

Quiescent flow leaching as a tool for assessing long-term weathering of ferronickel slag: insights from a 1.5-year experiment

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ARTICLE INFO

Editorial handling by: Elisa Sacchi

ABSTRACT

Long-term leaching experiments provide estimates of the release of potentially toxic elements under controlled laboratory conditions. In this study, metallurgical slags originating from a ferronickel smelting operation were subjected to a leaching test under stagnant laboratory conditions at pH levels of 2.9 and 4.9 for 18 months. The results revealed significant variation in the leaching potential of the studied slags, with nickel and cobalt release ranging from less than 0.01% to as high as 90%. Of the samples analyzed, electric furnace slag produced by smelting predominantly Indonesian ores (IS-A slag) exhibited the greatest potential as a source of nickel, primarily due to the dissolution of nickel sulfides (mostly pentlandite) and the presence of finely dispersed, micron- to nanometer-scale nickel-bearing grains. The dissolution of the major silicate phases, particularly forsterite, was evidenced by the release of magnesium, reaching 1.9–2.1% in the same type of slag (IS-A). Slags from the same source (IS-B and IS-C), which were rich in magnesiochromite, contributed to chromium mobilization in the range of 0.3–0.6%. Furthermore, the dissolution of calcium silicate (mostly larnite) was confirmed by substantial calcium release (at least 13%) in 40-year-old slag formed after the magnetic separation (MS slag) of ores from the Republic of North Macedonia (Ržanovo Mine). This contrasts with slags lacking this phase. This study indicates that long-term stagnant-flow experimental conditions can illustrate the reactivity features of slags and demonstrate the growing influence of laboratory experiments in estimating the geochemical stability of individual slag phases.

1. Introduction

Incubation experiments known as leaching tests have a long tradition of evaluating the environmental stability of various solid materials under laboratory-controlled conditions (Tiwari et al., 2015). The underlying principle of leaching experiments is to blend the reactivity of solids and influencing factors to gain a better understanding of their potential behavior in the environment (Van Der Sloot, 1996). A quantitative approach enables changes in the dynamics and efficiency of elements released over time to be assessed. The leaching trend and intensity are specific to the dissolution factor that influences the material and constitute *prima facie* evidence of the geochemical stability of

certain solids (Faraji et al., 2022). In addition, the focus placed on the observation of the solid surface after termination of the experiment enables us to assess how individual metal-bearing phases contribute to element input to the leachate. Leaching typically relies on the immersion of a solid in a liquid solution whose composition is specific to the context under study. Different experimental strategies have been employed to investigate the environmental footprint of solid weathering (De Windt and Badreddine, 2007; Lewin, 1996; Neuhold et al., 2019). In general, abiotic and biotic factors can be used together. However, in real environments, they often have different roles (Schwartzman and Volk, 1989). In addition, field conditions differ from those simulated on a laboratory scale. Therefore, the reactivity of solids in the environment

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<https://doi.org/10.1016/j.apgeochem.2026.106757>

Received 28 October 2025; Received in revised form 3 March 2026; Accepted 3 March 2026

Available online 9 March 2026

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may be much more variable (Calisi and Bentley, 2009).

A particular example is employing an experimental approach to evaluating environmental risk associated with the disposal of metal-bearing materials, such as slags, ashes, and other anthropogenic residues that cause considerable environmental concern (Faraji et al., 2022; Piatak et al., 2015; Shi et al., 2022). Two types of leaching tests are used for slags: those with standardized protocols and those with designed protocols (Ettler and Vítková, 2021). The former tests enable the behavior of the slag to be related to the concentration levels of specific elements considered hazardous according to regulatory limits, whereas the latter are designed to evaluate the influence of a specific factor on the stability of the material (Ettler and Vítková, 2021; Piatak et al., 2015; Shanmuganathan et al., 2008). Furthermore, leaching tests include the simulation of soil and rhizosphere conditions, bacterially mediated weathering, and acid rain, all of which contribute to a better understanding of slag disposal scenarios (Derkowska et al., 2023; Ettler et al., 2004; Kierczak et al., 2013; Potysz et al., 2016; Tyska et al., 2021; Warchulski et al., 2019). In this regard, the reliance on and use of experimental approaches has provided guidance on the environmental risks associated with slag disposal (da Silva et al., 2017; Kicińska, 2021; Król et al., 2020; Kierczak et al., 2008). Technically, most leaching tests are carried out under dynamic conditions that include flask shaking to ensure better reactivity between the solid and liquid (Ettler and Mihaljević, 2023; Potysz, 2023; Warchulski et al., 2015). This approach allows for the repeatability of liquid–solid interactions in environments related to frequent rainfall to be illustrated. In addition, the contact of slags with the weathering solution in real time is dependent on the slag's placement within the heap. Migration of the weathering solution (e.g., acid rain) through the dump results in slag located in the interior being exposed for a longer time, whereas slag placed on the outer parts is exposed for a shorter duration due to the solution passing through faster (Potysz et al., 2017; Tyska et al., 2014). In this regard, the stagnation of the solution inside the dump could be simulated by conducting leaching experiments under laboratory conditions without shaking. Thus, the pertinent question regarding long-term stability under stagnant laboratory conditions remains unanswered. In addition, relatively limited attention has been given to studies considering slags originating from ferronickel processing (Kierczak et al., 2009; Ettler et al., 2016; Gre-gurek et al., 2014; Lanteigne et al., 2014; Schindler, 2014). Therefore, this experimental study was undertaken to demonstrate the reactivity of metallurgical slags originating from ferronickel smelting operations. The toxicity characteristic leaching procedure (TCLP) 1311 was modified in terms of leaching time and conditions, with the time extended to 18 months and the incubation performed under stagnant-flow conditions. Slags with different chemical and phase compositions were tested to allow for comparative estimates of environmental hazards.

The research hypothesis of this study states that ferronickel slags contain metal-bearing phases present in different volumetric proportions, which in turn affect the potential of these wastes to release potentially toxic elements (PTE). The specific research questions are as follows: (i) What is the susceptibility of individual phases to contaminant release? (ii) What is the quantitative estimate of contaminants potentially transferred to the environment? (iii) Do the slags reveal any treatment perspectives for resource recovery?

2. Materials and methods

2.1. Sample description

Ferronickel slags were sampled from a large slag disposal site near Vozarci village, adjacent to the city of Kavadarci, in the Republic of North Macedonia. Approximately 40 kg of material was collected to represent various processing stages and weathering histories. For a detailed description of the operational settings, see (euronickel.com). The samples included (a) electric furnace slags (EFSs) from various sources and of different ages, including freshly produced slags and slags

that had been weathered for up to 40 years (IS-A, IS-B, IS-C, ICS, and RS); (b) converter slags of different ages (CS); and (c) magnetic slags (MS) from the earlier separation process of the raw ferronickel.

The melt generated in the ferronickel plant is discharged onto a dump and allowed to cool under ambient conditions. This quenching strategy results in the formation of massive slags containing crystalline phases. Therefore, the samples were crushed and milled prior to leaching experiment.

2.2. Long-term leaching experiment

A long-term leaching experiment was conducted under quiescent flow conditions to evaluate the materials' leaching behavior and environmental stability over extended timescales. TCLP 1311 was carried out using two types of solutions: (1) one fixed at pH 4.9 prepared through the addition of 5.7 mL of acetic acid ($\text{CH}_3\text{CH}_2\text{OOH}$) and 64.3 mL of 1 M NaOH to 1 L of distilled water and (2) one fixed at pH 2.9 prepared through the addition of 5.7 mL of acetic acid ($\text{CH}_3\text{CH}_2\text{OOH}$) to 1 L of distilled water. The leaching test was performed at a solid-to-liquid ratio of 1:15 by adding 2 g of slags (particle size 30–35 μm) into 30 mL of the respective solution. The solutions were kept in the dark under ambient temperature and stagnation conditions without shaking for 18 months (1.5 years). The containers were gently closed to permit airflow while minimizing solution evaporation. Subsequently, the leachates were sampled and filtered using 0.45 μm PTFE filters, and the pH was measured using an ELMETRON CP-505 pH meter equipped with an ERH-111 electrode (no. 9997). Other portions of leachate aliquots were acidified using Suprapur 65 wt% HNO_3 (Merck). The concentrations of the elements in the leachates were measured using an inductively coupled plasma optical emission spectrometer (ICP-OES; Avio 200, PerkinElmer, Waltham, MA, USA). The following wavelengths were used for analysis: As (188.969 nm), Cr (267.716 nm), Zn (213.857 nm), Cu (327.393 nm), Co (228.616 nm), Ni (231.604 nm), Mn (257.610 nm), Al (396.153 nm), Fe (259.939 nm), Mg (285.21 nm), and Ca (317.933 nm). Calibration curves were prepared using multielement standard solutions prepared from single-element standard solutions (Merck). The analysis conditions were as follows: a 12 L/min plasma argon gas flow, a 0.2 L/min auxiliary gas flow, a 0.7 L/min nebulizer gas flow, 1500 W power, and a 1 mL/min sample flow rate. Diluted multielement-certified reference material (CPAchem Ltd.) was analyzed to check the correctness of the calibration curves. The stock solution with a concentration of 1000 mg dm^{-3} of each element was diluted in a glass volumetric flask to a concentration of 1 mg dm^{-3} . The concentrations of elements measured in the diluted certified reference material were as follows: 1.04 mg dm^{-3} for As, 1.06 mg dm^{-3} for Cr, 1.03 mg dm^{-3} for Zn, 1.01 mg dm^{-3} for Cu, 1.11 mg dm^{-3} for Co, 1.12 mg dm^{-3} for Ni, 1.08 mg dm^{-3} for Mn, 1.05 mg dm^{-3} for Fe, 1.01 mg dm^{-3} for Al, 1.02 mg dm^{-3} for Mg, and 0.98 mg dm^{-3} for Ca.

2.3. Phase characterization

To determine the bulk phase composition of the crystalline phases, powder X-ray diffraction (PXRD) analyses were conducted on five distinct slag groups (IS, MS, CS, ICS, and RS) prior to leaching as well as after 18 months of leaching at pH 2.88 and pH 4.93. The aim was to evaluate their mineralogical composition and long-term phase stability under acidic conditions. The corresponding PXRD patterns are presented in Fig. S1–S3 (Supplementary materials).

The powders used for the long-term leaching experiments were analyzed using PXRD prior to and following leaching. A Bruker D8 Advance diffractometer (Cu-K α radiation, Lynxeye detector, 40 kV, 25 mA, step scanning from 5° to 125° 2 θ at room temperature, step size of 0.01° 2 θ , and dwell time of 1 s per step) was used for the PXRD analyses. Peak and phase identification was performed using DIFFRAC.EVA software, version 4.2, and the ICDD powder diffraction file PDF-2.

The chemical composition and micromorphology of the mineral-like

phases were studied before and after leaching using a JEOL JSM-6610 LV scanning electron microscope (SEM) with a tungsten filament (15 kV) and an energy-dispersive X-ray (EDX) detector (Bruker E-Flash HR+, resolution 127 eV). Esprit 2.0 software from BRUKER was used for the evaluation. Semi-quantitative (standard-less) analyses and imaging were carried out in high vacuum mode (10^{-4} mbar). The spot size was 60—a dimensionless number composed of the beam current and the beam diameter. The current could not be individually controlled or read. Spectra were recorded for 60 s, with dead times of less than 5%.

Transmission electron microscopy (TEM) was employed because the slag powder consisted of very fine particles (many of them smaller than 10 μm), which were too small for reliable characterization by SEM; additionally, TEM sample preparation in ethanol enabled effective dispersion and separation of individual powder particles used for XRD and leaching experiments, allowing more accurate microstructural analysis. For TEM analysis, the sample material must be electron transparent, which requires it to be sufficiently thin. We achieved this by regrounding the material, with the resulting fine powder mixed with ethanol to separate the particles. A droplet of this mixture was placed on a 3-mm TEM grid, which was covered with a perforated carbon support film. The ethanol then evaporated, leaving individual particles of the sample material on the disk surface. A detailed microstructural investigation was performed using an FEI Tecnai F20 S-TWIN analytical high-resolution microscope operated at 200 kV in combination with a windowless Apollo XLTW silicon drift detector for EDS experiments at the University Service Facility for Transmission Electron Microscopy at TU Wien.

3. Results

3.1. Phase composition of slags prior to 18 months of leaching

The PXRD patterns of all three IS slags (IS-A, IS-B, and IS-C) prior to leaching indicated the dominance of silicate phases, primarily minerals of the olivine group (Fig. S1, Supplementary materials). The most abundant phase in all samples was Fe-bearing forsterite (Mg_2SiO_4), hereafter referred to as a forsterite–fayalite ($\text{Mg}_2\text{SiO}_4\text{--Fe}_2\text{SiO}_4$) solid solution, with variable chemical composition depending on the individual slag. Pyroxenes were also present, mainly enstatite ($\text{Mg}_2\text{Si}_2\text{O}_6$) and clinoenstatite ($\text{Mg}_2\text{Si}_2\text{O}_6$). All IS diffractograms exhibited a pronounced diffuse background hump, indicating a significant amorphous component attributed to glassy phases typical of rapidly cooled ferromagnetic slags.

The ICS slags (Fig. S2, Supplementary materials) were similarly dominated by forsterite as the principal crystalline phase. Minor secondary silicate phases may have been present, but they did not contribute significantly to the overall diffraction signal. The RS slags (Fig. S2, Supplementary materials) exhibited a mineral assemblage dominated by forsterite, accompanied by substantial amounts of chromite-group minerals identified as magnesiochromite–chromite ($\text{MgCr}_2\text{O}_4\text{--Fe}^{2+}\text{Cr}_2^{3+}\text{O}_4$) solid solutions. Pyroxenes, primarily diopside ($\text{CaMgSi}_2\text{O}_6$) with subordinate enstatite–clinoenstatite, were also present. The occurrence of chromite was a distinguishing feature of the RS slags and reflected the compositional characteristics of the original ore from the Ržanovo deposit in North Macedonia.

In contrast, the PXRD patterns of both MS and CS slags prior to leaching (Fig. S3, Supplementary materials) were dominated by iron-rich oxide phases, reflecting their converter slag origin. The main crystalline phases were magnesioferrite (MgFe_2O_4) and hematite (Fe_2O_3), which exhibited strong and well-defined diffraction peaks indicative of a predominantly crystalline material. Minor calcium-bearing phases were also detected. Calcium sulfate phases, including gypsum ($\text{CaSO}_4\cdot 2\text{H}_2\text{O}$) and anhydrite (CaSO_4), were present in the CS slag, while calcium silicates (larnite, Ca_2SiO_4) were present in both the MS and CS slags. Compared with the IS slags, the amorphous component in the converter slags was less pronounced, as indicated by a lower background

contribution.

3.2. Phase composition of slags after 18 months of leaching

After 18 months of leaching, the PXRD patterns of IS-A (Fig. S1, Supplementary materials) showed pronounced mineralogical changes, particularly under more acidic conditions (pH 2.88). The most notable feature was a partial disappearance and reduction in the intensity of the forsterite–fayalite reflections, indicating partial dissolution of olivine during leaching. In addition, magnetite ($\text{Fe}^{2+}\text{Fe}_3^{3+}\text{O}_4$) formation was observed in the IS-A sample leached at a pH of 2.88, as evidenced by the appearance of characteristic reflections absent in the initial material. This suggests secondary iron oxide formation resulting from iron release during silicate dissolution and subsequent reprecipitation under acidic conditions.

In contrast, the IS-B slag exhibited only minor changes in the PXRD patterns after leaching at both pH values (Fig. S1, Supplementary materials). The olivine and pyroxene phases remained largely stable, with no significant reduction in peak intensities. Minor phase evolution was inferred from the appearance of Fe-rich enstatite, suggesting limited Fe redistribution within the pyroxene structure. Overall, IS-B displayed the highest resistance to leaching among the IS slags.

The IS-C slag (Fig. S1, Supplementary materials) showed the most pronounced mineralogical transformation following leaching. PXRD patterns revealed extensive dissolution of olivine, reflected by a strong decrease or near disappearance of its characteristic reflections. Cristobalite (SiO_2), present in the initial IS-C slag, was no longer detected after leaching. Since cristobalite occurs as a minor phase, the grains used in the leaching experiments may not have contained it. This is consistent with the high heterogeneity of the slag and the low volumetric abundance of SiO_2 . As a result, clinoenstatite became the dominant crystalline phase in the leached samples. Minor reflections corresponding to calcium silicate phases appeared after leaching, suggesting secondary phase formation or exposure to previously masked components.

Comparisons of PXRD patterns before and after leaching showed no significant mineralogical changes in either the ICS or RS slags under both pH conditions (Fig. S2, Supplementary materials). Diffraction peaks corresponding to forsterite, chromite (magnesiochromite–chromite solid solutions), and pyroxenes remained unchanged in position and relative intensity. No evidence of phase dissolution, secondary mineral formation, or amorphization was observed, confirming the high chemical stability of these phases.

Similarly, the PXRD patterns of both the MS and CS slags showed only limited changes after leaching (Fig. S3, Supplementary materials). Reflections of magnesioferrite and hematite remained essentially unchanged, indicating these iron oxides' strong resistance to long-term acidic leaching. However, selective dissolution of the calcium-bearing phases was evident, particularly at a pH of 2.88. In the CS slag, gypsum and anhydrite reflections disappeared or significantly decreased, while in the MS slag, a reduction in larnite-related peaks was observed. At pH 4.93, these phases were largely preserved, highlighting the strong pH dependence of calcium phase stability.

3.3. Electron microscopy characterization of preleaching slags

The mineralogical characteristics of the Fe–Ni slags produced by EuroNickel Industries were previously reported by Đorđević et al. (2024). The powdered materials used in the present 18-month leaching experiment were identical to those used in earlier PXRD and chemical extraction studies. While general mineralogy was previously described based on PXRD data, the powdered samples were not examined using SEM-EDX. In this study, the same powders were therefore analyzed using transmission electron microscopy (TEM) for the electric furnace slags (IS, RS, and ICS), while the converter slags (CS and MS) were characterized using Raman spectroscopy.

TEM-EDS point analyses of the EFS slags (IS-A, RS, and ICS; Table S1,

Supplementary materials) indicated a silicate glass matrix hosting olivine, pyroxene, and spinel crystals with minor intermetallic inclusions. The olivine-group phases were predominantly Fe-bearing forsterite, with Fe contents ranging from 2 to 10 at.%, locally transitioning to fayalite where Fe exceeds Mg. Pyroxenes are enstatite-type and Fe-bearing (2–5 at.% Fe). The glassy matrix contained minor Ca, Al, and Fe. Bright-field TEM images (**Fig. S5, Supplementary materials**) showed irregularly shaped particles with, reflecting differences in the thickness and aggregation of finer particles.

STEM-EDS mapping (**Fig. 1**) revealed heterogeneous elemental distributions, with Mg–Si-rich regions corresponding to olivine or pyroxene and Fe–Cr-rich domains attributed to chromite. Electron diffraction patterns (**Fig. S4 and S5, Supplementary materials**) confirmed a cubic spinel structure (space group $Fd\bar{3}m$), consistent with chromite identified by the combined chemical and crystallographic evidence. Similar mappings of the Fe oxide phases in the RS samples (**Fig. S6, Supplementary materials**) indicated the presence of magnetite and Ni-bearing oxides.

Raman spectroscopy on the slags' powders prior to leaching was published in [Đorđević et al. \(2024\)](#). MS samples were dominated by magnetite and chromite, with fayalite and monticellite (CaMgSiO_4) as accompanying silicates. CS samples showed a more oxidized assemblage dominated by magnesioferrite and hematite, along with sulfate and carbonate minerals, such as anhydrite, calcite, and syngenite. The Raman signatures of the intermetallic particles further confirmed the presence of reduced metallic phases.

Overall, the preleaching slags were mineralogically and chemically heterogeneous, comprising Mg–Fe silicates, silicate glass, oxides, spinels, sulfates, carbonates, and intermetallic phases. This complexity reflected variable cooling histories and redox conditions and strongly influenced subsequent leaching behavior.

3.4. Electron microscopy characterization of postleaching slags

The solid residues obtained after 18 months of leaching were analyzed using SEM-EDS. The postleaching materials consisted of a limited number of chemically stable phases, indicating strong selective dissolution. The dominant components were Mg–Fe silicates, silica-rich glass, iron oxides, and hydrous ferric oxides (HFOs). Silicates were mainly represented by forsterite–fayalite solid solutions and enstatite-type phases with minor Al, Cr, Ca, and Mn substitutions (**Table S2, Supplementary materials**). Calcium-bearing silicates were rare, suggesting preferential Ca removal during leaching.

Glassy phases persisted but exhibited more homogeneous, silica-rich compositions, characterized by high Si and O contents with variable Fe, Mg, Al, and Ca and trace Cr, Mn, Na, K, Ti, and S. These features indicated incongruent dissolution, leaving behind silica-dominated residual structures. Iron released during leaching was immobilized as secondary Fe oxide and HFO phases enriched in Fe and O, with minor Mg, Al, and trace elements. The formation of secondary Fe oxides is depicted in an SEM-EDX map of part of the IS-A sample (resulting from incubation with a pH 2.88 solution) (see **Fig. 2**). This map also shows how the chromite spinels and the Fe–Ni intermetallic particles remained largely unaltered, confirming their high chemical stability.

3.5. Comparison between pre- and postleaching states

Prior to leaching, the slags exhibited greater chemical and mineralogical heterogeneity, dominated by primary silicates embedded in compositionally complex glass. Fe was mainly incorporated into silicates and glass, while alkali and alkaline-earth elements were abundant, particularly in the glassy matrix ([Đorđević et al., 2024](#)). The discrete Fe oxide phases were relatively limited. Leaching induced substantial elemental redistribution rather than complete phase destruction (**Fig. 3**). Silicate minerals remained structurally intact but became chemically simplified, enriched in Mg and Fe, and depleted in more soluble

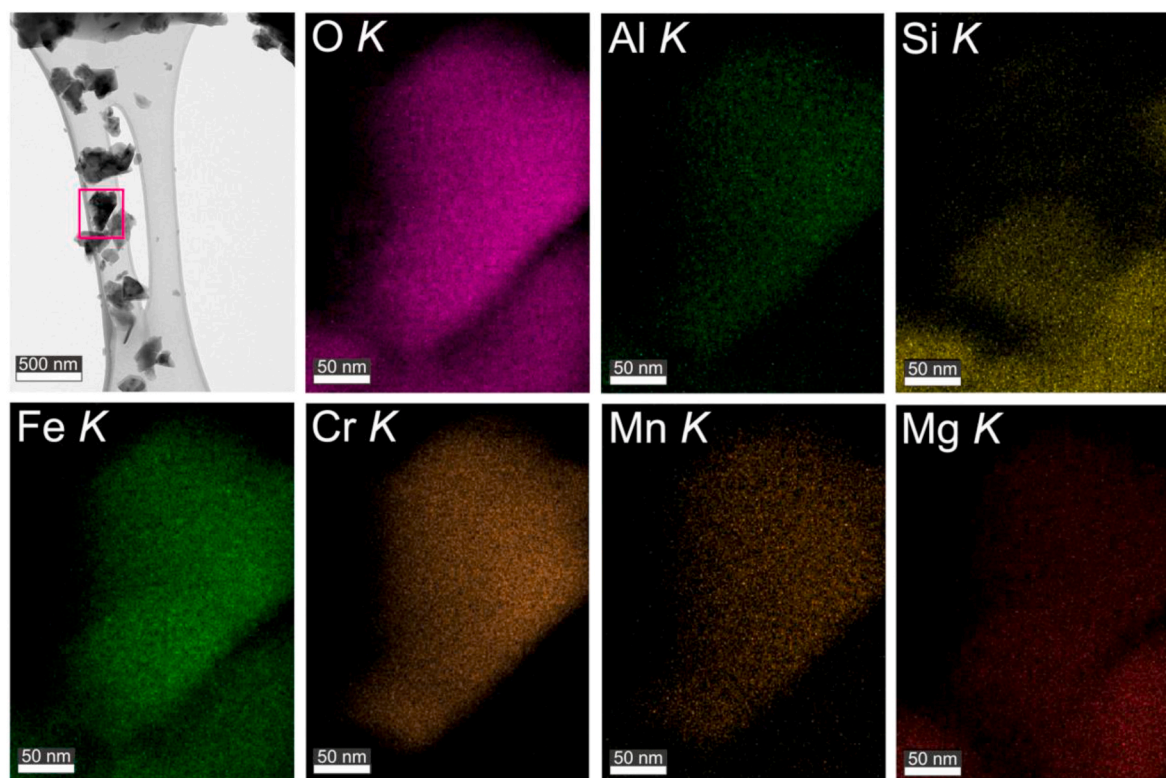


Fig. 1. Bright-field TEM image showing the form and size of the slag particles lying on a Cu-grid from the IS-A sample and STEM elemental maps of the Mg–Si-rich regions corresponding to olivine or pyroxene and the Fe–Cr-rich domains attributed to chromite.

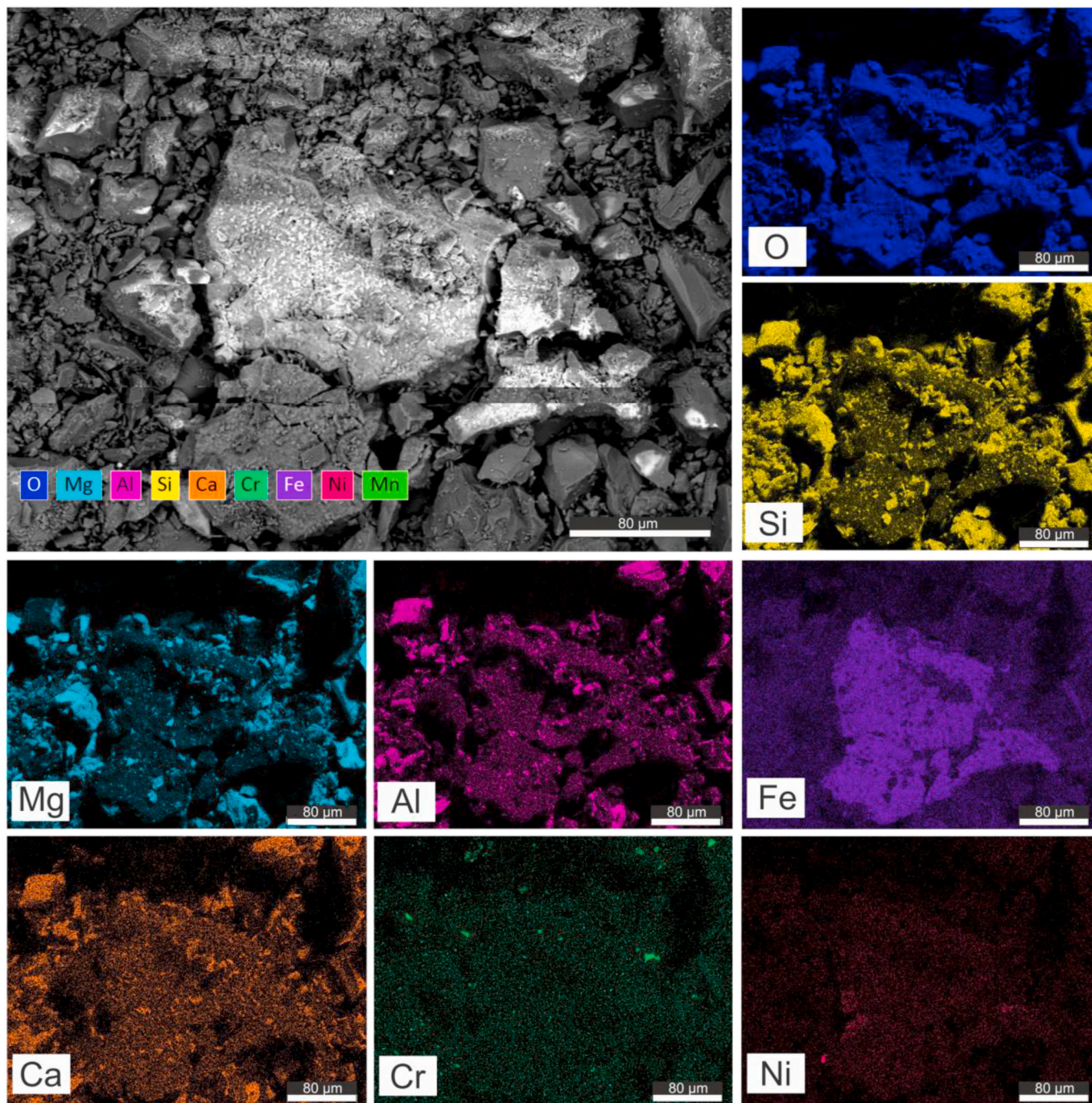


Fig. 2. SEI - SEM image of the IS-A sample showing the formation of Fe oxides and SEM-EDS elemental maps showing postleached Fe oxide, Mg-Fe silicates, chromite, and intermetallic Fe-Ni phase.

elements, such as Ca, Na, and K. Glassy phases evolved into silica-rich residual glasses. The most significant transformation involved Fe, which was redistributed from silicates and glass into secondary Fe oxide and HFO phases. In contrast, chromite spinels remained largely unaffected. The metal-bearing phases, particularly the ferronickel particles (Fe-Ni), were highly susceptible to weathering (Fig. 3), as supported by the appearance of weathering crusts, which were most likely formed after the weathering of Fe-Ni particles and Fe-Ni sulfides. Similarly, the iron oxides displayed clear signs of partial dissolution (Fig. 3c and h). Several samples exhibited a distinct weathering crust on the slag surfaces (Fig. 3f), indicating progressive surface alteration. In contrast, forsterite dissolution was sporadic and much less pronounced (Fig. 3d

and e).

In summary, long-term leaching transformed the slags from chemically complex silicate-glass systems into residual assemblages dominated by Mg-Fe silicates, silica-rich glass, and iron oxides, reflecting strong selective dissolution and preservation of the most chemically resistant phases.

3.6. Leaching results

The amount (mg kg^{-1}) of elements leached from slags to the solution after 18 months followed different patterns that varied from one slag to another (Figs. 4 and 5). Leaching efficiency, expressed as a percentage

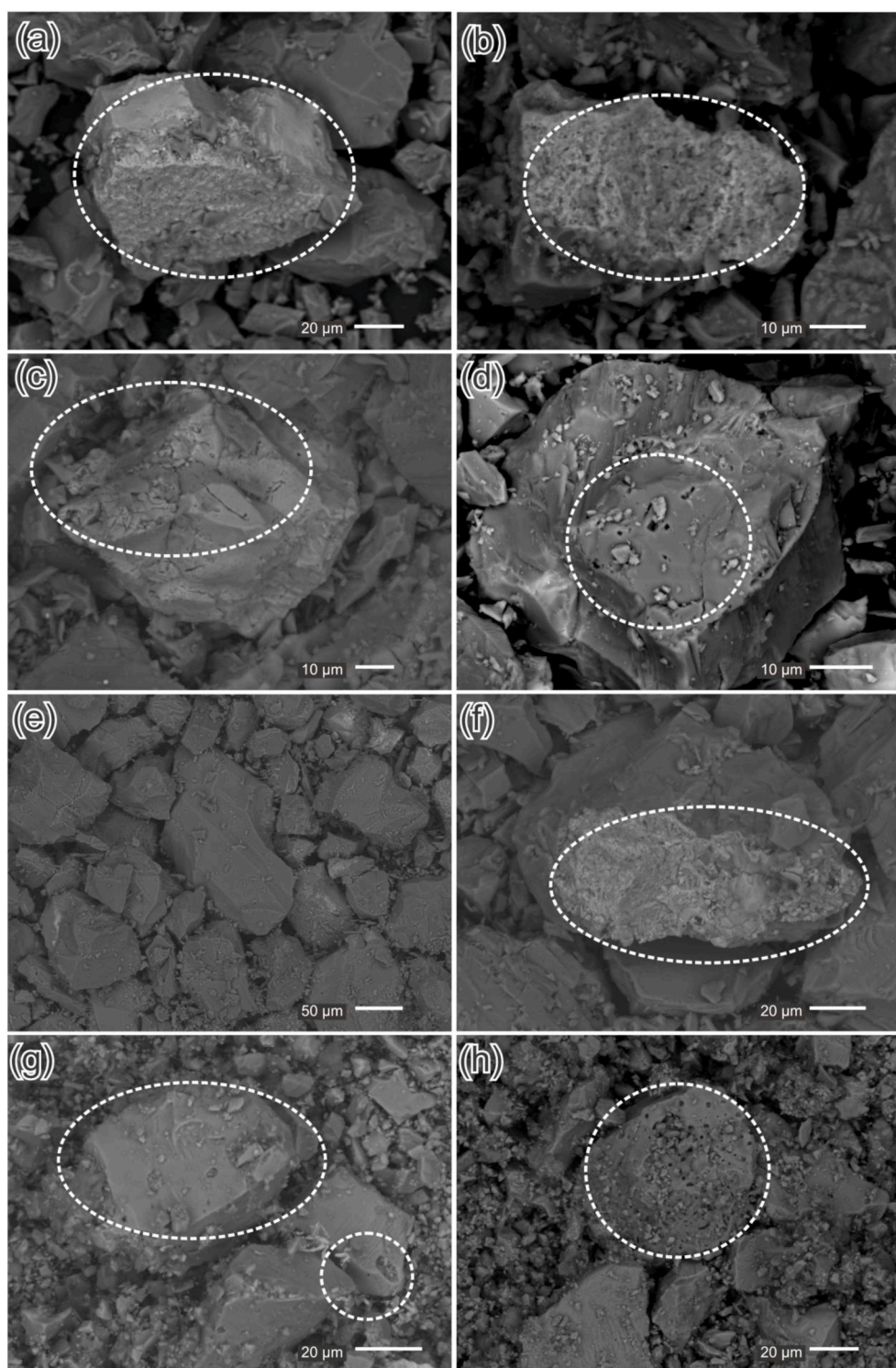


Fig. 3. Weathering features observed on slag grains after 18 months of the quiescent flow experiment. BSE micrographs presenting: (a) pronounced surface roughening of the ferronickel particle from the IS slag; (b) uniform micropitting resulting from the selective dissolution of ferronickel particle in the ICS slag; (c) weathering cracks on the Fe oxide of the IS slag; (d) dissolution pits on forsterite; (e) unweathered forsterite from the IS slag; (f) selective leaching texture with fine-scale surface irregularities - weathering crust of the IS slag consisting mostly of fine-grained nickel and sulfur-bearing Fe oxide or HFO; (g) partial dissolution of Fe oxide in the MS slag; (h) dissolution pits on hematite in the CS slag.

and showing the relative amount of the element leached to the amount present in the specific slag, generally revealed preferential leaching of metallic elements rather than major elements (Table S3 and S4, Supplementary Materials). In all cases, the leaching peak was observed in the treatment with an initial pH of 2.9, compared with that achieved under conditions with an initial pH of 4.9. The highest leaching of Ni

was noted for IS-A slag (up to 90.1%), whereas it did not exceed 10.8% for IS-B, 17.5% for IS-C, and 13.3% for ICS. Cobalt was released in high quantities, reaching up to 93.3% for IS-A slag. The release of Co observed for the slags was up to 2.5% for RS, up to 4.3% for ICS, up to 8.1% for IS-C, and up to 4.3% for IS-B, whereas it was negligible for MS. The release of other elements reached their maximum values: Mn 1.72%

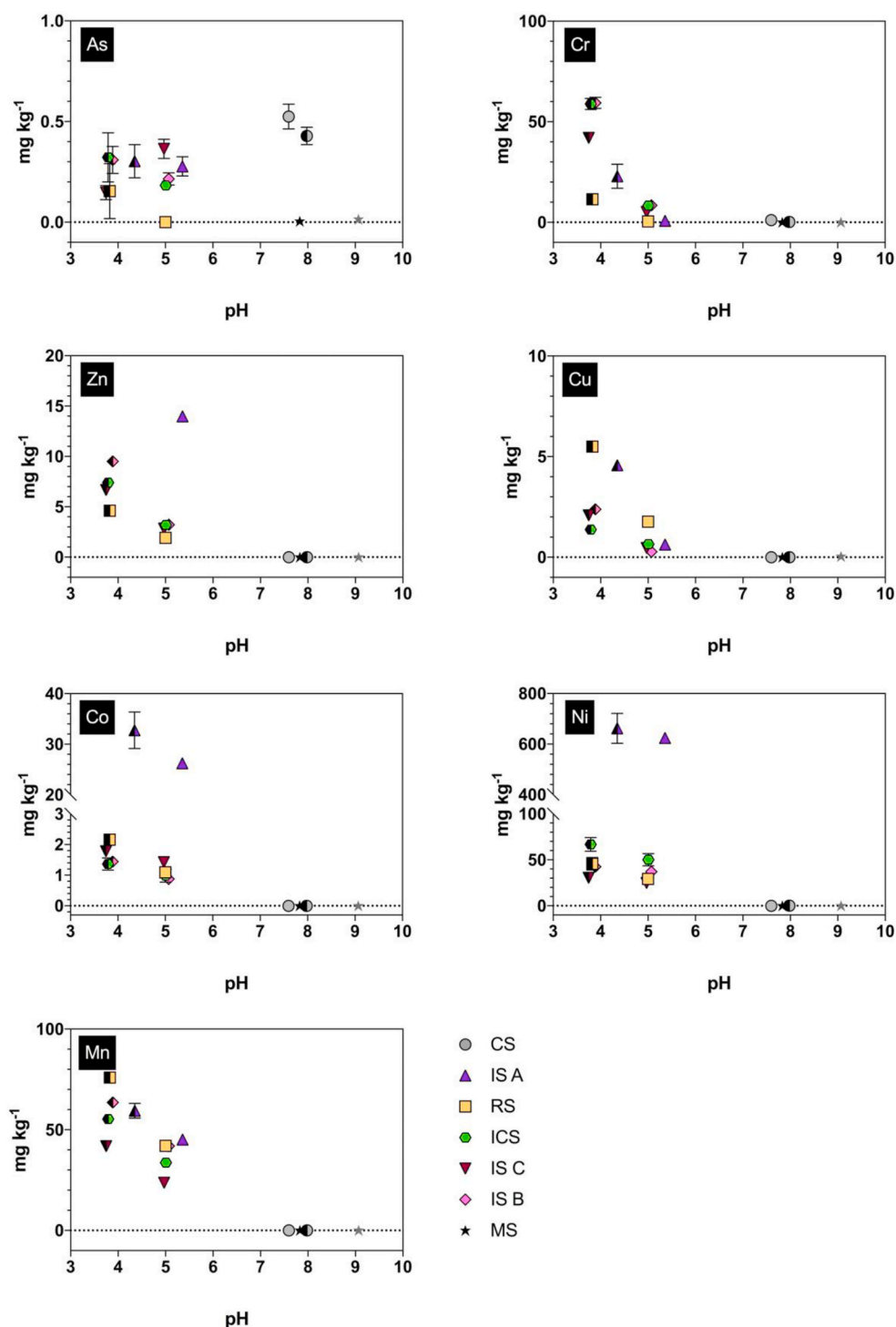


Fig. 4. Metallic element leaching resulting from slag exposure to long-term immersion in liquid. Abbreviations: CS: converter slag, IS-A/B/C: electric furnace slags, RS: Rżanowo ore slag, ICS: electric furnace slag – Ivory Coast ore, MS: magnetic slag. Black-filled data points represent leaching with solution at an initial pH of 2.88, whereas color-filled data points represent leaching with solution at an initial pH of 4.93. (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.)

(RS), Zn 6.1% (IS-A), Cu 26.9% (IS-A), As 20.6% (IS-B), Cr 0.62% (IS-B), and Pb 4.4% (IS-A).

The release of Ca reached 83.9% for MS and 22.8% for CS, whereas other slags revealed releases oscillating at 1.3–4.9%. The highest Mg leaching (10.1%) occurred in the case of CS slag, whereas for other slags, it did not exceed 2.5%. Fe and Al release was generally below 1% for all studied slags (Table S3 and S4, Supplementary Materials).

4. Discussion

4.1. Reactivity of slags exposed to stagnant-flow leaching conditions

This study focused on identifying the variables that affect the geochemical stability of metallurgical slags. A long-term experimental incubation test under stagnating flow conditions demonstrated that element leaching was linked to the composition of the slag phases. This

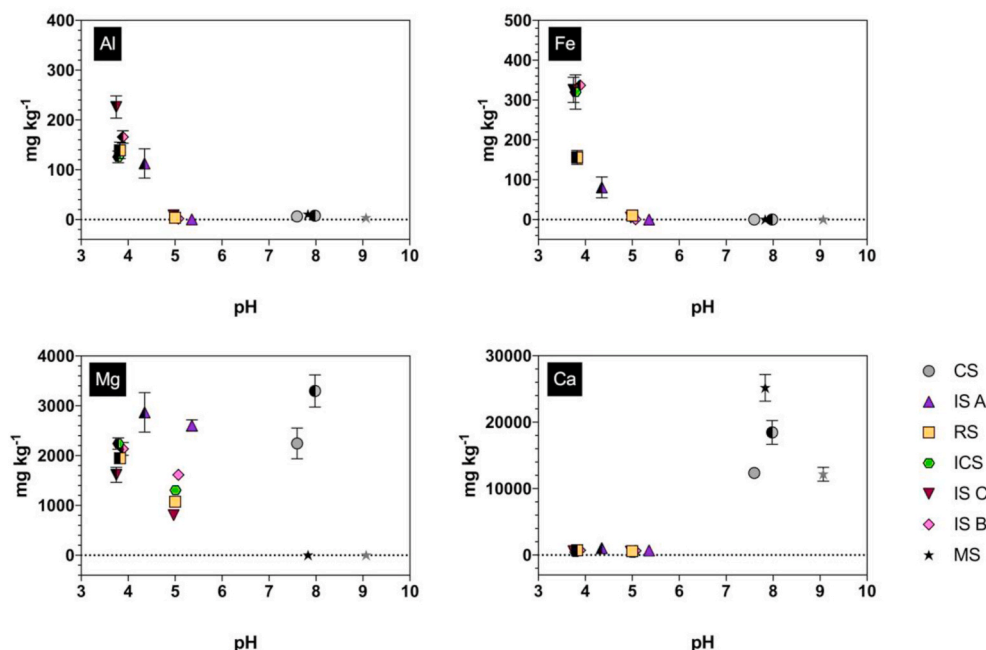


Fig. 5. Major element leaching resulting from slag exposure to 18-month immersion in liquid. Abbreviations: CS: converter slag, IS-A/B/C: electric furnace slags, RS: Ržanovo ore slag, ICS: electric furnace slag – Ivory Coast ore, MS: magnetic slag. Black-filled data points represent leaching with solution at an initial pH of 2.88, whereas color-filled data points represent leaching with solution at an initial pH of 4.93. (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.)

approach is consistent with conditions that may be encountered in natural settings, thereby highlighting its environmental validity and importance.

The weathering of slags, which results in the decomposition of individual phases, is driven by various factors, including the characteristics of the slags (e.g., their chemical and phase composition and the size and species of the metal-bearing inclusions), as well as environmental conditions (Potysz et al., 2016). In this study, two types of slag were distinguished in terms of their long-term leaching potential: Group I, which included CS and MS slags, and Group II, which included IS-A, IS-B, IS-C, ICS, and RS. Group I revealed leaching of no more than 1.5%, apart from Ca and Mg, which showed notably higher leaching of these elements (more than 10% for Mg in the case of CS slag and more than 21% for Ca in both slags) than Group II. In contrast, the latter group was found to release higher quantities of metallic elements, which was not observed in Group I. The first reason for the differing behaviors of the two groups can be attributed to the buffering potential of the individual slags (Shamim et al., 2008; Yan et al., 2000). As shown in Fig. 4, Group I ranged from pH 7.6 to 9.1, whereas Group II ranged from pH 3.8 to 5.4. Given that pH is a critical factor in controlling the dissolution of slag components, the distinct behavior exhibited by the two groups can be attributed to their contrasting pH conditions during incubation. Primarily, the slags categorized as Group I demonstrated pronounced leaching of Ca and Mg while maintaining minimal metal leaching (Figs. 4 and 5) (Đorđević et al., 2024). It is worth noting that larnite is one of the primary phases present in CS and MS slags. This phase is known for its high buffering potential due to the release of Ca^{2+} ions during the reaction of larnite with acid (Doucet, 2010; Mei et al., 2021; Rosso and Rimstidt, 2000). As Mombelli et al. (2014) explained, larnite dissolution over 24 h is negligible (1%), whereas extending the leaching time to over 100 days may result in 50% hydration. Thus, larnite was likely responsible for buffering the solution's pH, shifting its values from acidic to slightly alkaline (Fig. 4). Another phase observed in CS and MS is magnesioferrite, which is an environmentally stable phase (Lumpkin, 2001) with metal capture potential (Ivanets et al., 2021). However, our study revealed some signs of weathering on the Fe oxides and the hematite (Fig. 3). We hypothesize that these phases dissolved during the

initial stage of incubation because acidic conditions, such as those applied at the beginning of the experiment, weakened iron–oxygen bonds, leading to iron dissolution (Schwertmann, 1991). In addition, the Fe content in the leachate was negligible at the end of incubation (after 18 months), indicating that it had been captured in secondary phases. Secondary phase formation during slag weathering plays an important environmental role, as such phases can capture metallic elements, thereby limiting their leaching into groundwater, surface waters, and soils (Jarošíková et al., 2017; Yin et al., 2016). Furthermore, the slags aligned to Group II generally consisted of forsterite and magnesiochromite. While forsterite exhibits some pH-buffering capacity, larnite demonstrates markedly higher buffering potential. Consequently, Group II remained within the acidic range even after prolonged exposure to the initially acidic solution (see Fig. 4). Under acidic conditions, forsterite dissolution proceeds through the exchange of metal cations for H^+ ions in solution (Pokrovsky and Schott, 2000; Traynor et al., 2020). Considering Group II, which showed Mg leaching of less than 2.5%, Ca leaching of less than 5%, and Ni leaching of above 85%, there was a clear tendency in the sequence of phases being leached. High leaching of metallic elements indicates preferential dissolution of metal-bearing phases (Fig. 3a and b), whereas Ca leaching higher than that of Mg indicates dissolution of the glass, with slight retardation of forsterite (Fig. 3d and e). Observations derived from solution chemistry are consistent with direct surface analyses, both of which indicate preferential dissolution of metal-bearing phases, ultimately leaving behind a forsterite-barren residue (Fig. 3e). The IS-A slag was found to be the most efficient Ni donor (over 85%) due to the dissolution of pentlandite and micron-to nanometer-sized ferronickel grains. In contrast, chromium (Cr) release (0.3–0.6%) can be attributed to the dissolution of magnesiochromite in the IS-A, IS-B, and IS-C slags. However, the leaching behavior of Cr and Ni differed significantly due to the lower solubility of magnesiochromite compared to ferronickel (Garnier et al., 2008). It is also important to note that magnesiochromite can be retained within silicate crystals and that its release may therefore depend on the solubility of the silicates. Given the low solubility of the silicate phases, magnesiochromite may have remained trapped within the silicates without dissolving, or it may have been released as

nanosized particles (Bolaños-Benítez et al., 2018).

To substantiate the observations of phase susceptibility to dissolution, we used elemental molar ratios as a quantitative tool to identify the preferential release of elements. This was achieved by comparing the leaching observed in experiments with the dissolution calculated theoretically based on the slag's bulk composition. As shown in Fig. 6, the Ca/Mg molar ratio revealed preferential leaching of Ca, confirming that the dissolution of glass in Group II slags proceeded faster than that of forsterite. The pronounced preference for Ca release in CS and MS slags provides further evidence of larnite being a Ca donor. The preferential release of Ca rather than Al suggests the dissolution of glass, with a clear preference for Ca leaching. Such behavior in glass is known to be due to the chemical bonding of these elements in glass, whereby the Ca network modifier ion is less strongly bound than the Al ion, resulting in the former element being released more easily (Melcher and Schreiner, 2006). Similarly, the molar ratios calculated for the metallic elements also showed a clear preference for the leaching of metal-bearing phases, particularly in the case of electric furnace slags as Ni donors, through intermetallic phase dissolution (Fig. 6). Our results are consistent with previous studies evaluating contaminant leaching from Ni-slugs (Ettler et al., 2016). Conversely, Kierczak et al. (2009) found that Ni-slugs were less reactive due to their presence in alkaline soil conditions, which is consistent with the observations for Group I slags, in which residence in slightly alkaline conditions resulted in lower leaching than that observed for Group II.

4.2. Validity of long-term quiescent flow leaching for the assessment of environmental risk

This study shows that using a combined geochemical approach involving solution chemistry analysis, direct surface observations, and quantitative tools, such as molar ratios and enhancement factors, provides valuable insights into the environmental stability of slag materials.

Furthermore, apart from a few studies on long-term slag leaching (Ettler and Johan, 2014; Ettler and Mihaljević, 2023; Potysz, 2023; Potysz et al., 2016), very little is still known about their long-term behavior. The analysis of short-term (Đorđević et al., 2024) and

long-term experiments presented in this study has certain limitations in terms of quantitative comparison. First, agitation was implemented in the short-term experiments (Đorđević et al., 2024), but not in the long-term experiments. This is an obvious reason why the leaching rates were higher in the short-term experiments (Al-Abed et al., 2006; Townsend et al., 2003). However, quiescent flow is a more accurate reflection of the real scenario and better simulates field conditions. Quiescent flow may occur when slags are disposed of at sites with clay-rich soils. Clay minerals, particularly those of the 2:1 type, are characterized by a high water sorption capacity (Wang et al., 2020). As a result, clay-rich soils overlying slags may promote water stagnation within the slag heap (in our study area, the clay and silt fractions in soils reached up to 30% and 46%, respectively; data not published). Importantly, the environmental impacts of water runoff enriched in metallic elements, as well as water retention within slag heaps, remain poorly documented, despite a recent increase in research (Souter and Watmough, 2017). Since the same samples were used in both experiments, we calculated the factors showing how the potential environmental risk increased or decreased over time (see Fig. 7). The results demonstrated that long-term weathering may result in an increased release of (Cr), as most of the slags showed an enhancement factor of up to 21.5 over time. Interestingly, this effect was more pronounced for slags belonging to Group I (MS and CS), which were generally less susceptible to dissolution than those belonging to Group II. For other elements, the factors were 1.2 (Ni), 1.9 (Zn), 35.6 (Cu), 1.4 (Co), and 1.2 (As). However, this tendency varied for specific slags, with the most notable enhancement occurring for most elements in IS-A slag. MS slag and CS slag, on the other hand, were found to be more stable (Fig. 7). As mentioned above, the greater stability of these two samples compared to the others may be due to their greater buffering potential, resulting in a higher pH in the leachates. It is of high importance to note that in the long term, Ni release did not show significant enrichment in the leachate. However, given Ni values of up to 660 mg kg⁻¹ (IS-A), concern should be raised about the environmental stability of these materials, even if most elements do not exceed regulatory limits.

A previous study by Đorđević et al. (2024) demonstrated the occurrence of secondary phases on slags disposed of at the dump. These

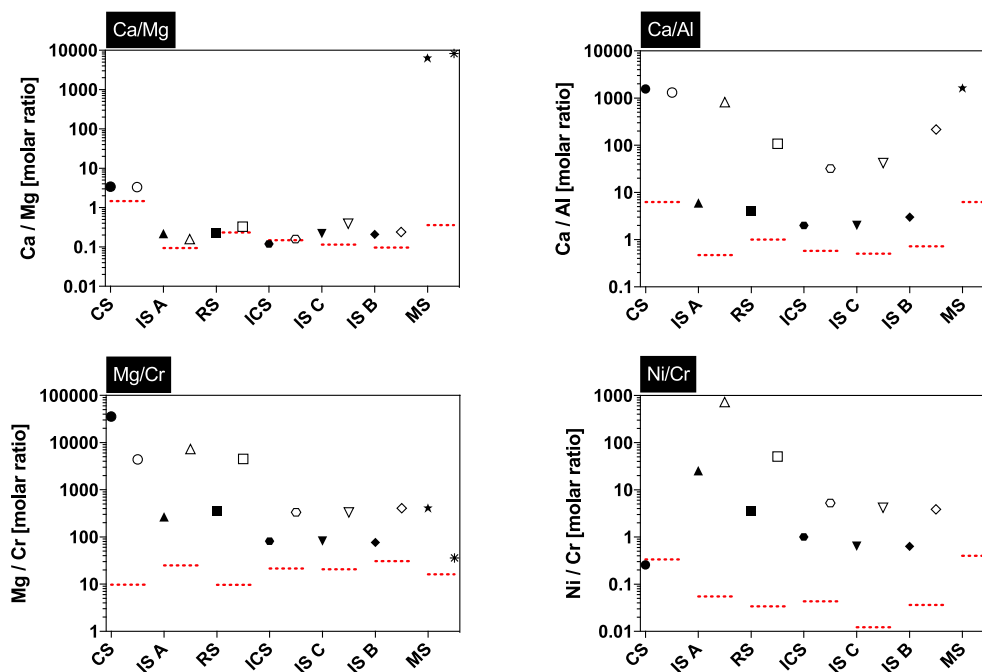


Fig. 6. Molar ratios of elements. Dashed lines represent congruent dissolution values calculated based on bulk slag chemistry. Data points above the lines indicate preferential leaching of the element in the numerator of the ratio, while points below the lines indicate preferential leaching of the element in the denominator. Black-filled data points represent leaching with solution at an initial pH of 2.88, whereas white data points represent leaching with solution at an initial pH of 4.93.

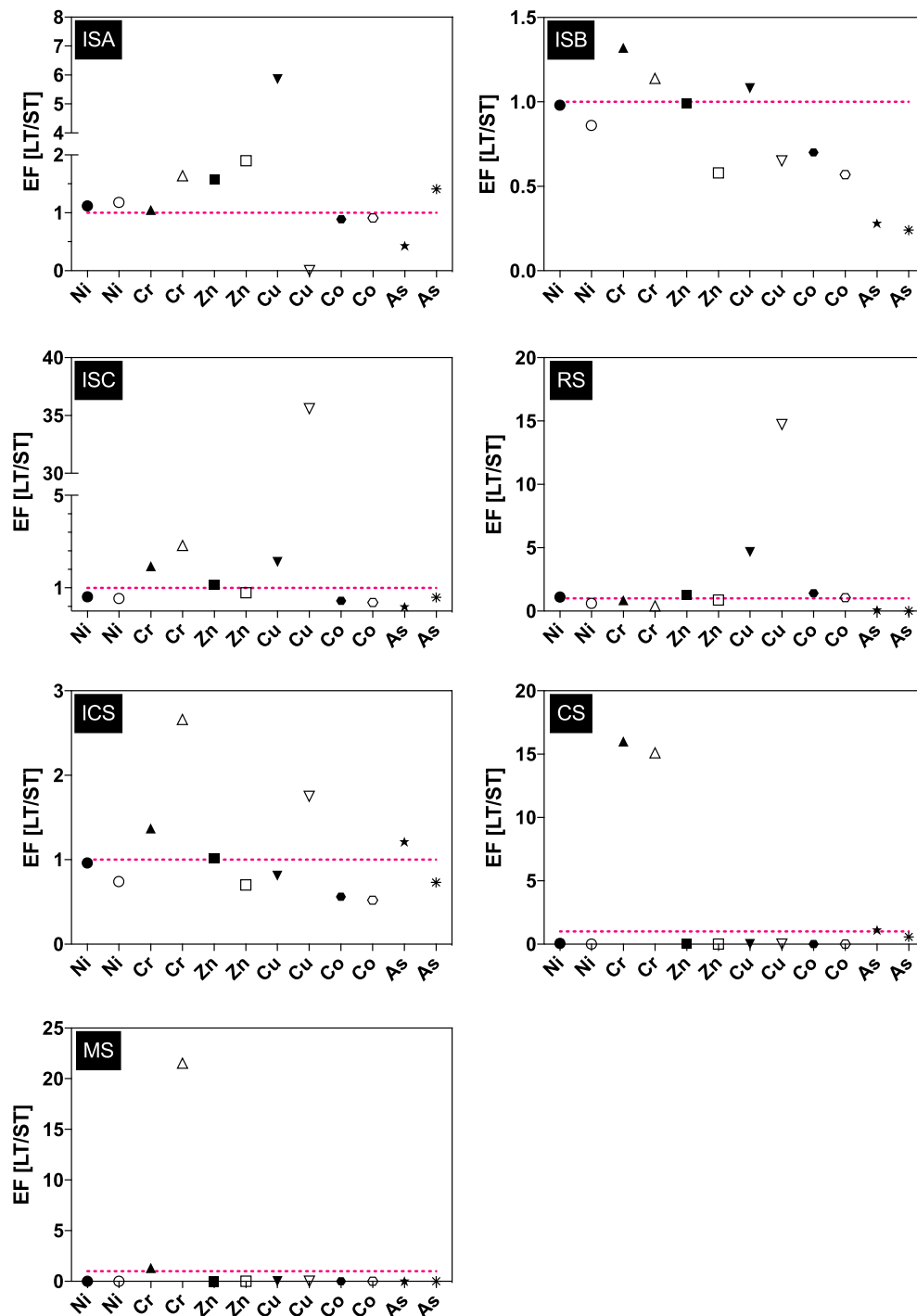


Fig. 7. Calculated long-term enhancement factor relative to leaching recorded under short-term conditions (Dordević et al., 2024). The dashed lines represent a value of 1, indicating equal leaching between the short-term and long-term experiments. Data points above the lines suggest increased leaching of specific elements over the long term, while points below the lines indicate potential precipitation of secondary phases during prolonged exposure.

phases predominantly included iron oxyhydroxides and sulfate weathering products, such as gypsum ($\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$), anhydrite (CaSO_4), syngenite ($\text{K}_2\text{Ca}(\text{SO}_4)_2 \cdot \text{H}_2\text{O}$), and apthitalite ($(\text{K},\text{Na})_3\text{Na}(\text{SO}_4)_2$). The dominance of iron oxyhydroxides among the secondary phases indicates that oxidative weathering had taken place on site. Moreover, as these phases typically form under wet or periodically wet conditions, their presence suggests that solute migration through the dump had occurred. This observation supports the use of incubation experiments under quiescent flow to simulate the progression of weathering experimentally.

Gypsum forms under conditions of high water activity, in contrast to anhydrite, which preferentially forms under dry conditions and may result from gypsum dehydration. Syngenite forms under intermediate water activity conditions and requires the presence of Ca^{2+} and K^+ (Klimchouk, 1996). Thus, the occurrence of syngenite and apthitalite may indicate the weathering of potassium- and calcium-bearing minerals. This phase was identified in CS slag exposed to natural weathering conditions. Consistent with this observation, the leaching results from the present study show that CS slag is among the most prominent slags that release Ca during contact with a leaching solution.

Taken together, the field observations and experimental results provide important insight into the progression of slag weathering and confirm that the applied weathering simulation effectively reflects field conditions to a high degree.

5. Conclusions

A comprehensive evaluation of the environmental impact of the disposal of slags from nickel-smelting processes was conducted through a long-term stagnation experiment. Based on the observed leaching behaviors, the slags were categorized into two groups: Group I exhibited relatively high buffering capacity, while Group II had lower buffering potential. This classification correlates closely with the dominant phase composition: larnite for Group I (CS and MS) and forsterite for Group II.

Group I slags released notably higher amounts of major elements, with Ca leaching exceeding 20%. In contrast, Group II slags showed increased mobilization of environmentally critical metallic elements, such as Cr, Ni, and Co. The weathering sequence established for these materials was as follows: Group I: larnite > glass > metal-bearing phases; Group II: metal-bearing phases > glass > forsterite.

Comparative analysis of short-term and long-term leaching tests revealed a significant decrease in elemental concentration over time, suggesting that precipitation of secondary phases under stagnant conditions is more significant than in dynamic systems. Therefore, the findings of this long-term experiment provide valuable insight into slag dissolution mechanisms and more accurately reflect the geochemical processes likely to occur under natural field conditions. Given the amount of metal (e.g. Ni) trapped in the slag materials, future perspectives of this study include evaluating a hydrometallurgical approach for metal recovery.

CRediT authorship contribution statement

Anna Potysz: Conceptualization, Data curation, Formal analysis, Funding acquisition, Investigation, Methodology, Project administration, Visualization, Writing – original draft, Writing – review & editing. **Tamara Đorđević:** Conceptualization, Data curation, Formal analysis, Funding acquisition, Investigation, Methodology, Project administration, Visualization, Writing – original draft, Writing – review & editing. **Artur Pędziwiatr:** Methodology. **Wencke Wegner:** Methodology. **Sabine Schwarz:** Methodology. **Goran Tasev:** Formal analysis. **Ivan Boev:** Resources.

Declaration of competing interest

The authors declare the following financial interests/personal relationships which may be considered as potential competing interests.

Acknowledgments

Cooperation between the TU Wien and the University of Wrocław is supported by a bilateral mobility project granted by Austrian Wissenschaftlich-Technische Zusammenarbeit (WTZ) under project agreement PL 14/2024 and Polish National Agency for Academic Exchange (NAWA) under project agreement BPN/BAT/2023/1/00021. The University of Wrocław is acknowledged for providing financial support for the research visit of Anna Potysz (01.04.2025–02.07.2025) to the University of Vienna in the frame of the Excellence Initiative – Research University program (IDUB). Experimental work (mineralogical characterization and weathering experiment) was financially supported by the National Science Center (NCN) in Poland in the frame of the OPUS25 program under grant agreement 2023/49/B/ST10/01227. The leachate analysis using ICP-OES was supported by the Own Science Development Fund of the Warsaw University of Life Sciences. The authors would like to thank the editors and reviewers for their time dedicated to evaluating our work.

Appendix A. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.apgeochem.2026.106757>.

Data availability

Data will be made available on request.

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