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# Investigation of the deportment of gold and silver in a refractory sulfide ore by diagnostic leaching

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**Abstract**: The refractory behavior of a sulfide-zone copper-rich gold ore from the Mastra deposit (Gümüşhane, Türkiye) was investigated by diagnostic leaching to quantify the deportment of gold and silver and to identify the controls on cyanidation performance. Gold extraction of only 18.4% was obtained by direct cyanidation of the as-received ore, confirming its highly refractory behavior. Sequential pre-treatments with HCl and $HNO_3$, each followed by cyanidation, increased the cumulative Au extraction to 88.5%. The largest increment, 47.6% of the total Au, was obtained after the $HNO_3$ stage, indicating that this fraction is associated with a sulfide-bearing matrix. The remaining 11.5% of the Au is reported as an unresolved residual refractory fraction. Silver behaved differently from gold in that 52.9% of the Ag was dissolved directly by the HCl and $HNO_3$ stages, mainly as chloride species in the HCl stage, and cyanide extracted a total of 38.0%. The high cyanide consumption in direct cyanidation (6.75 kg/Mg) is largely attributed to the dissolved copper (22.7% of the Cu). The study demonstrates that diagnostic leaching provides a quantitative basis for identifying the causes of gold refractoriness and for selecting appropriate pre-treatment strategies for refractory gold ores that are not amenable to conventional cyanidation.

***Keywords:*** refractory gold, cyanidation, diagnostic leaching, acid treatment, extraction methods

## 1. Introduction

Gold (Au) ores exhibit varying degrees of amenability to conventional cyanidation and are therefore classified as either refractory or free-milling (La Brooy et al., 1994; Marsden and House, 2006; Celep et al., 2019; Msumange, 2019; Msumange et al., 2020). Free-milling ores usually attain Au recoveries above 90% through cyanidation. Conversely, refractory ores typically yield recoveries below 80% (La Brooy et al., 1994). Refractory behavior is primarily caused by the presence of carbonaceous matter with preg-robbing properties, sulfide/silicate encapsulation, and phases that consume large amounts of reagents such as cyanide and oxygen, including reactive copper minerals and pyrrhotite (Celep and Serbest, 2015; Msumange et al., 2021; Msumange et al., 2022; Msumange et al., 2023; Navas Jaramillo and Gonzalez Zuñiga, 2025; Shafiei et al., 2025). The considerable challenges of Au recovery from refractory ores, including telluride, carbonaceous, and sulfide-encapsulated gold ores, require sophisticated analytical techniques. Consequently, understanding the occurrence and nature of refractory gold ores is critical for optimizing Au extraction (Quaicoe et al., 2023; Li et al., 2023). Diagnostic leaching, originally developed by Anglo American Research Laboratories (AARL) in the mid-1980s, is a well-established analytical tool for determining the deportment of Au in ores (Lorenzen, 1995; Lorenzen and Tumilty, 1992; Tumilty et al., 1987). The technique involves the sequential, selective oxidative leaching/destruction of the least stable minerals associated with Au, followed by the extraction of liberated Au via cyanidation at each stage (Celep et al., 2008; Lorenzen, 1995; Tumilty et al., 1987). Although diagnostic leaching is a powerful analytical technique, it possesses certain drawbacks. One

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critical concern is that the pre-treatment leach stages in diagnostic leaching can dissolve multiple minerals, reducing selectivity for any single mineral (Henley et al., 2001). Furthermore, the destruction of the target minerals may not be entirely complete (Lorenzen, 1995).

Table 1 presents the pre-treatment stages and the minerals expected to dissolve during diagnostic leaching. The leaching sequence may be customized based on the sample's specific mineralogical characteristics, allowing the omission of certain steps in accordance with established guidelines (Celep et al., 2008; Celep et al., 2009; Lorenzen, 1995; Guner et al., 2023). Future development of diagnostic leaching depends on further refinement of the technique to improve selectivity and broaden its applicability to a wider range of ore types. Progress in automated mineralogy and analytical techniques will be vital for increasing the accuracy and efficiency of diagnostic leaching (Guner et al., 2023). Furthermore, integrating diagnostic leaching with other advanced ore characterization techniques can result in a more comprehensive understanding of ore mineralogy, thereby contributing to the development of more efficient Au recovery approaches. The dissolution of various mineral phases during different acid leaching stages is described by the reactions presented below (Lorenzen and Tumilty, 1992):

$$CaCO_3 + 2HCl \rightarrow CaCl_2 + H_2O + CO_2 \quad (1)$$

$$MS + H_2SO_4 \rightarrow MSO_4 + H_2S \text{ [M: Metals like Cu, Zn, Pb etc.]} \quad (2)$$

$$2FeS_2 + 10HNO_3 \rightarrow 2Fe^{3+} + 2H^+ + 4SO_4^{2-} + 10NO + 4H_2O \quad (3)$$

$$SiO_2 + 6HF \rightarrow H_2SiF_6 + 2H_2O \quad (4)$$

Table 1. Selective pre-treatment leach stages and the target minerals for dissolution (Lorenzen and Van Deventer, 1993, Lorenzen, 1995, Henley et al., 2001)

| | Pre-treatment stages | Mineral phases undergoing dissolution |
|---|---|---|
| 1 | NaCN washes | Precipitated gold |
| 2 | NaCN | Gold |
| 3 | $Na_2CO_3$ | Gypsum and arsenates |
| 4 | HCl | Pyrrhotite, galena, calcite, goethite, dolomite, calcium carbonate |
| 5 | $HCl/SnCl_2$ | Calcite, ferrites, hematite |
| 6 | $H_2SO_4$ | Uraninite, sphalerite, labile pyrite, labile copper sulfides, labile base metal sulfides |
| 7 | $FeCl_3$ | Sphalerite, tetrahedrite, sulfide concentrates, labile sulfides, galena |
| 8 | $HNO_3$ | Pyrite, arsenopyrite, marcasite |
| 9 | Oxalic acid washes | Oxide coatings |
| 10 | HF | Silicates |
| 11 | Acetonitrile elution | Gold adsorbed on carbon, coal, kerogen |

Diagnostic leaching has been applied to a wide range of refractory ores. Direct cyanidation of the ores studied by Lorenzen and Van Deventer (1993) and Celep et al. (2008) extracted less than 2% and 72% of the Au, respectively, and sequential acid pre-treatments raised the extraction to more than 95% and 100%. Teague et al. (1998) used diagnostic leaching to show that Au locked in pyrite and pyrrhotite controlled ore flotation recovery, and Henley et al. (2001) quantified the partitioning of Au between native gold and tellurides. Celep et al. (2019) and Guner et al. (2023) combined diagnostic leaching with automated mineralogy and showed that the two techniques together identify the mineralogical controls on Au recovery more reliably than either technique alone. In the study by Guner et al. (2023), the largest increase in Au extraction was obtained after the $HNO_3$ stage, which decomposes pyrite and arsenopyrite. Javanshir et al. (2025) used diagnostic leaching results from a refractory sulfide concentrate to select pre-treatments (ultrafine grinding, roasting, pressure oxidation, and ammoniacal conditioning) for subsequent thiosulfate leaching. However, interpreting the stages is operational, since each acid attacks several minerals and complementary mineralogical data are needed to identify the host phases (Lorenzen, 1995; Henley et al., 2001).

Earlier work on the ore from the sulfide zone of the Mastra deposit showed that it is highly refractory. Cyanidation at elevated NaCN concentrations (1-8 g/dm$^3$), physical pre-treatment (ultra-fine grinding to $d_{80}$: 8-73 µm), and acid pre-treatment (1 mol/dm$^3$ $H_2SO_4$) failed to enhance gold extraction to acceptable standards during cyanide leaching, with both methods resulting in less than 80% Au recovery. However, roasting at 650 °C increased Au extraction in cyanide leaching to 73.4% at 1.5 g/dm$^3$ NaCN and to 96% at 5 g/dm$^3$ NaCN (Msumange, 2019; Msumange et al., 2020 and 2023). These studies did not, however, identify which fractions of Au, Ag and Cu become accessible after which treatment, nor what drives the high cyanide consumption of the ore. The ore characteristics and poor direct-cyanidation response of the Mastra sulfide-zone ore were reported previously (Msumange, 2019; Msumange et al., 2020). The present study extends previous work by quantifying the deportment of Au and Ag with co-deportment of Cu, Fe and Zn through each diagnostic stage, and by using this deportment to explain the response of the ore to previously tested treatments, namely the limited effect of ultrafine grinding and $H_2SO_4$ pre-treatment and the improvement obtained by roasting (Msumange et al., 2023).

## 2. Materials and methods

### 2.1. Materials

The sample was taken from one of the copper-rich sulfide zones of the Mastra gold deposit (Gümüşhane, Türkiye). The sample represents the Cu-bearing zone rather than the plant feed, and the results apply to this specific, high-grade sample (107.7 g/Mg Au, 9.3 g/Mg Ag and 1.6% Cu). The ore is mainly composed of quartz and pyrite (Fig. 1). Trace-element and whole-rock compositions were analyzed by inductively coupled plasma mass spectrometry (ICP-MS) and X-ray fluorescence (XRF), respectively, and the Au content was determined by fire assay at an accredited laboratory (ARGETEST, Ankara, Türkiye). The ore sample was initially crushed to -4 mm and subsequently milled in a laboratory rod mill for 30 min, producing a final particle size of -73 µm ($d_{80}$). The fine-ground ore was subdivided using a riffle splitter and a rotary sample divider (Fritsch Laborette 27) to obtain subsamples for characterization and leaching tests. Four independent feed splits were digested in hot aqua regia and analyzed by AAS, yielding 98.5 g/Mg Au (relative standard deviation, RSD, 4.2%). Table 2 presents the ore's chemical composition.

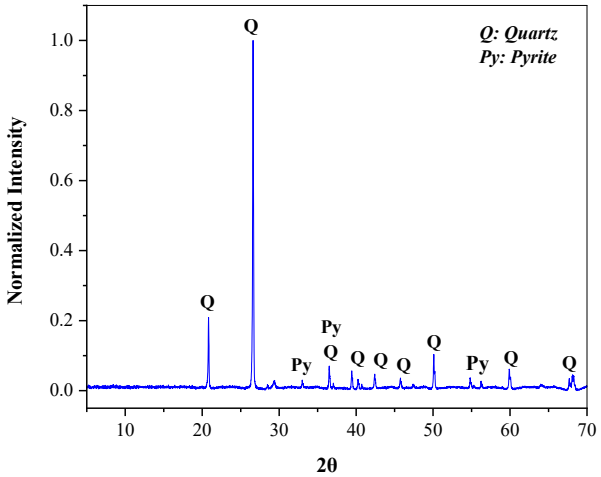



Fig. 1. XRD profile of the ore sample (Msumange et al., 2020)

### 2.2. Methods

#### 2.2.1. Cyanide leaching tests

Pyrex beakers (1000 cm$^3$) served as reaction vessels during cyanide leaching tests conducted over a 24 hour duration**.** The beakers were agitated at a constant stirring speed of 650 rpm using mechanical stirrers fitted with four-blade, PTFE-coated stainless-steel impellers with a 45° pitch and a 6.4 cm blade

Table 2. Chemical composition of the ore sample (Msumange et al., 2020)

| Element/Compound | Content (%) | Element | Content (g/Mg) |
|---|---|---|---|
| $SiO_2$ | 67.3 | Au | 107.7 |
| $Al_2O_3$ | 11.51 | Cu | 15960 |
| $Fe_2O_3$ | 6.06 | Al | 3900 |
| MgO | 0.45 | Ni | 604.5 |
| CaO | 0.46 | Zn | 926.1 |
| $Na_2O$ | 0.06 | Pb | 838.1 |
| $K_2O$ | 2.58 | As | 225 |
| $TiO_2$ | 0.02 | Ba | 1211 |
| $P_2O_5$ | 0.19 | Ag | 9.3 |
| MnO | 0.18 | Sb | 93.21 |
| $Cr_2O_3$ | 0.015 | Mo | 24.64 |
| Fe | 4.02 | Cd | 10.7 |
| S | 3.46 | La | 1.64 |
| K | 0.21 | Na | 270 |
| Ca | 0.22 | Mg | 710 |
| P | <0.001 | Ti | 23 |
| LOI | 6.03 | Ga | <5 |
| Total C | 0.15 | Bi | <5 |
| Total S | 4.56 | Hg | <5 |
| **Sum** | 95.31 | Be | <1 |

S: determined by XRF. Total C and total S: determined by combustion analysis (LECO).

diameter. Fig. 2 illustrates the experimental set-up. Leach solutions were prepared with sodium cyanide (≥95% NaCN; Merck) at 1.5 g/dm$^3$ NaCN. In the cyanide leaching tests, a pulp density of 25% (w/w) was maintained. An air pump supplied continuous aeration at 1.5 dm$^3$/min. At predetermined intervals, samples were withdrawn and centrifuged to obtain clear supernatants for the determination of Cu, Au, Ag, Fe, and Zn concentrations by atomic absorption spectrometry (AAS). The pH of the leach solutions was continuously monitored and maintained between 10.5 and 11.0 by adding lime as needed to correct deviations. Free cyanide concentration in the tests was monitored by titration with 0.02 M silver nitrate ($AgNO_3$) using *p*-dimethylaminobenzylidene rhodanine as the indicator, allowing determination of cyanide consumption. A constant NaCN concentration of 1.5 g/dm$^3$ was maintained by adding NaCN from a 50 g/dm$^3$ stock solution whenever the free cyanide level dropped below this threshold. The volumes of the samples withdrawn, evaporation losses, and the make-up additions were taken into account in the calculation of the metal extractions. It is worth noting that the $AgNO_3$ titration approach has limitations, as it may overestimate the free cyanide concentration due to partial release of cyanide from copper-cyanide complexes present in solution (Jiménez-Velasco et al., 2014). After 24 h of leaching, the residues were filtered, oven-dried at 105 °C, and then digested in hot aqua regia before metal analysis. Metal extractions were calculated from the metal in the final leach solution and in the solid residue.

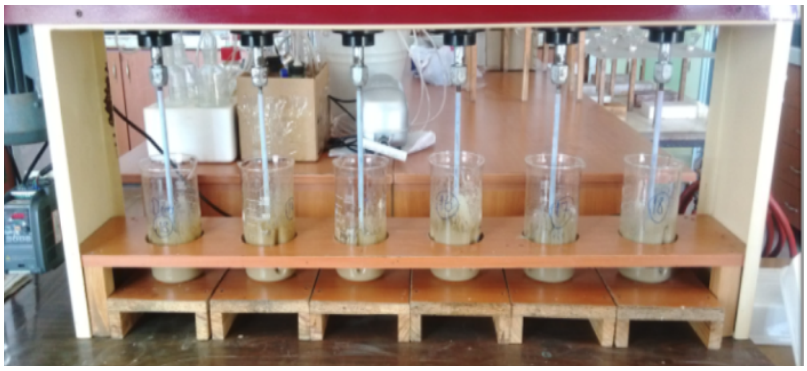


Fig. 2. Multi-mechanical stirrer set-up used in leaching tests

### 2.2.2. Diagnostic leaching tests

Diagnostic leaching was performed as a sequence of acid-leaching stages, each followed by cyanidation of the residue (Table 3). The sequence started with cyanide leaching of 200 g of ore. After each stage, the residue was filtered and washed thoroughly, first with a dilute solution of the reagent used in that stage and finally with distilled water to remove residual acid (or cyanide). The residue was then dried and weighed, a subsample (about 10 g) was taken for analysis, and the remaining solid was fed to the next stage. The wash volumes were recorded for each stage. In each stage, the pulp was prepared from the dry mass of the solid fed, at 25% w/w solids for cyanidation and at a solid-to-liquid ratio of 1:2 for the acid stages. The HCl (12% v/v, ≈1.45 mol/dm$^3$) and $HNO_3$ (33% v/v, ≈4.8 mol/dm$^3$) solutions were prepared from 37% HCl and 65% $HNO_3$ (Merck). Au, Ag, Cu, Fe, and Zn were determined in all leach solutions, wash solutions, and solid residues. The solid residues and the replicate feed samples were digested in hot aqua regia, and all solutions were analyzed by AAS. The AAS calibration solutions were prepared from 1000 mg/dm$^3$ standard metal solutions (in $HNO_3$), and a check standard and a reagent blank were analyzed with each batch of samples. Each solution was read three times by the instrument; the three individual readings were recorded, and their mean and RSD were calculated automatically by the instrument. The extraction in each stage was calculated as the metal in the final leach solution divided by the sum of the metal in the solution and in the solid residue, i.e., relative to the feed grade back-calculated in each test, and is reported as a percentage of the total metal in the feed unless stated otherwise.

Table 3. Conditions of diagnostic leaching tests

| Treatment step | Lixiviant | Concentration | Leaching conditions | Minerals likely to be decomposed |
|---|---|---|---|---|
| Cyanide leaching | NaCN | 1.5 g/dm$^3$ | Time: 24 h, pH 10.5, pulp ratio: 25% w/w | Gold |
| Acid leaching | HCl | 12% (v/v), ≈1.45 mol/dm$^3$ | Time: 8 h, S/L=1:2, Temperature: 60 °C | Calcite, Pyrrhotite, Hematite, Galena, Dolomite |
| Cyanide leaching | NaCN | 1.5 g/dm$^3$ | Time: 24 h, pH 10.5, pulp ratio: 25% w/w | Gold |
| Acid leaching | $HNO_3$ | 33% (v/v), ≈4.8 mol/dm$^3$ | Time: 6 h, S/L=1:2, Temperature: 60 °C | Arsenopyrite, pyrite, marcasite |
| Cyanide leaching | NaCN | 1.5 g/dm$^3$ | Time: 24 h, pH 10.5, pulp ratio: 25% w/w | Gold |

## 3. Results and discussion

### 3.1. Direct cyanide leaching

Conventional cyanide leaching of the ore using 1.5 g/dm$^3$ NaCN, performed without any prior pre-treatment such as acid leaching or roasting, resulted in relatively low extractions of Au, Ag, and Cu, achieving recoveries of only 18.4%, 26.9%, and 22.7%, respectively, after 24 h of leaching (Fig. 3a). These limited metal recoveries provide strong evidence of the refractory nature of the ore, indicating that the precious metals are not readily accessible to cyanide during direct leaching of the ore. Despite limited precious metal extraction, the cyanide consumption of the added NaCN reached 6.75 kg/Mg (Section 2.2), highlighting a substantial reagent consumption. The combination of low gold and silver recovery and high cyanide consumption suggests that a significant proportion of the cyanide was consumed through side reactions with copper-bearing minerals and other cyanide-reactive constituents present in the ore. The dissolution of copper, as reflected by the 22.7% Cu leaching, likely promoted the formation of Cu-CN complexes, thereby reducing the concentration of free cyanide available for gold and silver leaching. As depicted in Fig. 3b, the low selectivity ratios (Au/Cu and Ag/Cu) observed during the initial stages of leaching indicate preferential dissolution of copper-bearing minerals. Consequently, the efficiency of cyanide utilization decreased, contributing to both poor gold extraction and elevated

reagent consumption. The low gold and silver extractions, along with high cyanide consumption, indicate that pre-treatment prior to cyanide leaching is necessary to improve precious metal extractions and mitigate cyanide consumption.

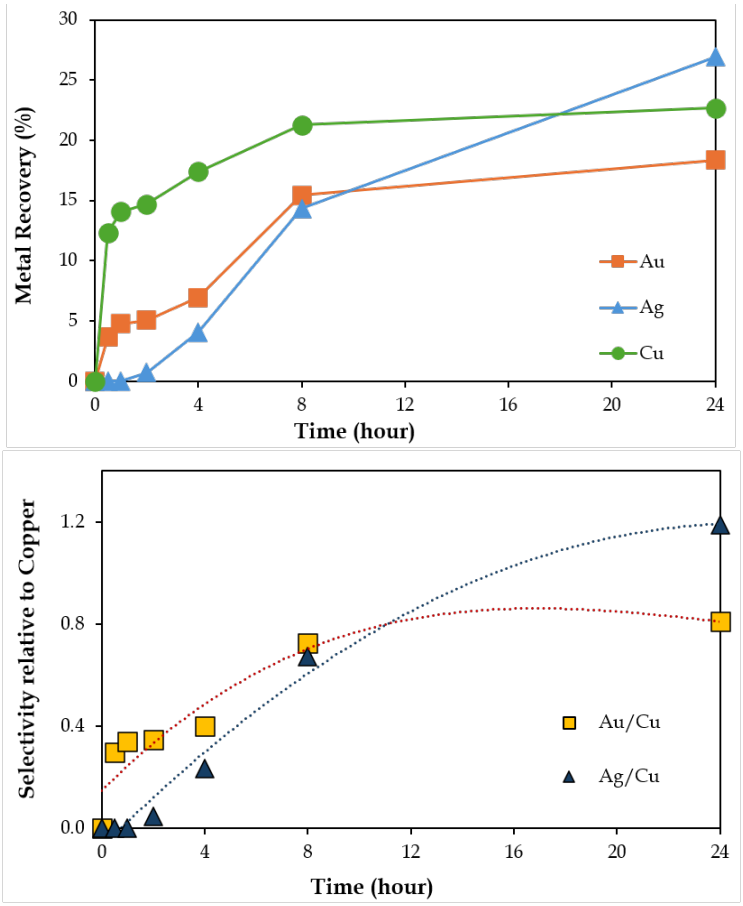



Fig. 3. (a) Extraction of Au and Ag in direct cyanide leaching tests (1.5 g/dm³ NaCN, pH 10.5-11, 25% w/w solids ratio, air flow rate: 1.5 dm³/min) (b) Evolution of Au/Cu and Ag/Cu recovery ratios during direct cyanidation.

To assess the cyanide demand of the dissolved Cu, the distribution of cyanide among its species was calculated with the SPANA software (SPANA, 2025) for the Cu concentration of the 24 h leach solution (1215 mg/dm³, 19.1 mmol/dm³) at 25 °C and pH 11 (Fig. 4). Free $CN^-$ appears only above a total cyanide concentration of about 57 mmol/dm³. Below this level, all cyanide is bound to Cu as CuCN(s) and $Cu(CN)_2^-$ at low concentrations, and mainly as $Cu(CN)_3^{2-}$ above about 45 mmol/dm³. At 82 mmol/dm³ of cyanide, about 80% of the cyanide is bound to Cu as $Cu(CN)_3^{2-}$ and $Cu(CN)_4^{3-}$, and the free $CN^-$ is only about 16 mmol/dm³ (about 0.8 g/dm³ NaCN). The amount of Cu dissolved in the first cyanidation stage would bind 5.56 kg to 8.34 kg NaCN/t as $Cu(CN)_2^-$ or $Cu(CN)_3^{2-}$, respectively. The measured consumption of 6.75 kg/Mg falls within this range, indicating that dissolved copper alone accounts for most of the cyanide consumed. The calculated free cyanide during cyanide leaching is lower than the 1.5 g/dm³ NaCN indicated by $AgNO_3$ titration, in line with the overestimation of free cyanide by this method in Cu-bearing solutions (Jiménez-Velasco et al., 2014). The measured consumption is therefore likely to be underestimated. Other consumers, such as the formation of thiocyanate and cyanate and the loss of HCN under aeration, were not quantified.

### 3.2. Diagnostic leaching

Fig. 5a depicts the outcomes of the diagnostic leaching test, in which standard cyanidation was systematically applied after each successive acid treatment step. The initial cyanide leaching of the as-received ore yielded an 18.4% Au recovery (Fig. 3a), indicating the portion of Au that is readily extractable through direct cyanidation. The remaining 81.6% of the Au can therefore be considered refractory

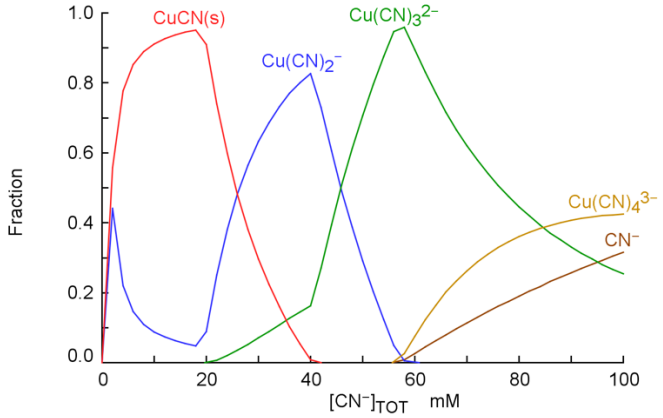



Fig. 4. Distribution of cyanide species as a function of the total cyanide concentration ($[Cu^+]_{TOT}$ = 19.1 mmol/dm³, pH 11, 25 °C), calculated with SPANA (SPANA, 2025)

most likely due to physical encapsulation or limited permeability of the ore matrix, which hampers cyanide solution access to the Au particles. Successive acid treatments with HCl and $HNO_3$ significantly enhanced Au recovery, increasing extraction to 88.5%.

During sequential leaching, each acid attacks several minerals, and the XRD pattern of the ore identifies only quartz and pyrite (Fig. 1), so the stage results indicate the reactivity of the Au-bearing matrix rather than specific host minerals. The HCl-labile and $HNO_3$-reactive fractions are therefore discussed below in terms of the phases that these acids can dissolve (Table 3). The extraction of Au in each stage, expressed as a percentage of the total Au, is shown in Figs. 5a and 6a. Direct cyanidation extracted 18.4% of the Au, so 81.6% of the Au is refractory to cyanide. Treatment of the cyanidation residue with HCl did not dissolve any measurable Au. Subsequent cyanidation of the HCl-treated residue extracted a further 22.6% of the total Au (27.6% of the Au entering the stage), indicating that this fraction was probably associated with carbonate and oxide phases and may not be limited to those. The low total carbon content of the ore (0.15%, Table 2) suggests that carbonates make only a minor contribution. Cyanidation of the $HNO_3$-treated residue extracted 47.6% of the total Au, i.e., 58.3% of the refractory Au (not extracted by direct cyanidation) and 80.5% of the Au entering this stage. This fraction is associated with an $HNO_3$-reactive, sulfide-bearing matrix, with pyrite as the only sulfide identified by XRD. The cumulative Au extraction after the $HNO_3$-cyanidation stage was 88.5%. The remaining 11.5% of the total Au is not accounted for by the stages applied and is reported as an unresolved residual refractory fraction. No HF stage was included, and the residue was not examined mineralogically. Encapsulation in silicate minerals is a plausible explanation given the high $SiO_2$ content of the ore. Association with other minor non-sulfide phases is also possible. An HF stage and/or mineralogical characterization of the final residue would be required to resolve this fraction.

Silver behaved differently from gold (Figs. 5b and 6b). Direct cyanidation extracted 26.9% of the Ag. In contrast to Au, 52.5% of the total Ag (71.9% of the Ag entering the stage) was dissolved directly in the HCl stage and a further 0.3% in the $HNO_3$ stage, whereas cyanidation after the HCl and $HNO_3$ stages extracted 3.4% and 7.7%, respectively. Cyanide therefore extracted 38.0% of the Ag in total, and the acid stages dissolved 52.9%. The cumulative extraction of 90.9% is the sum of the cyanide- and acid-extracted Ag and cannot be taken as evidence that Ag is hosted in the same phases as Au. The direct dissolution of Ag in ≈1.45 mol/dm³ HCl at 60 °C is consistent with the formation of chloro-complexes ($AgCl_2^-$, $AgCl_3^{2-}$). These data cannot identify the host phases of Ag. The remaining 9.1% of the Ag is part of the unresolved residual fraction.

The deportment of the base metals (Cu, Fe, and Zn) across the diagnostic leaching stages provides complementary information (Figs. 7a–c). Direct cyanidation extracted 22.7% of the Cu and 2.41% of the Zn. The HCl stage dissolved some Fe and Zn, suggesting partial dissolution of acid-soluble phases. The largest increases in the extraction of all three metals occurred in the $HNO_3$ stage, with cumulative extractions reaching 76.32% (Cu), 90.58% (Fe), and 90.3% (Zn), indicating extensive decomposition of a sulfide-bearing matrix under oxidative acid-leaching conditions. This is consistent with the Au deportment, in which 47.6% of the total Au was released after the $HNO_3$ stage. After the $HNO_3$–

cyanidation stage, the cumulative extractions of Cu, Fe, and Zn reached 97.9%, 90.8%, and 91.7%, respectively. Previous studies by the authors showed that sulfide flotation enriched Au, Cu, Zn, and Fe, together with sulfur, in the flotation concentrate (Msumange et al., 2018), consistent with the association of these metals with sulfide phases.

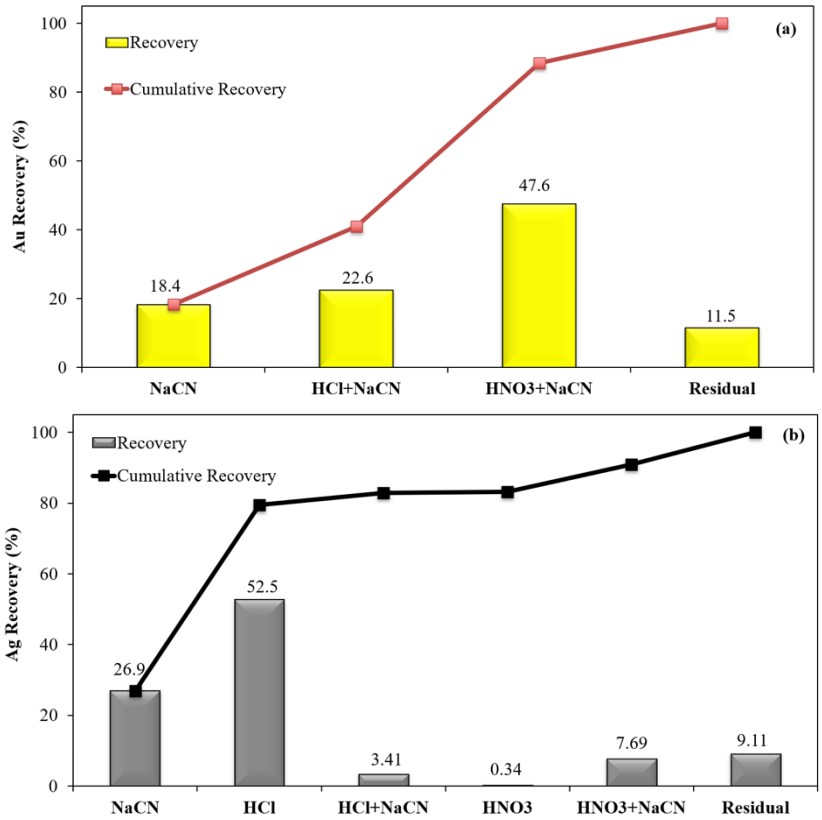



Fig. 5. Deportment of gold (a) and silver (b) in the Mastra ore sample following successive diagnostic leaching stages

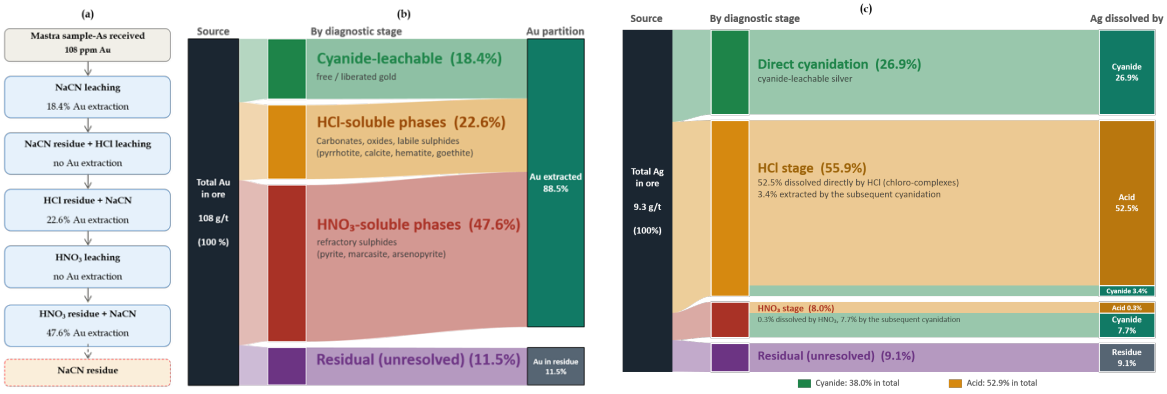



Fig. 6. (a) Diagnostic leaching steps showing stage-wise recovery and distribution of Au following sequential acid pre-treatment and cyanidation, and overview of the results in Sankey diagrams for Au (b) and Ag (c), in which the residual fraction is shown as unresolved and the acid-dissolved and cyanide-extracted Ag are distinguished

The results indicate that direct cyanidation of this ore is inefficient and that a pre-treatment that destroys the $HNO_3$-reactive sulfide matrix, which held 47.6% of the total Au, and manages the cyanide-soluble Cu should be considered. Roasting (650 °C) on this ore showed increasing Au extraction to 73.4% at 1.5 g/dm$^3$ NaCN and to about 96% at 5 g/dm$^3$ NaCN (Msumange, 2019; Msumange et al., 2023). In contrast, ultrafine grinding, increased cyanide concentration, and $H_2SO_4$ pre-treatment alone did not raise Au extraction above 80% (Msumange, 2019; Msumange et al., 2020), consistent with Au encapsulation in the sulfide matrix. Pressure oxidation, bio-oxidation, and chemical oxidation are other

possible options, but they have not been tested on this ore. The cyanide-soluble Cu (22.7% of the Cu in direct cyanidation) would also need to be managed, for example by ammonia- or EDTA-assisted cyanidation or by the recovery of Cu and cyanide from the leach solution (Baş et al., 2012; Dai et al., 2012; Msumange et al., 2021, 2022; Kuzu et al., 2026). Msumange et al. (2023) showed that ion-exchange (IX) resins can selectively recover gold over copper from cyanide leach solutions of roasted ore.

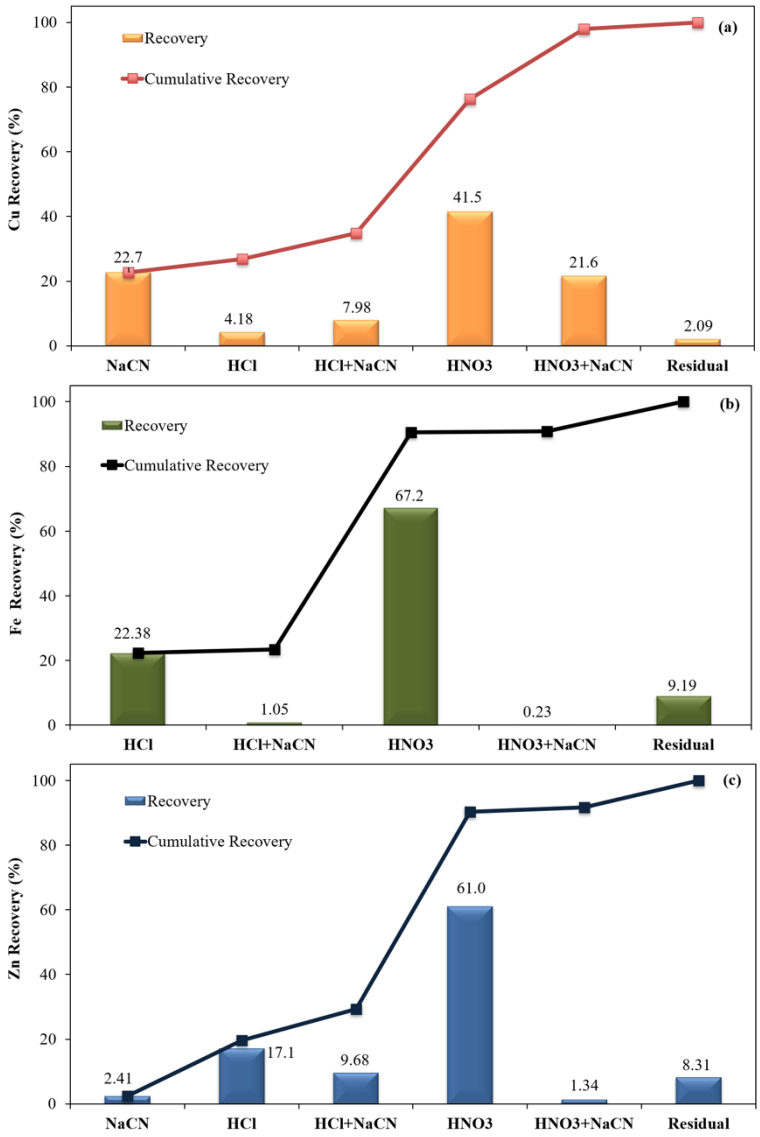



Fig. 7. Deportment of copper (a), iron (b), and zinc (c) in the Mastra ore sample at diagnostic leaching stages

## 4. Conclusions

Diagnostic leaching of the Cu-rich Mastra sulfide-zone gold ore confirmed its strongly refractory nature. Direct cyanidation extracted only 18.4% of the Au, and sequential HCl and $HNO_3$ pre-treatments, each followed by cyanidation, increased the cumulative Au extraction to 88.5%. The high cyanide consumption of direct cyanidation (6.75 kg/Mg) is largely attributed to the dissolved Cu (22.7% of the Cu), which can account for most of the cyanide consumed, as shown by the stoichiometric estimate and the speciation of the Cu(I)–$CN^-$ system. The 47.6% of the total Au released after $HNO_3$ treatment is probably associated with a sulfide-bearing matrix. The HCl stage released a further 22.6% of the Au from an HCl-labile fraction. The remaining 11.5% of the Au is an unresolved residual refractory fraction, for which encapsulation in silicate minerals is a plausible but undemonstrated explanation. Silver behaved differently from gold. The acid stages dissolved 52.9% of the Ag, mainly in the HCl stage, and cyanide extracted 38.0% in total, so the Ag host phases cannot be inferred from the Au deportment. The

base metals Cu, Fe, and Zn dissolved mainly in the $HNO_3$ stage, consistent with decomposition of a sulfide-bearing matrix. From a processing perspective, the results indicate that direct cyanidation of this ore is inefficient and that a pre-treatment that destroys the $HNO_3$-reactive sulfide matrix and manages the cyanide-soluble Cu should be considered. The diagnostic leaching results, therefore, provide valuable data for flowsheet development, highlighting oxidative pre-treatment as a requirement for improving precious metal recovery from this complex refractory ore.

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