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Sequential Acid–Cyanide Diagnostic Leaching to Quantify Cyanide-Soluble Copper in a Gold-Bearing Oxidized Copper Ore

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ABSTRACT **Original Research Article**

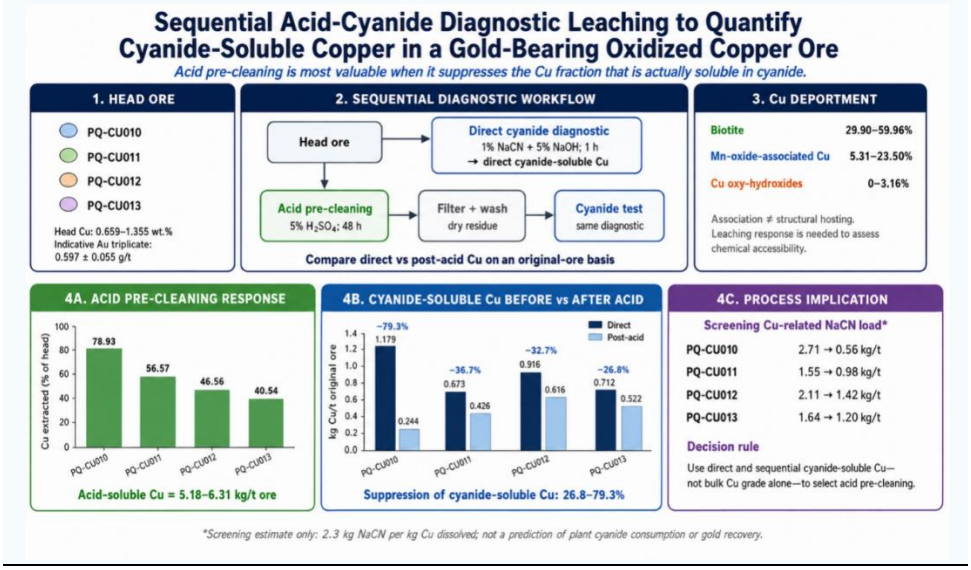
Copper-bearing minerals can strongly interfere with gold cyanidation because dissolved copper forms stable Cu(I)–cyanide complexes and competes for free cyanide. This study applies sequential acid–cyanide diagnostic leaching to four oxidized copper ore samples from a gold-bearing ore population to quantify cyanide-soluble Cu and determine whether sulfuric acid pre-cleaning materially suppresses that burden. Head Cu concentrations ranged from 0.659 to 1.355 wt.%. A triplicate head-Au determination on PQ-CU010 gave 0.66, 0.56, and 0.57 g/t, corresponding to 0.597 ± 0.055 g/t Au. PQ-CU010 was considered representative of the same ore population as PQ-CU011–PQ-CU013; therefore, this mean was adopted as the representative head-Au value for the investigated sample set, while Au was not independently determined in the other three samples. A 48 h sulfuric-acid diagnostic treatment removed 40.54–78.93% of head Cu, equivalent to 5.18–6.31 kg Cu/t ore. Direct cyanide diagnostic tests dissolved 0.673–1.179 kg Cu/t, corresponding to 5.57–17.88% of head Cu. After acid pre-cleaning, the cyanide-soluble Cu load decreased to 0.244–0.616 kg/t on an original-ore basis, representing suppression of 26.8–79.3%. Mineralogical analysis assigned 76.5–93.8% of Cu to biotite-associated populations, 5.3–23.5% to Mn-oxide-associated populations, and 0–3.2% to Cu oxy-hydroxides. The response was strongly sample-specific: PQ-CU010 showed the greatest suppression of cyanide-soluble Cu, whereas PQ-CU013 retained a comparatively high post-acid burden despite substantial acid-soluble Cu removal. Because paired gold-extraction and actual NaCN-consumption tests were not included in the analyzed dataset, the study supports pretreatment selection on a diagnostic basis but does not establish plant-scale reagent savings, gold-recovery improvements, or an economic optimum. Direct and sequential cyanide-soluble Cu measurements provide a more defensible process-selection criterion than bulk Cu grade alone.

Keywords: Acid Pre-Cleaning, Cyanide-Soluble Copper, Gold Cyanidation, Diagnostic Leaching, Copper Deportment, Oxidized Copper Ore.

Highlights

- Sequential diagnostic leaching identified 0.673–1.179 kg Cu/t of directly cyanide-soluble copper across the four investigated samples.
- Sulfuric-acid pre-cleaning extracted 40.54–78.93% of head Cu and suppressed the subsequent cyanide-soluble Cu load by 26.8–79.3%.
- Copper deportment was dominated by biotite-associated Cu (76.5–93.8%); Mn-oxide-associated Cu represented 5.3–23.5% and was associated with lower chemical reactivity in the present sample set.
- Pretreatment selection should be based on direct and post-acid cyanide-soluble Cu, not on bulk Cu grade alone.

Graphical Abstract



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Introduction

Cyanidation remains one of the principal hydrometallurgical routes for gold recovery because cyanide combines high selectivity for Au with mature solid–liquid separation and downstream recovery technologies. Its long industrial history has nevertheless been accompanied by continuing efforts to reduce reagent consumption and improve performance for refractory and impurity-rich ores (Udupa et al., 1990).

Copper is among the most consequential impurities in gold cyanidation. Dissolved Cu can consume free cyanide, increase weak-acid-dissociable cyanide loading, interfere with activated-carbon adsorption and solution management, and increase downstream cyanide recovery or destruction requirements. These penalties are widely recognized in the processing of copper-bearing gold ores and concentrates (Dai et al., 2012; Maganga et al., 2023; Sceresini, 2005).

In alkaline cyanide solution, dissolved copper forms stable Cu(I)–cyanide complexes, principally $Cu(CN)_2^-$, $Cu(CN)_3^{2-}$, and $Cu(CN)_4^{3-}$, with their distribution controlled by free-cyanide concentration, Cu/CN molar ratio, pH, temperature, and solution composition (Kyle & Hefter, 2015). Under cyanide-deficient or copper-rich conditions, copper speciation and free-cyanide availability can strongly influence Au dissolution and the extent of Cu suppression (Breuer et al., 2005; Deschênes & Prud'homme, 1997; Oraby & Eksteen, 2015, 2016).

The mineralogical form and chemical accessibility of Cu are therefore as important as bulk Cu grade. Chalcopyrite is comparatively resistant under many conventional cyanide conditions, whereas oxidized, secondary, surface-associated, or otherwise labile Cu populations can be substantially more reactive. Several mitigation strategies have been investigated, including ammonia pretreatment (Bas et al., 2015), EDTA-based Cu removal (Kuzu et al., 2026), acidic pretreatment (Portilla et al., 2020), and alternative sulfate–chloride treatment schemes for oxidized Au–Cu ores (Sekisov et al., 2018). Although these approaches are not directly equivalent, they collectively demonstrate that reactive Cu must be characterized and managed before process selection.

Pereira and Barbosa (2017) demonstrated the process relevance of sulfuric-acid pre-cleaning for copper-bearing gold ore. Their work showed that removal of acid-soluble Cu ahead of cyanidation can decrease subsequent Cu complexation by cyanide and reduce the associated reagent burden, while also emphasizing that pretreatment response depends strongly on Cu mineralogy and chemical accessibility.

The present study applies that sequential diagnostic concept to four oxidized copper-ore samples containing gold. The objectives are to quantify direct cyanide-soluble Cu, determine the fraction of that burden suppressed by sulfuric-acid pre-cleaning, relate the response to mineralogical Cu deportment, and evaluate whether bulk Cu grade alone is an adequate pretreatment criterion. The study intentionally treats direct and post-acid cyanide-soluble Cu as operational diagnostic descriptors; it does not assume that the diagnostic values equal plant-scale Cu dissolution or NaCN consumption.

Materials and Methods

Ore Samples and Data Reconciliation

Four laboratory samples were evaluated and, for publication, are identified exclusively as PQ-CU010, PQ-CU011, PQ-CU012, and PQ-CU013. Original internal sample identifiers are omitted from the publication-facing dataset.

Original analytical and operating records were audited before calculation. Where pre-existing spreadsheet summary cells showed inconsistencies between sample identification and analytical values, calculations were reconstructed directly from the original duplicate assay records. The original sample descriptions associated with each cyanide-liquor analysis were retained as the primary traceability criterion during reconciliation.

This reconciliation procedure was necessary to prevent spreadsheet-label and formula-offset errors from propagating into the metallurgical interpretation. No missing experimental values were generated or statistically imputed.

Head Chemical and Mineralogical Characterization

Head Cu concentration was obtained from the archived chemical dataset, and major-element composition was complemented by automated mineralogical characterization. Copper deportment among the principal Cu-associated mineral groups was taken from the archived mineralogical dataset. The surviving records do not retain sufficient instrument/model metadata to reconstruct the complete analytical method for every determination; therefore, no unrecorded instrumental details are inferred in this paper.

The analyzed samples contained less than 0.01 wt.% sulfur, indicating that sulfide minerals were not major components of the investigated material. Copper was predominantly associated, by automated mineralogical classification, with biotite and manganese oxides, with minor Cu oxy-hydroxides. These associations are treated as mineralogical deportment classes rather than as proof of the structural incorporation of Cu into a specific mineral lattice.

A separate initial gold determination was performed in triplicate on sample PQ-CU010, producing values of 0.66, 0.56, and 0.57 ppm. For a solid ore, these values are numerically equivalent to g/t, yielding an arithmetic mean of 0.597 g/t Au, a standard deviation of 0.055 g/t, and a relative standard deviation of 9.23%. Sample PQ-CU010 was considered representative of the same ore population represented by PQ-CU011, PQ-CU012, and PQ-CU013; therefore, the mean gold grade of 0.597 g/t Au was adopted as a representative head-Au value for the four investigated samples. This value should be interpreted as a representative grade for the investigated ore population rather than as an independently determined, sample-specific Au assay for PQ-CU011, PQ-CU012, and PQ-CU013.

Table 1 summarizes the principal chemical and mineralogical characteristics relevant to copper reactivity.

Table 1. Chemical composition and copper deportment of the investigated samples

Sample	Cu (wt.%)	Fe (wt.%)	Mn (wt.%)	S (wt.%)	Cu in biotite (%)	Cu in Mn oxides (%)	Cu in Cu oxy-hydroxides (%)
PQ-CU010	0.659	36.00	0.184	<0.01	93.84	5.31	0.85
PQ-CU011	0.951	17.33	0.178	<0.01	83.40	16.60	0.00
PQ-CU012	1.355	32.10	0.639	<0.01	90.47	6.37	3.16
PQ-CU013	1.278	24.90	1.396	<0.01	76.50	23.50	0.00

Source: Authors' calculations from experimental chemical and automated-mineralogy data.

Acid-Soluble Copper Test

Approximately 50 g of each ore sample were subjected to sulfuric-acid leaching. The operating records specify a nominal 5% H_2SO_4 solution, an initial liquid volume of 500 mL, a contact time of 48 h, and a combined final liquor-plus-wash volume of 2,000 mL after filtration and washing. The archived record does not preserve the concentration basis for the nominal percentage or sufficient information to reconstruct the temperature and agitation conditions; therefore, these values are not inferred.

The acid-soluble copper extraction, E_acid, was calculated from:

$$E_{acid} = \frac{C_{acid} V_{acid}}{m_0 w_{Cu}}$$

where C_acid is the Cu concentration in the combined acid liquor, V_acid is the final combined liquor volume, m_0 is the initial ore mass, and w_Cu is the head-Cu mass fraction. The equation gives a fractional extraction; reported extraction percentages are 100 × E_acid.

The acid test is interpreted as a diagnostic extraction procedure rather than an optimized industrial leach. The nominal acid concentration, long contact time, and high liquid-to-solid ratio were used to characterize acid-labile Cu and should not be transferred directly to an industrial flowsheet.

Direct Cyanide-Soluble Copper Test

The direct cyanide test was performed on approximately 2 g of untreated ore. The archived work program specifies nominal solutions of 1% NaCN and 5% NaOH, an initial solution volume of 280 mL, a final liquor-plus-wash volume of 500 mL, and a 1 h contact period. As for the acid test, the surviving record does not preserve the concentration basis of the nominal percentages or sufficient information to reconstruct temperature and agitation conditions.

The cyanide-soluble copper loading, Q_Cu, CN, was expressed directly in kg Cu/t ore:

$$Q_{Cu,CN} = \frac{C_{CN} V_{CN}}{m_{test}}$$

where C_CN is the Cu concentration in the final cyanide liquor, V_CN is the final liquor-plus-wash volume, and m_test is the dry ore mass used in the diagnostic test. With consistent units, mg Cu/g ore is numerically equivalent to kg Cu/t ore.

This parameter is an operational measure of cyanide-soluble, potentially cyanide-burdening Cu under the standardized diagnostic conditions. It should not be interpreted as the dissolved-Cu concentration or the NaCN consumption expected in an industrial CIL, CIP, or heap-leaching circuit because the diagnostic test deliberately uses reagent concentrations and liquid-to-solid conditions that differ from plant practice.

Sequential Acid–Cyanide Test

After acid treatment, the solid residue was filtered, washed, dried, and representatively subsampled before being subjected to the same cyanide-soluble Cu procedure used for the untreated ore.

Because acid leaching caused a measurable loss of solid mass, cyanide-soluble Cu measured on the acid residue was converted to an original-ore basis:

$$Q_{Cu,CN,post} = Q_{Cu,CN,residue}\left(\frac{m_{residue}}{m_0}\right)$$

The relative suppression of cyanide-soluble Cu due to acid pre-cleaning was calculated as:

$$R_{Cu,CN} = 100\left(1 - \frac{Q_{Cu,CN,post}}{Q_{Cu,CN,direct}}\right)$$

Where Q_Cu,CN,residue is the cyanide-soluble Cu measured per unit mass of dried acid residue and m_residue/m₀ is the residue-yield fraction. This original-ore normalization permits direct comparison of Cu burden before and after acid pre-cleaning and avoids overstating the post-acid response solely because the residue mass is lower than the original ore mass.

The experimental sequence used to distinguish direct cyanide-soluble Cu from the fraction suppressed by sulfuric-acid pre-cleaning is summarized in Figure 1.

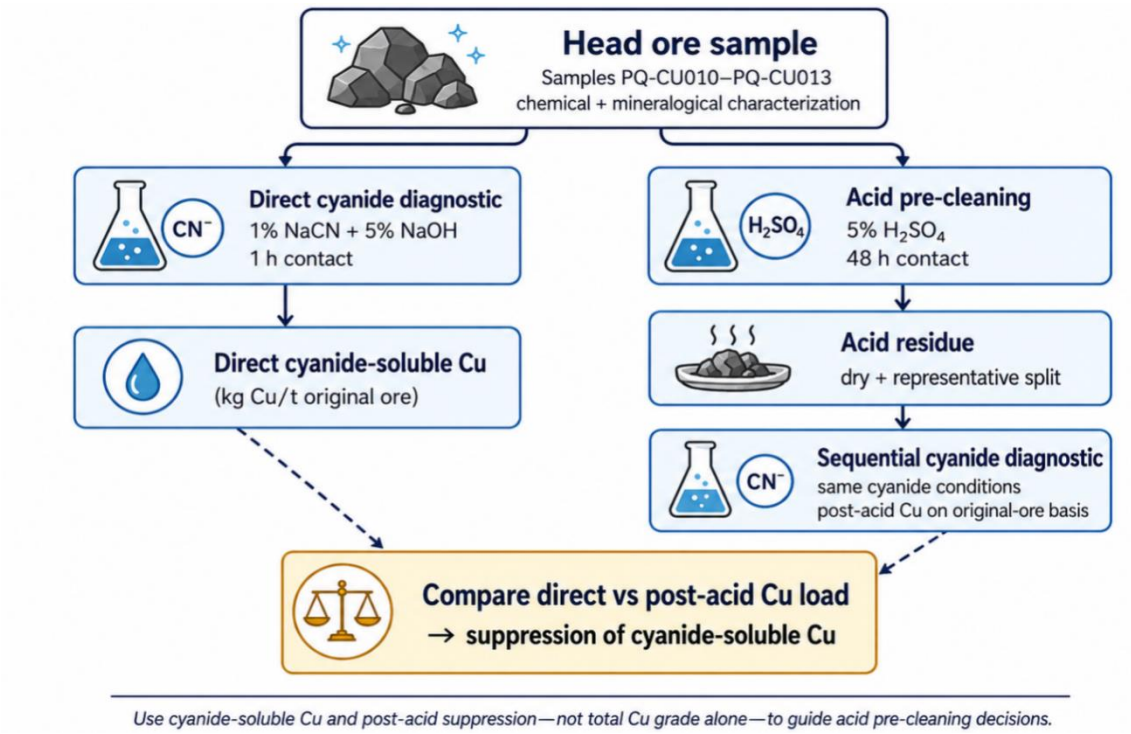

Figure 1. Sequential acid–cyanide diagnostic leaching workflow used for process-oriented copper classification.

Source: Authors' elaboration from the experimental procedure described in this study.

The paired direct and post-acid cyanide tests provide the key process comparison: acid-soluble Cu is a useful pretreatment target only to the extent that its removal materially lowers the subsequent cyanide-soluble Cu burden on an original-ore basis.

Analytical Quality Control

The archived dataset retained duplicate assay results and laboratory acceptance criteria, although complete instrument-specific analytical metadata were not preserved. Duplicate Cu analyses of the acid liquors showed dispersion of approximately 0.50–1.03%, below the documented laboratory acceptance criterion of 5%.

Duplicate analyses of the sequential cyanide liquors showed dispersion of approximately 1.46–8.24%, within the corresponding 10% criterion. Direct cyanide-test duplicates similarly showed dispersion of approximately 0.95–8.11%.

Consequently, the observed sample-to-sample differences are substantially greater than the documented duplicate variability and are metallurgically meaningful within the scope of this diagnostic dataset. This statement refers to

analytical repeatability and does not substitute for full method-validation metadata.

Data Treatment

Because only four mineralogical samples were available, statistical treatment was intentionally descriptive. Means, ranges, relative reductions, and exploratory Pearson correlations were calculated, but no population-level statistical inference was attempted. Correlation coefficients are therefore used only to describe trends within the four-sample dataset.

Results

Gold and Copper Head Grades

The triplicate head-Au determination performed on PQ-CU010 averaged 0.597 ± 0.055 g/t. Because PQ-CU010 was considered representative of the same ore population as PQ-CU011–PQ-CU013, this mean was used as the representative head-Au grade for screening comparisons, while recognizing that Au was not independently assayed in the other three samples. Head Cu concentrations ranged from 0.659 to 1.355 wt.%, equivalent to approximately 6.59–13.55 kg Cu/t ore.

Despite the absence of sample-specific Au assays for PQ-CU011–PQ-CU013, the large mass-concentration contrast between Cu and the representative Au grade justifies directly measuring Cu reactivity before cyanidation rather than relying on the bulk Cu grade alone.

Using the PQ-CU010 triplicate mean as representative of the same ore population for screening purposes, total Cu occurs at approximately 11,000–23,000 times the mass concentration of Au. Dissolution of even a small fraction of the Cu can therefore impose a substantial cyanide burden relative to the amount required for Au dissolution.

All four samples also exceed the approximately 0.5 wt.% Cu warning level discussed by Pereira and Barbosa (2017) for direct cyanidation of oxidized copper-bearing gold ores. This value is not treated here as a universal process cut-off; rather, it reinforces the need to quantify cyanide-soluble Cu directly.

Copper Mineralogical Deportment

Copper deportment was strongly dominated by association with biotite, which accounted for 76.50–93.84% of the total Cu.

Manganese oxides represented the second principal association, accounting for 5.31–23.50% of Cu. Copper oxy-hydroxides were minor and reached a maximum of only 3.16% in PQ-CU012.

The low sulfur concentration (<0.01 wt.%) indicates that highly reactive secondary copper sulfides are unlikely to be responsible for the observed cyanide demand. The results therefore provide an opportunity to examine cyanide reactivity in material dominated by phyllosilicate- and Mn-oxide-associated copper.

Automated mineralogical association does not demonstrate that all Cu assigned to biotite is structurally incorporated into the biotite lattice. Surface coatings, fine inclusions, adsorbed Cu, and intergrown phases may also influence automated classification. Sequential leaching responses provide complementary evidence of chemical accessibility.

The contrasting distribution of copper among the three principal mineralogical associations is shown in Figure 2.

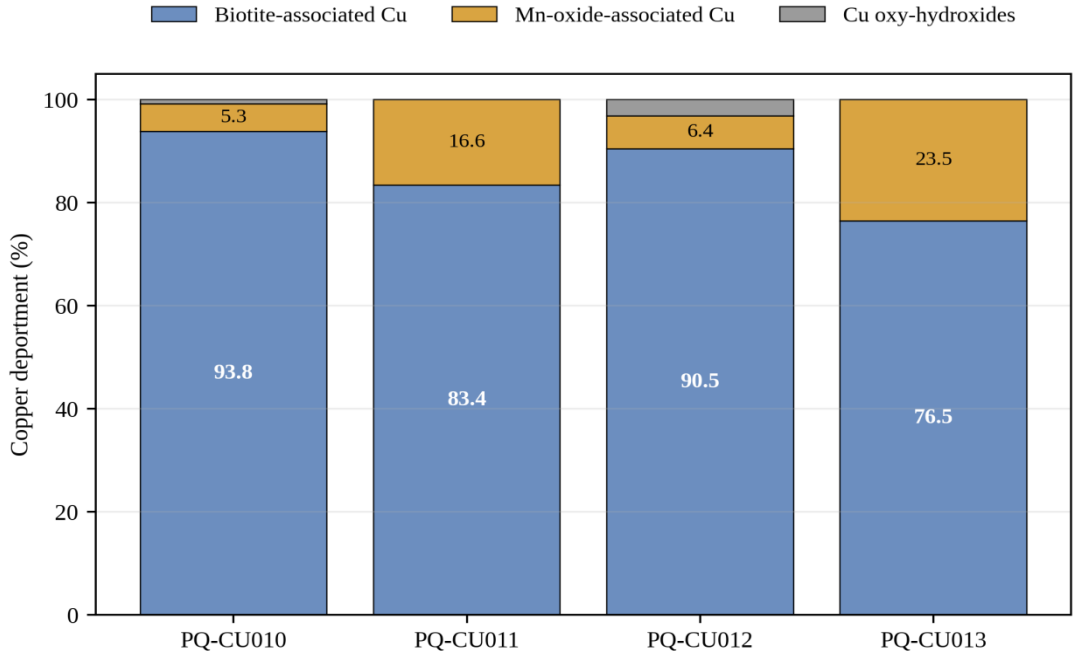

Figure 2. Copper deportment among biotite, manganese oxides, and copper oxy-hydroxides for the four investigated samples.

Source: Authors' elaboration from the automated-mineralogy data summarized in Table 1.

Figure 2 emphasizes the dominance of biotite-associated copper in all samples while also showing the comparatively high Mn-oxide-associated contribution in PQ-CU013. This mineralogical contrast is consistent with the markedly different acid and cyanide responses discussed below.

Acid-Soluble Copper

Sulfuric-acid treatment extracted between 40.54 and 78.93% of the total copper.

An important observation is that the absolute amount of Cu removed by acid was considerably less variable than the corresponding extraction percentages. Acid-soluble copper ranged only from approximately 5.18 to 6.31 kg/t despite a more than twofold relative range in percentage extraction.

Table 2. Copper extraction during sulfuric-acid pre-cleaning

Sample	Head Cu (kg/t)	Acid-soluble Cu (kg/t)	Acid Cu extraction (%)	Residueyield (%)
PQ-CU010	6.594	5.205	78.93	85.74
PQ-CU011	9.508	5.378	56.57	87.20
PQ-CU012	13.554	6.311	46.56	87.72
PQ-CU013	12.784	5.183	40.54	90.35

Source: Authors' calculations from original duplicate laboratory records.

Figure 3 compares the total copper inventory with the acid-soluble copper load and the corresponding extraction percentage for each sample.

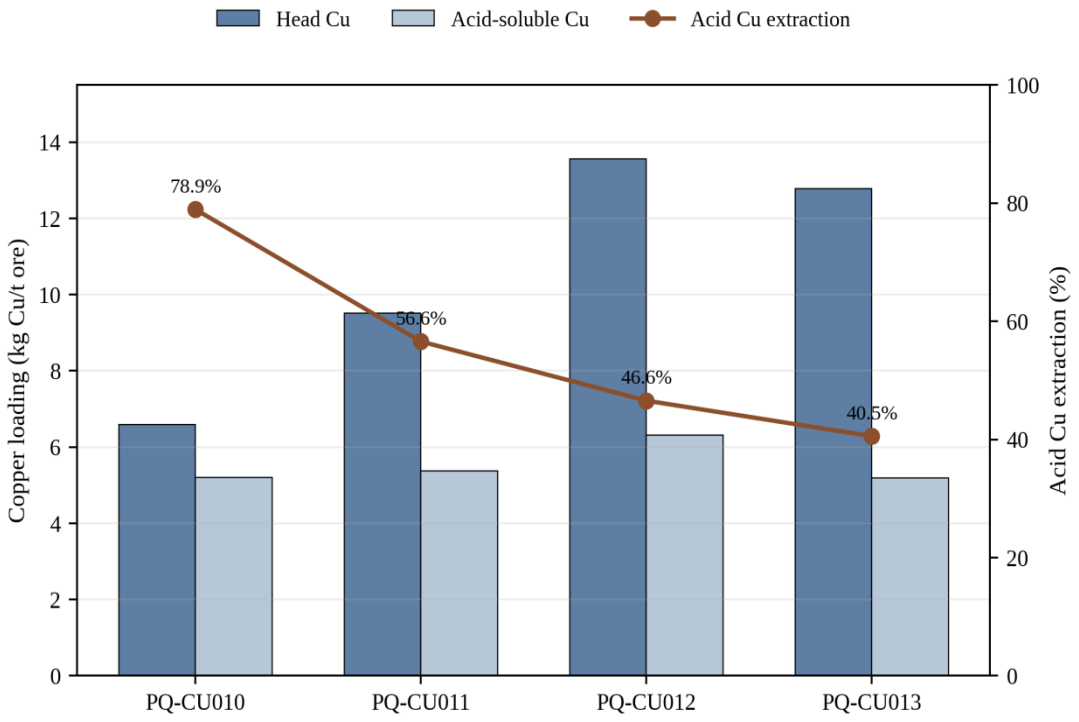

Figure 3. Head copper, acid-soluble copper, and sulfuric-acid copper extraction.

Source: Authors' elaboration from the experimental results reported in Table 2.

The figure makes clear that a broadly similar absolute mass of copper (about 5–6 kg/t) was removed from all four samples, but this represented very different fractions of the head copper inventory. PQ-CU010 therefore shows the highest proportional acid response despite having the lowest head Cu grade.

The data suggest the presence of a broadly comparable acid-labile Cu inventory of approximately 5–6 kg/t across the four samples, superimposed on a variable quantity of more acid-resistant copper.

PQ-CU010 was particularly responsive: despite having the lowest head Cu concentration, approximately 79% of its Cu was acid-soluble.

By contrast, PQ-CU013 had the highest Mn-oxide-associated contribution to Cu deportment and the lowest acid extraction, at 40.54%. This behavior is consistent with the lower cyanide accessibility of some Mn-oxide-associated Cu populations reported for comparable oxidized Cu–Au material (Pereira & Barbosa, 2017).

The apparent solid mass loss during acid treatment was approximately 9.6–14.3%. Because only Cu was analyzed in the acid liquor, it is not possible to determine how much of this mass loss resulted from dissolution of Fe-, Al-, Mg-, Mn-, or other gangue phases. This observation is important for process economics because high gangue dissolution would increase acid consumption and downstream neutralization requirements.

Direct Cyanide-Soluble Copper

The direct cyanide diagnostic test dissolved 0.673–1.179 kg Cu/t of original ore. When expressed relative to head Cu, 5.57–17.88% of total Cu was directly cyanide soluble.

Table 3. Direct and post-acid cyanide-soluble copper

Sample	Direct cyanide-soluble Cu (kg/t ore)	Direct cyanide-soluble fraction (% of head Cu)	Post-acid cyanide-soluble Cu (kg/t original ore)	Post-acid cyanide-soluble fraction (% of head Cu)	Reduction in cyanide-soluble Cu (%)
PQ-CU010	1.179	17.88	0.244	3.70	79.29
PQ-CU011	0.673	7.08	0.426	4.48	36.70
PQ-CU012	0.916	6.76	0.616	4.55	32.71
PQ-CU013	0.712	5.57	0.522	4.08	26.80
Mean	**0.870**	—	**0.452**	—	—

Source: Authors' calculations reconstructed from the original sample-labelled duplicate assays.

The results demonstrate that bulk Cu and cyanide-soluble Cu are not proportional across the four samples.

PQ-CU010 contains only 0.659 wt.% total Cu, the lowest grade among the four samples, yet has the greatest cyanide-soluble Cu load, at 1.179 kg/t. In contrast, PQ-CU013 contains 1.278 wt.% Cu but only 0.712 kg/t is directly cyanide soluble.

This finding is central to process selection. Bulk Cu grade alone would rank PQ-CU012 and PQ-CU013 as the largest Cu inventories, whereas the direct diagnostic test identifies PQ-CU010 as the greatest cyanide-soluble Cu burden per tonne of ore.

Effectiveness of Acid Pre-Cleaning

Acid pre-cleaning reduced the mean diagnostic cyanide-soluble Cu load from 0.870 to 0.452 kg/t original ore, corresponding to an approximately 48% decrease in the mean load.

The magnitude of the improvement was strongly sample dependent.

PQ-CU010 showed the clearest benefit, with cyanide-soluble Cu decreasing from 1.179 to only 0.244 kg/t, a 79.29% reduction.

PQ-CU011 showed a moderate reduction of 36.70%, while PQ-CU012 showed a 32.71% decrease.

PQ-CU013 showed the smallest improvement: 26.80% of its direct cyanide-soluble Cu burden was suppressed by the acid pretreatment.

The sample-specific effect of acid pre-cleaning on the cyanide-soluble copper load is compared directly in Figure 4.

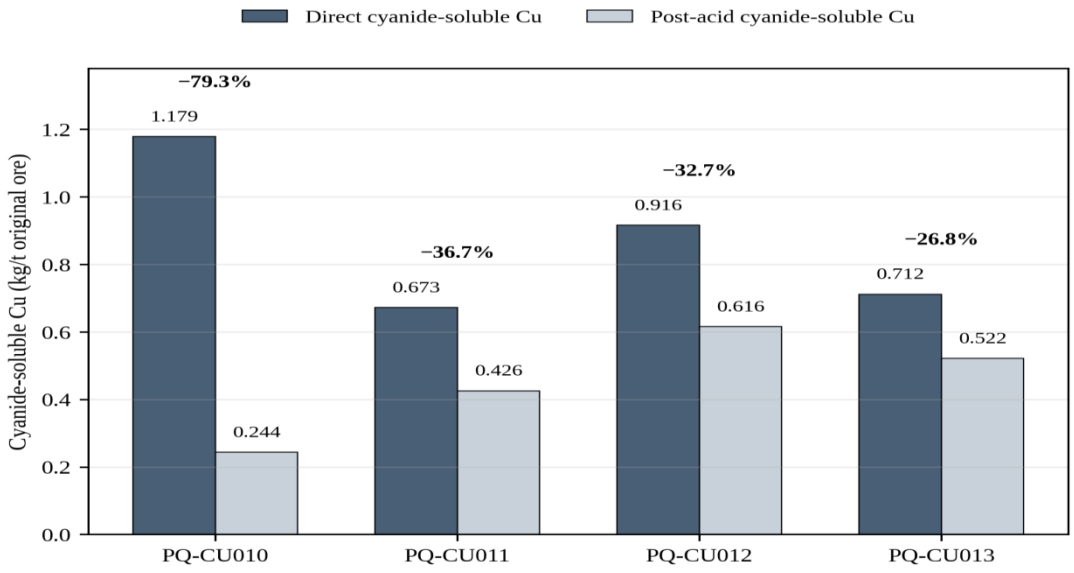

Figure 4. Direct and post-acid cyanide-soluble copper, normalized to one tonne of original ore.

Source: Authors' elaboration from the sequential diagnostic-leaching results summarized in Table 3.

PQ-CU010 is the clearest pretreatment-responsive sample: its cyanide-soluble Cu load falls from 1.179 to 0.244 kg/t original ore, whereas the reductions for PQ-CU011, PQ-CU012, and PQ-CU013 are smaller. The comparison confirms that total Cu grade alone cannot rank cyanidation risk.

A strong descriptive relationship was observed between acid-Cu extraction and the direct cyanide-soluble Cu fraction (Pearson $r \approx 0.95$, $n = 4$). Acid extraction also showed a strong descriptive association with the percentage suppression of subsequent cyanide-soluble Cu ($r \approx 0.97$, $n = 4$).

Because only four samples are available, these correlation coefficients are descriptive only and should not be interpreted as inferential statistical evidence. Within this limited dataset, they are consistent with the interpretation that the most acid-labile Cu populations are also among the most accessible to cyanide complexation.

Discussion

Operational Definition and Importance of Cyanide-Soluble Copper

For the present study, cyanide-soluble Cu is operationally defined as the amount of Cu dissolved during the standardized direct cyanide diagnostic test. It is used as a process-oriented proxy for the Cu fraction capable of imposing a cyanide burden under the test conditions, not as a direct measurement of plant NaCN consumption.

This definition avoids assuming that all head Cu has the same chemical reactivity and keeps the interpretation tied to an experimentally measured response.

Copper dissolved during cyanidation forms stable Cu(I)–cyanide complexes. Under alkaline conditions, $Cu(CN)_3^{2-}$ is commonly important, while $Cu(CN)_2^-$ and $Cu(CN)_4^{3-}$ can also contribute depending on free cyanide and solution chemistry (Kyle & Hefter, 2015).

Copper–cyanide complex formation reduces the free cyanide available for Au dissolution and can increase dissolved-Cu and WAD-cyanide loads requiring downstream treatment. Copper can also complicate activated-carbon and solution-recovery circuits (Dai et al., 2012; Sceresini, 2005).

The diagnostic result is particularly relevant because the directly soluble Cu concentration of 0.673–1.179 kg/t is extremely high relative to approximately 0.597 g/t Au.

Assuming the Au result is representative of the same ore population, approximately 1,100–2,000 times as much copper as gold, on a mass basis, can enter solution during the direct cyanide diagnostic test. This explains why apparently modest percentages of cyanide-soluble Cu can dominate cyanide chemistry.

Potential Cyanide Penalty if Acid Pre-Cleaning Is Omitted

For screening purposes, the Cu-related NaCN burden was estimated at 2.3 kg NaCN per kilogram of dissolved Cu. This factor is used only as an engineering normalization for copper–cyanide complexation; actual plant NaCN consumption depends on mineralogy, cyanide speciation, oxygen, pH, competing reactions, residence time, and solution recycle (Oraby & Eksteen, 2015, 2016).

Applying this factor to the measured direct and post-acid cyanide-soluble Cu values gives the comparative estimates shown in Table 4.

Table 4. Screening estimate of the cyanide burden associated with dissolved copper

Sample	Without acid pre-cleaning (kg NaCN/t original ore)	After acid pre-cleaning (kg NaCN/t original ore)	Potential reduction (kg NaCN/t original ore)
PQ-CU010	2.71	0.56	2.15
PQ-CU011	1.55	0.98	0.57
PQ-CU012	2.11	1.42	0.69
PQ-CU013	1.64	1.20	0.44
Mean	**2.00**	**1.04**	**0.96**

Source: Authors' calculations using the experimentally measured cyanide-soluble Cu and a literature screening factor of 2.3 kg NaCN per kg Cu dissolved.

The same contrast can be expressed as a screening estimate of the copper-related cyanide burden, as illustrated in Figure 5.

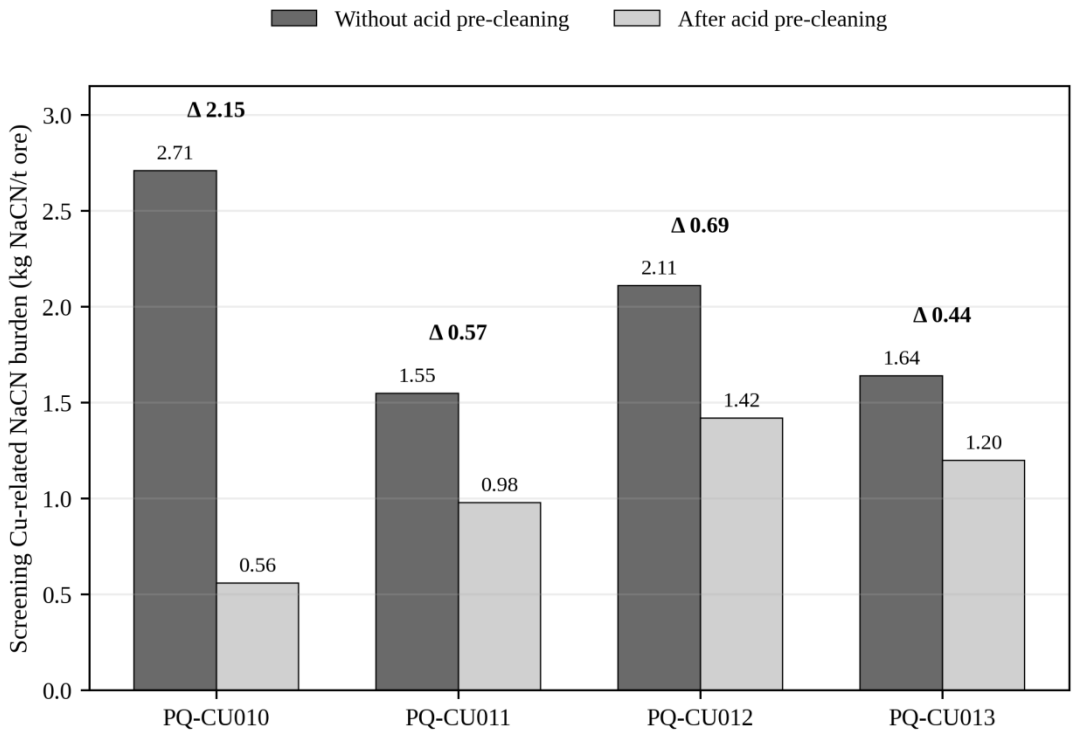

Figure 5. Screening estimate of Cu-related NaCN burden before and after acid pre-cleaning.

Source: Authors' elaboration from Table 4 using 2.3 kg NaCN per kilogram of Cu dissolved as a screening factor (Oraby& Eksteen, 2015, 2016).

The largest screening reduction is associated with PQ-CU010 (2.15 kg NaCN/t original ore), whereas PQ-CU013 shows only a 0.44 kg NaCN/t original-ore decrease. These values are comparative indicators rather than predicted plant reagent consumptions.

The diagnostic cyanide assay uses reagent concentrations and liquid-to-solid ratios that differ markedly from industrial cyanidation; therefore, the screening values must not be interpreted as predicted plant reagent consumption.

The calculation is useful only for comparing the relative magnitude of the Cu-related cyanide burden among samples and the potential effect of selectively removing cyanide-soluble Cu.

Under this interpretation, processing the ore without acid pre-cleaning could expose the cyanidation circuit to a Cu-related NaCN requirement equivalent to approximately 1.55–2.71 kg NaCN/t original ore, before accounting for all other cyanide-consuming reactions.

The particularly high value predicted for PQ-CU010 reinforces the importance of mineralogical reactivity over total Cu concentration.

Mineralogical Control of Acid and Cyanide Response

The predominance of biotite-associated Cu is a distinctive feature of the investigated samples, but the automated association classes must be interpreted together with the chemical-leaching response.

PQ-CU010 contains approximately 94% of its Cu in the biotite-associated class and simultaneously exhibits the highest acid extraction and the highest direct cyanide-soluble Cu fraction. This response indicates that a substantial part of the Cu assigned to that mineralogical population is chemically accessible under the diagnostic conditions.

Possible explanations include exchangeable or adsorbed Cu at phyllosilicate surfaces, fine secondary Cu-bearing coatings, or Cu-bearing microphases that are not completely differentiated during automated mineral classification. The present dataset does not resolve among these mechanisms.

By contrast, PQ-CU013 contains 23.5% of its Cu in the Mn-oxide-associated class, the largest such fraction among the four samples. It also shows the lowest acid-extraction percentage and the lowest proportional direct cyanide solubility.

This observation is consistent with Pereira and Barbosa (2017), who reported lower cyanide reactivity for some Mn-rich oxidized Cu populations than for more labile secondary or surface-associated Cu phases. The comparison supports a mineralogy-plus-reactivity interpretation rather than a bulk-assay interpretation.

The present results therefore indicate that the sequential leaching response cannot be interpreted from the total Cu

assay alone. Mineralogical deportment and chemical accessibility must be considered jointly.

Is Acid Pre-Cleaning Necessary?

From a metallurgical perspective, direct cyanidation of the ore population without prior assessment of Cu reactivity is not advisable. All four samples release measurable Cu during the 1 h cyanide diagnostic test, and the mean cyanide-soluble Cu load of 0.870 kg/t original ore represents a substantial competing Cu burden under the diagnostic conditions.

However, the results do not support treating all four samples or ore domains identically.

For PQ-CU010, acid pre-cleaning is strongly supported. Nearly 79% of head Cu is acid soluble and the treatment reduces cyanide-soluble Cu by approximately 79%. The estimated Cu-associated cyanide burden falls from approximately 2.71 to 0.56 kg NaCN/t original ore.

For PQ-CU011 and PQ-CU012, pre-cleaning provides intermediate suppression, reducing the cyanide-soluble Cu load by approximately 37% and 33%, respectively.

PQ-CU013 presents a different situation. Although the acid stage removes 5.18 kg Cu/t ore, the cyanide-soluble Cu burden decreases by only approximately 0.19 kg Cu/t, or 26.8%. The corresponding Cu-related NaCN screening reduction is about 0.44 kg NaCN/t original ore. For this sample, acid cleaning solely to reduce the cyanide-soluble Cu burden may be less attractive and should be evaluated against acid consumption, washing, neutralization, residue handling, and effluent-management requirements.

The process conclusion is therefore not that acid cleaning is universally required. Acid pre-cleaning is strongly supported for ore domains containing highly acid-labile and cyanide-soluble Cu, whereas its application to lower-reactivity domains should be evaluated through paired metallurgical testing, economic analysis, selective treatment, or blending.

Comparison with Previous Cu-Mitigation Studies

Pereira and Barbosa (2017) reported that sulfuric-acid pre-cleaning can substantially suppress subsequent Cu dissolution in cyanide when the ore contains a sufficiently acid-labile Cu population. The present results reproduce that central mechanistic trend, but they also show that suppression can be incomplete and strongly sample-specific.

Other Cu-mitigation studies reinforce the broader principle that reactive Cu should be removed, complexed, or otherwise controlled before or during cyanidation. Ammonia pretreatment has been used to alleviate Cu interference in copper-rich gold ore (Bas et al., 2015), acidic pretreatment has reduced Cu-related cyanide interference in a Cu–Ag system (Portilla et al., 2020), and EDTA-based pretreatment has been investigated for selective removal of soluble Cu before gold cyanidation (Kuzu et al., 2026). Alternative sulfate–chloride treatment schemes have likewise been examined for oxidized Au–Cu ores (Sekisov et al., 2018).

These literature examples involve different ore types, reagents, and operating conditions and are therefore used here as process analogs rather than direct quantitative benchmarks. Their common implication is that the value of pretreatment is governed by the identity and reactivity of the Cu removed, not simply by the total Cu extracted.

After acid pre-cleaning, the present samples still contain 0.244–0.616 kg/t original ore of Cu that is soluble in the diagnostic cyanide solution. Acid pre-cleaning therefore reduces, but does not eliminate, the copper–cyanide interaction in this sample set. This residual burden supports the use of ore-specific sequential diagnostic testing rather than direct transfer of a pretreatment recipe from one deposit or sample population to another.

Implications for Gold Recovery

No paired gold-extraction data from cyanidation before and after acid pre-cleaning were included in the experimental dataset analyzed in this study.

Consequently, the present results do not provide experimental evidence that acid pre-cleaning increases Au recovery in these four samples.

Nevertheless, the measured reduction in cyanide-soluble Cu provides a mechanistic basis for expecting improved control of free cyanide during subsequent Au leaching. Previous work has shown that Cu dissolution can reduce available free cyanide and complicate Au extraction unless reagent concentration or other process conditions are adjusted (Breuer et al., 2005; Deschênes & Prud'homme, 1997; Maganga et al., 2023; Pereira & Barbosa, 2017).

The next experimental stage should directly measure Au extraction kinetics and actual NaCN consumption under otherwise comparable cyanidation conditions, with and without acid pre-cleaning.

Such paired testing is essential because a reduction in diagnostic Cu burden alone does not establish the economic value of pretreatment. The decision criterion must combine Au recovery, reagent consumption, dissolved Cu, carbon loading, solution treatment, acid consumption, residue washing, neutralization demand, and overall operating cost.

Industrial Implications and Limitations

The sulfuric-acid procedure was designed as a diagnostic laboratory test rather than an optimized industrial leaching operation.

The 48 h contact time, nominal 5% H_2SO_4 solution, and high liquid-to-solid ratio should not be transferred directly to an industrial flowsheet. In addition, the archived records do not preserve the concentration basis of the nominal reagent percentages, test temperature/agitation, or complete instrument-specific analytical metadata. These parameters

should be fully documented in any confirmatory experimental campaign.

The apparent solid mass loss of approximately 9.6–14.3% demonstrates that Cu was not the only component affected by the acid stage. Quantification of Fe, Al, Mg, Mn, Ca, and other dissolved elements is required to assess acid selectivity, gangue dissolution, and downstream neutralization demand.

Actual sulfuric-acid consumption was not recorded in a form that permits a reliable kg H_2SO_4/t ore calculation. Consequently, an economic comparison between acid consumption and possible cyanide savings cannot yet be performed.

The 9.23% RSD observed in the PQ-CU010 Au triplicate is also relevant. At the measured PQ-CU010 grade of approximately 0.60 g/t, which was adopted as representative of the investigated ore population, additional replicate Au determinations, appropriate certified reference materials, and sample-specific Au assays for PQ-CU011–PQ-CU013 would improve confidence in subsequent Au mass balances.

Any transition from acid leaching to cyanidation requires adequate solid washing and restoration of strongly alkaline conditions before cyanide contact. This is both a metallurgical requirement and a process-safety requirement.

Process-Selection Implications

The results suggest that flowsheet selection should be controlled by reactive-Cu characterization rather than by a single bulk-Cu cut-off.

A practical characterization program would combine head Cu, automated mineralogical Cu deportment, acid-soluble Cu, direct cyanide-soluble Cu, and post-acid cyanide-soluble Cu normalized to the original-ore basis.

The direct cyanide test identifies the immediate diagnostic Cu burden, whereas the sequential test quantifies how much of that burden is actually suppressed by acid pretreatment.

The difference between direct and post-acid cyanide-soluble Cu is therefore more useful for deciding whether to introduce acid pre-cleaning than the absolute amount of Cu dissolved by acid alone.

$$\Delta Q_{Cu,CN} = Q_{Cu,CN,direct} - Q_{Cu,CN,post}$$

This distinction is illustrated by PQ-CU013. Acid leaching removes more than 5 kg Cu/t ore, yet the cyanide-soluble Cu burden decreases by only approximately 0.19 kg/t. Conversely, in PQ-CU010, removal of a similar absolute quantity of Cu by acid decreases the cyanide-soluble Cu load by approximately 0.94 kg/t.

Thus, the value of acid pre-cleaning is governed by the reactivity of the Cu removed, not simply by the quantity of Cu extracted in the acid stage.

Conclusions

Sequential sulfuric-acid and cyanide diagnostic leaching differentiated bulk Cu from the fraction of Cu that is directly soluble under the standardized cyanide diagnostic conditions.

The four samples contained 0.659–1.355 wt.% Cu. A triplicate head-Au determination on PQ-CU010 averaged 0.597 ± 0.055 g/t Au and was adopted as the representative head-Au value for the investigated ore population; Au was not independently determined in PQ-CU011–PQ-CU013. Direct cyanide-soluble Cu ranged from 0.673 to 1.179 kg/t original ore, equivalent to 5.57–17.88% of head Cu.

Sulfuric-acid pre-cleaning removed 40.54–78.93% of head Cu, corresponding to a comparatively narrow absolute range of 5.18–6.31 kg Cu/t ore. Subsequent cyanide-soluble Cu decreased to 0.244–0.616 kg/t on an original-ore basis.

Pretreatment effectiveness was strongly sample dependent. Suppression of cyanide-soluble Cu ranged from 26.8% in PQ-CU013 to 79.3% in PQ-CU010.

Mineralogical data indicate that this variability is associated with Cu deportment and chemical accessibility. Biotite-associated Cu dominated all samples, while the sample with the largest Mn-oxide-associated Cu fraction showed comparatively lower acid and cyanide reactivity. These automated associations are not interpreted as proof of structural Cu hosting.

Bulk Cu concentration alone is therefore insufficient to determine whether a gold-bearing oxidized Cu ore requires acid pre-cleaning. Direct and sequential cyanide-soluble Cu measurements provide a more process-relevant basis for selecting samples or ore domains.

For the investigated sample population, uncontrolled Cu dissolution would impose a substantial competing Cu burden under diagnostic cyanide conditions. Acid pre-cleaning is strongly supported for the most reactive sample and may be beneficial for selected ore domains, but implementation should be based on paired Au recovery and actual reagent consumption tests rather than on the diagnostic Cu response alone.

The present work establishes the metallurgical value of controlling cyanide-soluble Cu but does not establish the economic optimum for acid pretreatment. The next stage should combine optimized acid-cleaning conditions with paired Au-cyanidation tests measuring recovery kinetics, actual NaCN consumption, acid consumption, dissolved Cu, gangue dissolution, washing/neutralization demand, and downstream solution chemistry.

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Data Availability Statement

The data analyzed in this study were derived from existing laboratory analytical and operating records. The reconciled values supporting the calculations are reported in the article's tables and figures. No missing experimental values were generated or imputed.

Competing Interests

The author declares no known competing financial interests or personal relationships that could have influenced the work reported in this article.

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