decant waters (Fig. A4) at significantly greater concentrations than environmental quality standards for rivers established by the Peruvian Ministry of the Environment (MINAM) (MINAM, 2017).

Due to the lack of surface water in the coastal areas of the Arequipa region, water demands of the processing facilities are met either using industrial water of uncertain origin or local groundwater. At sites 4 and 6, source water is primarily made up of locally sourced groundwater. We observed both the source water to have higher than expected conductivity (48,200 μS/cm and 39,100 μS/cm respectively) compared to their inland counterparts (1192 μS/cm and 1043 μS/cm for sites 3 and 5 respectively) (Fig. A4). Extraction of groundwater for industrial purposes likely causes saltwater intrusion into the aquifer (Sherif and Singh, 2002), which causes high conductivities in gold cyanidation barren and decant waters and leaves Chala dependent on importing domestic water from surrounding areas.

In comparison to coastal facilities, inland facilities had significantly lower barren and decant water conductivities due to the usage of low conductivity source water (Fig. A4). Water samples from site 4 measured the highest conductivity in this study at 70,900 μS/cm for the decant, and 68,900 μS/cm for barren water, whereas site 2 measured the lowest conductivities at 4760 μS/cm for barren, and 5760 μS/cm for decant water. The reuse of decant water in the processing facilities also increases ion concentrations due to evaporative water losses (15 m^3/day from site 3 and 60 m^3/day from site 6, discussed in Section 3.3). The salinity and composition of source water is important for processing facilities as the water quality directly influences the efficiency of gold cyanidation, and was reported to be one of the major challenges coastal processing facilities face (Perky et al., 1999; Adams, 2001). Very alkaline saline conditions may further promote co-precipitation of metal hydroxides which results in acidic products that drive the pH down (Perky et al., 1999; Van Vuuren et al., 2000). Precipitation of metal hydroxides and diminishing gold dissolution rate at pH > 9.5 may be a contributing factor in gold cyanidation facility's to operate at pH < 11.5 (Marsden and House, 2006).

3.3. *Water balance and fate of cyanide*

The overall water budget of a gold cyanidation facility can significantly influence the management of process waters and other operations such as water treatment processes and chemical dosing. For facilities with negative water balances and no wastewater discharge (this study), operators estimated that 35–80 % of barren water was recovered as decant water for reuse. With no processes for removal of constituents including cyanide and cyanide complexes (WAD and SAD), metals, and other anions and cations, water recycling can result in accumulation of these constituents within unit processes and tailings and also negatively impact cyanidation efficiency (Bahrami et al., 2007). Information gained through site visits was used to develop a water usage model for sites 3 and 6 which is presented in Fig. 4. These sites have two contrasting operational water schemes: site 3 uses more source water at the start of the process (62.5 %) compared to site 6, which uses more recycled water (71.5 %). These differences are likely based on economic reasons. Site 3 opts for higher overall water consumption at a lower source water cost (4.4 PEN/m3) in favor of higher process water quality. Conversely, site 6 pays 12 PEN/m3 for their source water and to offset this higher cost, they recycle more of their decant water (water costs obtained through personal correspondence). Detailed water consumption and water usage profiles hold important information to optimize decant water recycling, which impacts economic considerations, process water quality, TSF contaminant loading, and effluent water discharges (if applicable).

Next, we aimed to characterize the fate of cyanide throughout the cyanidation process, including loss from volatilization, degradation, or accumulation in TSFs. Cyanide consumption at all sites ranges from 2 to 6.5 kg NaCN/metric ton of ore (Table 1), with sites 2, 5, and 6 being on the high end of reported average consumptions (1–6 kg NaCN/metric ton of ore) (Kondos et al., 1995; Kianinia et al., 2018). Sites 3 and 6 were chosen for a more in-depth analysis (Fig. 5). The percentage of new free cyanide to total free cyanide (recycled free cyanide + new free cyanide) at the start of the gold cyanidation process is similar between sites 3 and 6 (88.8 % and 82.3 %, respectively). Most of the free cyanide consumption occurs during the lixiviation process when cyanide complexes with metals. Low Cu and Zn concentrations (WAD forming metals) and high Fe (SAD forming metal) concentrations at site 3 (Fig. 2) result in more CN^- being bound to SADs than WADs. Conversely, high Cu and Zn, and low Fe concentrations at site 6 result in more CN^- being bound to WADs than SADs.

The gold-cyanide binding efficiency is comparable between the facilities, measuring both at 0.15 % demonstrating factors such as pH and conductivity do not appear to influence cyanide efficiency. Gold-cyanide binding efficiency was calculated with the following equation:

$$Gold - cyanide\ binding\ efficiency = 2 * \frac{gold\ grade * plant\ capacity}{M_{Au}} * \frac{M_{NaCN}}{NaCN\ consumption} \tag{1}$$

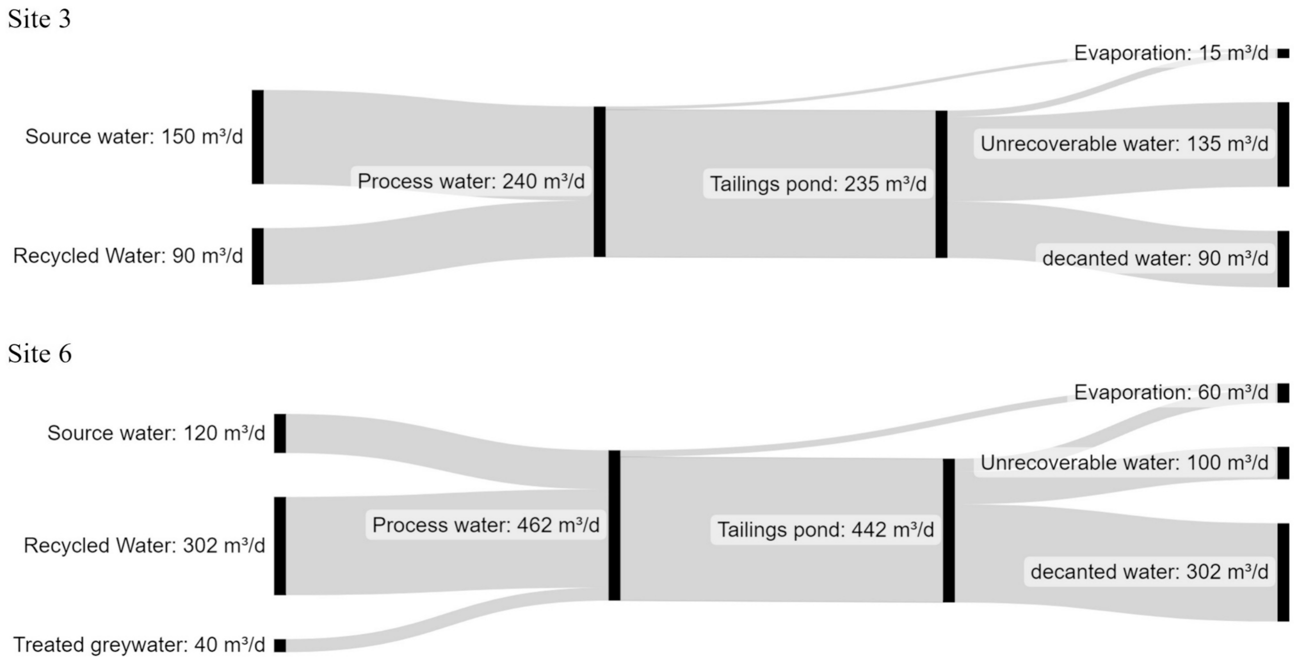

Fig. 4. Water flows and consumptions of sites 3 and 6.

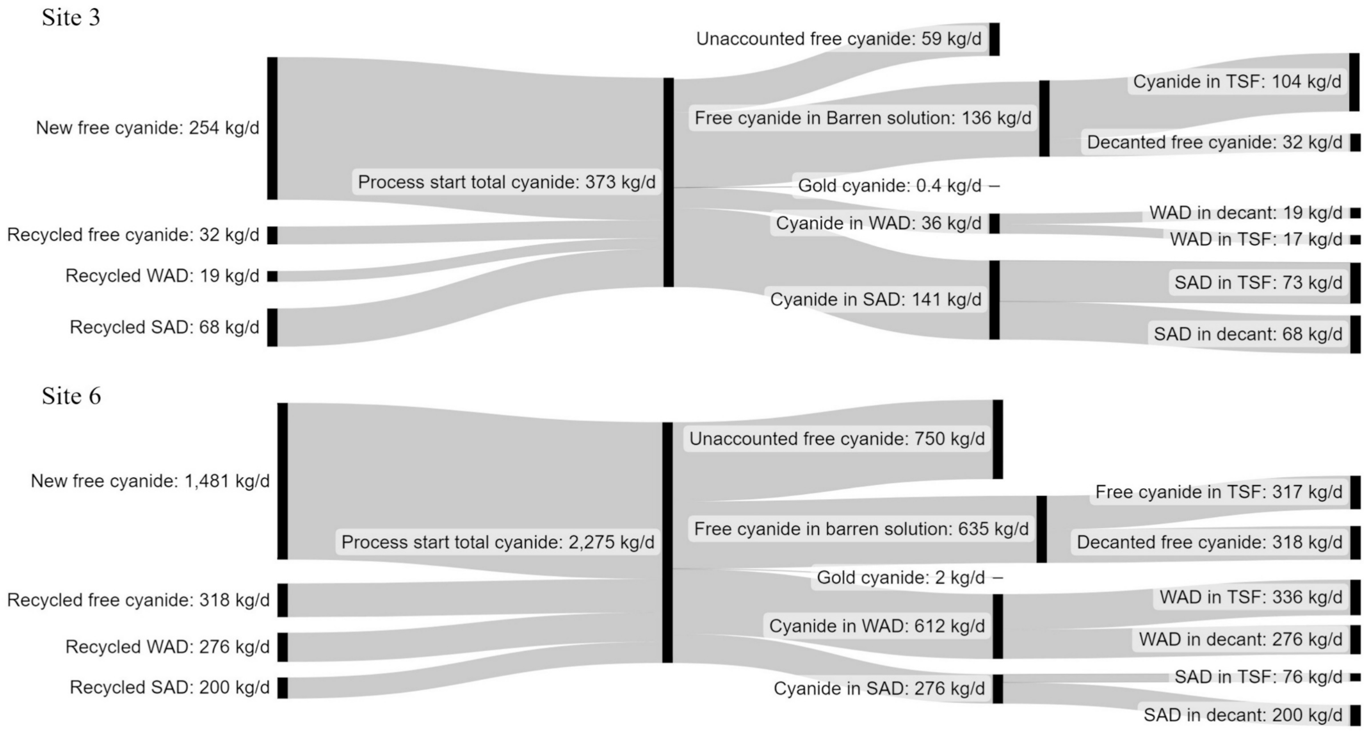

Fig. 5. Process flow of Cyanide at Sites 3 and 6.

Contrary to concerns brought up by process engineers at the study locations and La Brooy et al. (La Brooy and Muir, 1993), extensive decant water reuse does not appear to impact cyanide binding efficiency. This finding may enable the use of saline source waters, potentially relaxing the demand on groundwater wells and decreasing the amount of saltwater intrusion into the aquifer. However, more research is recommended to definitively ascertain co-variation and interdependence of operational parameters from saline water within the gold cyanidation process.

Unaccounted cyanide values, shown in Fig. 5 (middle section) encompass free cyanide lost to natural degradation and volatilization during the gold cyanidation process. Site 6 had a higher unaccounted for free cyanide percentage (41.6 %) compared to site 3 (20.6 %). As reported in Section 3.2, the pH of site 6 is lower than site 3, which increases HCN volatilization and causes higher unaccounted cyanide losses of site 6. Free cyanide remaining in the barren water is pumped into the TSF where it is either recycled through the decant water or settles in the TSF. Cyanide that remains in the TSF volatilizes, degrades naturally, persists in the porewater of the tailings, or dries out as NaCN (Johnson, 2015). The amount of free cyanide recycled through decant water is highly dependent on the amount of decanted water recycled. Site 6 recycles over twice the cyanide (50.1 %) compared to Site 3 (23.5 %) due to its higher decant water reuse.

At the time of the field sampling, none of the cyanidation facilities in this study participated in the ICMC and consequently, no additional treatment methods were used to reduce TSF cyanide concentrations to the recommended standard of 50 mg/L total cyanide. High cyanide concentrations in the TSF ponds are desirable for processing facilities with no effluent discharge as cyanide from decant waters can supplement overall cyanide consumption. However, volatilization of free cyanide is a major cyanide sink within the gold cyanidation process and an environmental concern. Processing water pH and TSF pond pH could be increased to avoid cyanide losses through volatilization and improve cyanide recycling through decant water. This would decrease the amount of new cyanide needed to maintain free cyanide levels while also mitigating the environmental and health hazard of HCN volatilization but could also lead to unwanted precipitation in high conductivity waters. Conversely, increasing free cyanide concentrations in the TSF poses an increased risk to wildlife, especially birds, with several facilities using bird deterrents such as speakers and shooting of blanks. The recovery of cyanide through novel technologies such as AVR, SART, and membrane contactors could provide a more direct pathway for cyanide reuse in the cyanidation process, greatly limiting cyanide discharge to the TSF altogether (Kuyucak and Akcil, 2013; Dai et al., 2012; Hammer et al., 2023; Whittle and Pan, 2020).

3.4. Tailings solids characterization

Tailings solids samples were analyzed for water content, pH, cyanide, and metals (Tables 2 and 3). The inactive TSF surface and subsurface samples retained an average of 11 % and 28 % water content, respectively, and the active TSF surface and subsurface layers retained an average of 24 % and 31 % water content, respectively. These data demonstrate that in subsurface layers of inactive TSF, minimal water loss is observed, even after 1 year of inactivation (timeline provided through communication with process engineers). Finely milled tailings and the formation of crusts caused by high salinity (Fig. A6) facilitate water retention at inactive facilities. To accurately determine the influence of water retention on TSF safety, an assessment of tailings shear strength and tailings liquefaction throughout the depth of the TSF is needed.

Next, we compared the free cyanide concentrations in the surface layer of the inactive TSF at sites 3 (38,589 mg/kg) and the active TSF at site 6 (731 mg/kg) (Table 2). Both concentrations were higher than previously reported values of 3–53 mg of CN^- per kg of tailings (Zagury et al., 2004; Bakatula and Tutu, 2016; Lemos et al., 2020; Fourie et al., 2008). The presence of high free cyanide concentrations in the inactive TSF of site 3 further indicates that the retention of free cyanide in TSFs might be greater than previously expected, increasing the potential environmental harm and impact of inactive TSFs. Free cyanide was also measured on the surface layer of the active TSF of site 1 (19.6 mg/kg), however, no free cyanide was found on the surface layer of sites 2 (active) and 4 (inactive). The tailings of site 2 likely exhibit lower cyanide concentrations due to low effluent flow and low effluent cyanide concentrations. In contrast, site 4 with its comparatively low tailings solids pH promotes cyanide mobility, potentially contributing to its lower cyanide content. For sites 1, 3, and 6 it was further found that the surface layers contained more free cyanide than the subsurface layers, suggesting that free cyanide is migrating and accumulating at the surface of the TSF. The retention of free cyanide in TSFs is influenced by a variety of factors such as volatilization, complexation, precipitation, adsorption, biological transformation, and sulfurization (Johnson, 2015). While the retention of free cyanide in TSFs is often low due to these processes, some TSFs like site 3 may retain dangerous concentrations of free cyanide even after months of inactivation.

As metal-cyanide complexes are less susceptible to mechanisms governing the removal of free cyanide, we expected WAD and SAD concentrations in the TSFs to be higher than free cyanide concentrations. Indeed, at all sites WAD and SAD metal-cyanide concentrations exceeded free cyanide concentrations (Table 2). Notably high concentrations of WADs were found in the surface layers of sites 3 (78,614 mg/kg) and 6 (1483 mg/kg). Similarly, SADs analysis revealed elevated concentrations at all TSFs with the surface layer of site 1 (6807 mg/kg) and site 3 (14,552 mg/kg) exhibiting the highest concentrations. Elevated concentrations of WADs and SADs generally coincided with higher copper, zinc, and iron concentrations in the TSF (Table 3). High iron concentrations in samples with low SAD concentrations suggest incomplete leaching of iron during gold cyanidation or natural degradation of the release of iron through natural degradation of iron-cyanide complexes in the TSF. High concentrations of metal cyanide complexes highlight potential long-term environmental concerns through the gradual release of free cyanide and mobilization of metals from the TSF (Johnson, 2015). Tailings with total cyanide concentrations above 5900 mg/kg and leachate (barren water) with total cyanide concentrations >64 mg/L are classified very dangerous tailings by Arequipa's environmental authority (Eppers, 2014). All sites in this study complied with the recommended standards for the deposition of very dangerous tailings into a TSF with leachate control and both chemical and physical stabilization. However, monitoring programs of inactive and abandoned TSFs, overseen by the Peruvian Ministry of Mining (MINAM, 2001), could be significantly strengthened by incorporating cyanide assessments to a more comprehensive risk assessment of TSFs in this region.

Capillary action is likely transporting cyanides and salts to the TSF surface and water evaporation deposits free cyanide and cyanide complexes in the remaining pore water and tailings solids (Shehong et al., 2005). High cyanide concentrations in the surface layer of TSFs increase exposure potential for wildlife and enhance the susceptibility of cyanide contamination through eolian mobilization. Dust and aerosol transport in arid environments like the Atacama Desert are well studied and show insoluble dust deposition originating from local soil environments (Li et al., 2019). The TSFs in this study, located in the outskirts of the Atacama Desert, likely experience similar erosion and aerosolization of cyanide laden dust from the dried-out surfaces of TSFs. This potentially contributes to the low cyanide concentrations found in the inactive TSF of site 4, compared to the active TSF of site 6. Chronic cyanide exposure, even in low concentrations, is known to cause adverse health effects

Table 2
Water content, tailings solids pH, and cyanide concentrations of analyzed tailings. Numbers represent the sample site, S represents surface samples and U represents subsurface samples. Standard errors are shown in Table A3.

	1S	1 U	2S	3S	3 U	4S	4 U	6S	6 U
TSF status	Active	Active	Active	Inactive	Inactive	Inactive	Inactive	Active	Active
Gravimetric water content (%)	19 %	35 %	29 %	12 %	35 %	9 %	20 %	24 %	24 %
pH, DI-water	11.15	10.63	9.78	10.43	10.36	8.03	8.28	9.2	8.96
pH 0.01 M $CaCl_2$	11.2	10.38	9.15	10.27	9.55	7.86	8.07	8.97	8.68
CN^- (mg/kg – tailings)	19.6	0.5	0	38,589	1130	0	0	731	349
WAD – CN^- (mg/kg – tailings)	357	17.5	1.9	78,614	4234	0.28	2.9	1483	852
SAD – CN^- (mg/kg – tailings)	6807	910	89.3	14,552	2485	96.3	283	637	383

Table 3
Concentration of metals in tailings. Standard errors are shown in Table A3.

Metal, mg/kg	1S	1 U	2S	3S	3 U	4S	4 U	6S	6 U
Al	3647	2771	22,389	5464	15,981	5152	7718	5007	6452
As	239.1	155.6	109.5	240.1	131.1	4611	3506	6653	5547
Cd	22.3	5.2	4.2	19.1	45.2	346.0	708.6	558.5	930.0
Cu	3272	7054	139.0	107,243	3469	3169	1987	4205	3861
Fe	210,058	14,078	49,136	19,808	30,476	75,798	69,704	147,015	122,698
Ni	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Pb	1403	294.2	68.3	441.8	51.1	3777	3129	11,250	8424
Zn	568.1	313.0	41.2	156.7	34.8	3966	4347	6739	6904
Co	53.7	14.0	29.9	84.5	25.2	62.9	58.4	63.5	49.8

observed in humans and animals (Cliff et al., 2015; Okolie and Osagie, 1999). Knowledge gaps exist regarding potential exposure pathways and the health consequences of inhaling cyanide-laden dust originating from TSFs deserves further investigation. To mitigate the risk of cyanide-laden dust contamination, we recommend the implementation of dust control and cover designs after TSF inactivation and prior to TSF closure.

Gold cyanidation leaves behind large amounts of mobilized heavy metals which are of high-risk for the sensitive water systems of southern Peru (Karimi et al., 2010). Arsenic occurs naturally in rock and soil throughout the Arequipa region and is present in the tailings at concentrations up to 6653 mg/kg (Table 3), 1000 times higher than typical As soil concentrations (6.5 mg/kg) (Huerta Alata et al., 2023). Other metals of concern, including Cd (930.0 mg/kg), Pb (11,250 mg/kg), Zn (6604 mg/kg), and Co (84.5 mg/kg), are present at concentrations exceeding regular soil levels (Cd = 0.18 mg/kg; Pb = 9 mg/kg; Zn = 30 mg/kg; and Co = 4 mg/kg) by >200, 1000, 200, and 200 times respectively (Huerta Alata et al., 2023). Generally, metals were found in higher concentrations on the TSF surface than the subsurface, indicating mobilization of metals through cyanide complexation plays a major role in the heterogenous distribution of metals. Sulfite weathering and metals depletion further increase tailings metal heterogeneity, creating enrichment and depletion potentially not fully captured by our samples (Blight and Bentel, 1983; Nikonow et al., 2019). Metal buildup and surface enrichment heighten environmental exposure risks through runoff and aerosolization, further highlighting the need for dust control or cover strategies.

Site visits highlighted tailings dam failures as a major concern of residents near TSFs. This concern is especially pronounced in Chala as many TSFs are located upslope from residential areas, potentially putting them into the spill zone of a potential dam breach. Historical data reveals that earthquakes are a major contributing factor in causing tailings dam failures, with 17 % of incidents over the last 100 years being caused by earthquakes (Lyu et al., 2019). The Arequipa region of Peru exhibits a high frequency of seismic events, with 53 earthquakes recorded within the last year alone (U.S. Geological Survey, n.d.). The 2001 southern Peru earthquake with a magnitude of 8.4 highlighted the susceptibility of TSFs to seismic activity by causing liquefications and partial damage to multiple TSFs (Rodriguez-Marek et al., 2001). The Medium- and small-scale processing facilities are estimated to operate TSFs containing up to 500,000 m^3 of tailings, a volume 5 times greater than the material released during the Baia Mare disaster. Consequently, the release of such quantities of tailings in the event of TSF failures would cause widespread cyanide and heavy metal contamination.

4. Conclusions

Extensive decant water recycling was found to be a major factor in the high concentrations of cyanide, cyanide complexes, and metals in both gold cyanidation effluents and tailings. The accumulation of these constituents is further increased by the absence of active effluent treatment at the visited sites. Decant water recycling was further found to increase the conductivity of process waters. The potential presence and accumulation of mercury cyanide complexes in process water and tailings through the reprocessing of mercury amalgamation tailings deserves further investigation as these complexes are persistent and pose harm through bioaccumulation.

This study reveals high amounts of unaccounted free cyanide throughout the gold cyanidation process. Analysis through the mass balance of the measured cyanide concentrations and the reported dosing of sodium cyanide indicated substantial losses of free cyanide attributed to volatilization of HCN. The presence of vapor phase cyanide in the ambient air within the processing facilities poses considerable health risks to workers.

TSF were found to retain cyanide, even after being inactive for several months. Surface layers appeared to have higher cyanide concentrations than lower depths. Elevated surface cyanide concentrations in TSFs heighten the potential for the dispersion of cyanide-laden dust with wind events as the sites studied did not implement dust mitigation on inactive TSFs. Metal cyanide complexes (WAD and SAD) were found to be a significant contributor to the total cyanide remaining in TSFs. Exposure pathways and health effects of cyanide-laden dust are unknown and warrant further investigation.

A more extensive study of TSF surfaces is needed to evaluate the heterogeneity of cyanide and metals concentrations found in the tailings. Samples taken over a wider area would give a better understanding of metal enrichment- and depletion zones. Deeper subsurface samples along a depth gradient would also help better understand the transport and mobility of metals and cyanide within the TSF and give a more representative view of the TSF's overall metals concentration.

CRediT authorship contribution statement

Vincent Hammer: Writing – original draft, Visualization, Methodology, Investigation, Formal analysis, Data curation. **Johan Vanneste:** Writing – review & editing, Supervision, Investigation, Funding acquisition, Conceptualization. **Francisco D. Alejo-Zapata:** Investigation, Conceptualization. **Julia Zea:** Investigation. **Héctor G. Bolaños-Sosa:** Investigation, Conceptualization. **Carlos A. Zevallos Rojas:** Investigation. **Linda A. Figueroa:** Writing – review & editing, Data curation, Conceptualization. **Aaron Malone:** Writing – review & editing, Project administration, Investigation, Conceptualization. **Christopher Bellona:** Supervision, Methodology, Funding acquisition, Conceptualization. **David C. Vuono:** Writing – review & editing, Supervision, Project administration, Conceptualization.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

Acknowledgements

This project was funded by the Center for Mining Sustainability. This is a collaboration between the Universidad Nacional San Agustin and Colorado School of Mines. We would like to thank Henry G. Polanco-Conejo for his contributions towards the center and this paper. Chiang Siew and Anica Newmann are acknowledged for writing assistance and proof reading. The authors thank the editor and reviewers for their thoughtful feedback and effort that improved the article.

Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.scitotenv.2024.174034.

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