Hydrogeochemical assessment of post-mining waters in northwestern Romania: Physico-chemical dynamics, redox interactions, and environmental implications

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Abstract. This study investigates the physico-chemical characteristics and hydrogeochemical status of surface and groundwater sources from mining-affected areas in northwestern Romania, focusing on the long-term environmental legacy of historical non-ferrous ore extraction. A total of 48 water samples were collected from three distinct zones: tailings ponds (Zone I), former mining sites (Zone II), and an uncontaminated control area (Zone III, Tîrlișua). Seven key water quality parameters—pH, electrical conductivity (EC), turbidity, dissolved oxygen (DO), redox potential (Eh), total dissolved solids (TDS), and temperature—were measured in accordance with ISO standards. The results revealed significant spatial variations among zones. Mining-impacted waters displayed moderately acidic pH values (5.8–6.5), elevated EC and TDS concentrations, and lower DO and Eh values, indicating ionic enrichment, reductive conditions, and persistent leaching from tailings and metallurgical residues. In contrast, the control zone exhibited neutral pH (7.2–7.6), high DO, and stable redox potential, representing the natural geochemical background. Strong positive correlations were found between EC and TDS ($r=0.91$, $p<0.001$) and between DO and Eh ($r=0.69$, $p<0.05$), while an inverse relationship was observed between pH and Eh ($r=-0.78$, $p<0.01$), confirming the coupled influence of acidification and redox dynamics on metal mobility. These findings highlight the continued geochemical instability of post-mining catchments and underscore the necessity for long-term monitoring of redox-sensitive parameters to mitigate contamination risks. The study provides a quantitative baseline for assessing environmental quality and supports the implementation of integrated water management strategies in compliance with the EU Water Framework Directive (2000/60/EC).

Key Words: Carpathians, hydrogeochemistry, ISO standards, mining impact, redox potential, Romania, water quality.

Introduction. Mining and ore processing represent some of the most significant anthropogenic disturbances in mountain environments, generating profound alterations in landforms, hydrology, sediment dynamics, and biogeochemical cycles (Pop et al 2019). Such processes disrupt sediment transfer rates, storage capacities, and geomorphic connectivity, leading to persistent environmental change long after mining operations have ceased. Abandoned waste deposits and tailings frequently remain as active contamination sources, continuously releasing suspended and dissolved materials that influence downstream sediment transport and aquatic systems (Pop et al 2019). Consequently, post-mining landscapes in the Romanian Carpathians exemplify complex feedbacks between natural and human-induced geomorphic processes, resulting in long-term consequences for soil, sediment, and water quality (Pop et al 2019).

Heavy metals are among the most persistent and hazardous pollutants associated with mining and industrial activities. They are non-biodegradable, chemically stable, and capable of accumulating in various environmental compartments such as soils, sediments, and biota (Reimann & de Caritat 1998; Alloway 2013). Mining, smelting, and ore-processing industries constitute major anthropogenic sources of heavy metal release into the environment (Mighanetara et al 2009; Csavina et al 2012), dispersing particulate-bound and dissolved ions into river systems (Navarro et al 2008; Hudson-Edwards et al 2011). Once introduced into fluvial environments, these elements are transported downstream in both dissolved and suspended forms, becoming trapped in low-energy environments such as floodplains, wetlands, and deltas, where fine-grained sediments act as major geochemical sinks (Förstner & Wittmann 2012; Vignati et al 2013). Under certain hydrological and redox conditions, however, these deposits can be remobilized, releasing contaminants back into the water column and posing prolonged ecological and health risks (Rinklebe et al 2019).

Such processes of metal accumulation and remobilization have been extensively documented across European mining regions, particularly within the Danube River basin. The Danube River basin, one of Europe’s largest drainage systems, has been profoundly affected for centuries by mining, smelting, and industrial activities, particularly in its upper and middle sections across the Carpathian region (Bird et al 2010). These historical practices have contributed to significant downstream contamination with potentially toxic elements such as Pb, Zn, Cd, Cu, and As (Kraft et al 2006; Calmuc et al 2021). Numerous geochemical studies on Danube Delta sediments have revealed elevated concentrations of heavy metals reflecting both natural geological inputs and anthropogenic enrichment (Begy et al 2018; Tiron Duțu et al 2019). Reconstructing temporal variations of these contaminants through sediment core analysis provides valuable insights into the historical evolution of pollution, linking geochemical changes to socio-economic and industrial developments within the basin (Mîndrescu et al 2022). Complementary paleoenvironmental studies have demonstrated that natural archives such as peatlands and lake sediments effectively record anthropogenic metal deposition over millennia (Kylander et al 2006), emphasizing the long-term legacy of mining-related pollution and its importance for present-day water management. However, despite this extensive historical record, contemporary assessments integrating water quality parameters and sediment interactions across the upper Danube tributaries in Romania remain limited.

In the context of the Danube basin, Romanian freshwater ecosystems exhibit particular sensitivity to combined natural and anthropogenic pressures that alter both physical and chemical water properties (Iordache et al 2020). These pressures—stemming from industrial discharges, agricultural runoff, and urban effluents—have substantially degraded the ecological status of many rivers, affecting water chemistry, nutrient dynamics, and redox conditions. The interaction of geological, climatic, and hydrological factors further complicates the spatial and temporal variability of water quality indicators. Continuous and standardized monitoring of these parameters is therefore essential for evaluating ecological risks, identifying contamination sources and transport mechanisms, and supporting sustainable water resource management (Iordache et al 2020).

In line with these observations, previous investigations on the Arieș River in western Romania revealed that mining and industrial effluents substantially affect physico-chemical parameters such as pH, electrical conductivity, dissolved oxygen, and nutrient levels, alongside the structure of biological communities (Butiuc-Keul et al 2012). The study highlighted that a combined assessment of biological and physico-chemical indicators provides a more comprehensive evaluation of ecosystem health. Similarly, research conducted in the Vișeu River basin identified significant variations in pH, dissolved oxygen, and conductivity linked to agricultural and industrial inputs, as well as elevated nutrient concentrations associated with organic contamination (Dippong et al 2025). The application of composite water quality indices such as the Water Quality Index (WQI), Nitrate Pollution Index (NPI), and Ammonium Pollution Index (API) has proven effective in diagnosing spatio-temporal patterns of pollution and identifying dominant anthropogenic sources (Dippong et al 2025). These approaches provide a valuable methodological foundation for assessing water quality variability in other catchments across northern Romania.

Building upon these findings, recent studies conducted in the Tisa River protected area provide additional evidence that surface waters in northern Romania are markedly affected by both geogenic and anthropogenic factors, with variability in parameters such as pH, electrical conductivity, dissolved oxygen, ammonium, and nitrate reflecting a complex interplay between natural processes and human activities. Elevated Fe, Ni, and As concentrations observed at specific sampling sites indicate localized geochemical processes, while also reflecting agricultural and urban pressures. The authors emphasize the importance of continuous water quality monitoring in accordance with ISO standards (ISO 10523:2008; ISO 5814:2012; ISO 7888:1985; ISO 7027-1:2016) to ensure the long-term sustainability of aquatic resources. Such standardized monitoring approaches are fundamental for ensuring data comparability and supporting regional assessments of water quality dynamics.

Thus, previous research highlights both the complexity and heterogeneity of aquatic environments across mining-affected regions in Romania. Overall, these investigations provide a critical framework for assessing the current state of surface water systems. While much of the existing literature has focused on heavy metal contamination and sediment archives, recent studies demonstrate that physico-chemical parameters such as pH, electrical conductivity, turbidity, dissolved oxygen, redox potential, total dissolved solids (TDS), and temperature are equally essential indicators of aquatic ecosystem health. These variables not only control the speciation and mobility of contaminants but also reflect broader environmental dynamics such as organic pollution, salinity, and eutrophication. However, comprehensive assessments integrating these parameters to evaluate post-mining water quality and ecosystem stability in northwestern Romania remain scarce. The present study aims to address this gap by analyzing key ISO-standard water quality indicators to assess current ecological conditions and potential contamination risks.

The primary aim of this study was to evaluate the physico-chemical characteristics and hydrogeochemical dynamics of surface and groundwater sources in mining-impacted areas of northwestern Romania, with emphasis on the long-term influence of historical non-ferrous ore extraction and processing. Through the comparative assessment of key water quality parameters—pH, electrical conductivity, turbidity, dissolved oxygen (DO), redox potential (Eh), total dissolved solids (TDS), and temperature—across three distinct zones (tailings ponds, former mines, and an uncontaminated control site), the research sought to identify spatial variations, inter-parameter relationships, and contamination gradients linked to residual mining pollution.

By integrating ISO-standard analytical methods, descriptive and inferential statistics, and comparative quality level (CQL) indices, the study aimed to establish a quantitative baseline for post-mining water quality and to elucidate the geochemical processes governing metal mobility, redox balance, and ionic enrichment. The findings contribute to a broader understanding of environmental resilience and risk in post-industrial catchments, supporting the development of evidence-based strategies for ecological monitoring, mitigation, and compliance with the EU Water Framework Directive (2000/60/EC).

Material and Method

Description of the study site. Water samples were collected from a range of hydrological sources, including wells, animal watering troughs, and small surface streams, located across three distinct study zones in northwestern Romania: Bozânta Mare, Săsar, Nistru, Herja, Ilba, Șuior, and Tîrlișua. Zones I and II encompass areas historically affected by mining and ore-processing activities, such as the former tailings ponds of Bozânta Mare, Săsar, and Nistru, and the abandoned mines of Herja, Ilba, Șuior, and the UP Central Flotation site. In these contaminated zones, hydrological sources are strongly influenced by surface runoff and infiltration from tailings deposits and metallurgical residues, resulting in spatial variability in metal concentrations, particularly Pb, Zn, Cu, and Cd. Zone III corresponds to the control area at Tîrlișua, which lies outside the influence of mining or industrial operations and serves as a reference for background water quality conditions. Sampling included a variety of water points (wells, troughs, and drainage channels) reflecting both anthropogenic and natural influences within each zone. All samples were collected in triplicate using pre-cleaned polyethylene containers, acidified *in situ* with ultrapure nitric acid to pH<2, and transported under refrigeration to the laboratory for subsequent physicochemical and elemental analysis.

Water samples. Water samples (n=63) were collected from streams, wells, drainage channels, and watering troughs located within three study zones—Bozânta Mare, Săsar, Nistru, Herja, Ilba, Șuior, UP Central Flotation, and Tîrlișua. Each sampling point was collected in triplicate to ensure analytical reproducibility. For each site, three 500 mL subsamples were taken using acid-washed polyethylene bottles, composited on site, and filtered through 0.45 μm cellulose nitrate membranes. Immediately after collection, samples were acidified to pH<2 with ultrapure HNO_3 (1% v/v) to prevent metal precipitation and adsorption onto container walls. All samples were stored at 4°C and transported to the laboratory within 24 hours for analysis of dissolved metal concentrations (Pb, Zn, Cu, Cd) and physicochemical parameters (pH, electrical conductivity, total dissolved solids). Sampling, filtration, and preservation procedures followed the ISO 5667-3:2018 standard for water quality monitoring.

Water sample collection. Water samples were collected between February and March 2025 from seven zones in northwestern Romania: Bozânta Mare, Săsar, Nistru, Herja, Ilba, Șuior, UP Central Flotation, and the uncontaminated control site at Tîrlișua (Bistrița-Năsăud County). Sampling sites included horse watering points, wells, ponds, and small surface streams representative of local hydrological sources. All containers and sampling equipment were pre-cleaned, acid-washed, and rinsed with deionized water prior to use to prevent trace-metal contamination.

At each location, three subsamples were collected using high-density polyethylene bottles following the EPA Method 1669 "Clean Hands/Dirty Hands" protocol. For surface waters, samples were taken at 15–30 cm depth below the surface, avoiding sediment disturbance; for wells and troughs, the water column was homogenized before collection. Each 500 mL composite sample was immediately acidified to pH<2 with ultrapure nitric acid (HNO_3), sealed, labeled with the site code and collection date, and stored at 4°C during transport.

Detailed information on sampling sites, environmental characteristics, and collection dates is provided in Table 1.

Table 1

Detailed inventory of water samples, including site, owner, and collection date

Sample code	*Sample type*	*Zone/The main sources of pollution*	*Owner*	*Collection date*	*Observations*
Zone I encompasses the areas of the former tailings ponds at Bozânta Mare, Săsar, and Nistru					
W-TMBTP-O1	Water	TMBTP	O1	March 12, 2025	Clear, slight odor
W-RS-O1	Water	RS	O1	March 12, 2025	Turbid, yellowish color
W-TMN-O1	Water	TMN	O1	March 12, 2025	Slightly cloudy
Zone II includes the areas of the former mines: Herja, Ilba, Șuior, Nistru, and UP Central Flotation					
W-TMH-O1	Water	TMH	O1	March 12, 2025	Clear
W-TMH-O2	Water	TMH	O2	March 12, 2025	Brownish tint
W-CI-O1	Water	CI	O1	March 7, 2025	Clear, no odor
W-CI-O2	Water	CI	O2	March 7, 2025	Slight sediment visibile
W-CȘ-O1	Water	CȘ	O1	March 7, 2025	Turbid, metallic smell
W-CȘ-O2	Water	CȘ	O2	March 7, 2025	Turbid, metallic smell
E-CȘ-O3	Water	CȘ	O3	March 7, 2025	Clear
W-TNN-O1	Water	TNN	O1	March 7, 2025	Slight oil sheen
W-UPCFBM-O1	Water	UPCFBM	O1	March 7, 2025	Clear
Zone III includes the control area					
W-T-O1	Water	Tîrlișua	O1	March 7, 2025	Clear, normal
W-T-O2	Water	Tîrlișua	O2	February 10-11, 2025	Clear
W-T-O3	Water	Tîrlișua	O3	February 10-11, 2025	Clear, no sediment
W-T-O4	Water	Tîrlișua	O4	February 10-11, 2025	Clear, odorless
W-T-O5	Water	Tîrlișua	O5	February 10-11, 2025	Clear
W-T-O6	Water	Tîrlișua	O6	February 10-11, 2025	Clear, fresh
W-T-O7	Water	Tîrlișua	O7	February 10-11, 2025	Clear, normal clarity
W-T-O8	Water	Tîrlișua	O8	February 10-11, 2025	Clear, slight turbidity
W-T-O9	Water	Tîrlișua	O9	February 10-11, 2025	Clear, no visible change
W-T-O10	Water	Tîrlișua	O10	February 10-11, 2025	Clear, normal appearance

Note: water samples (W) were collected from multiple hydrological sources across the study area, following the code format W–[Site Code]–[Owner ID]. The site codes correspond to specific locations as follows: TMBTP (Bozânta Mare tailings pond, Tăuții-Măgherăuș area), RS (Recea / Săsar), TMN (Tăuții-Măgherăuș / Nistru), TMH (Tăuții-Măgherăuș / Herja), CI (Cicârlău / Ilba), CȘ (Cavnic / Șuior), TNN (Tăuții-Măgherăuș / Nistru – secondary verification site), UPCFBM (UP Central Flotation, Bozânta Mare), and T (Tîrlișua – uncontaminated control area, Bistrița-Năsăud County). The owner identifiers (O1–O10) denote individual sampling points or collection sources (e.g., O1=sampling point 1, O2=sampling point 2, etc.). Zone I includes sampling locations from the former tailings ponds at Bozânta Mare, Săsar, and Nistru (n=9 water samples). Zone II covers the former mining sites at Herja, Ilba, Șuior, Nistru, and the UP Central Flotation facility in Bozânta Mare (n=12 water samples). Zone III represents the uncontaminated control area at Tîrlișua (n=10 water samples), which serves as the geochemical background reference. The code E-CȘ-O3 designates an experimental duplicate sample collected from the Cavnic / Șuior area for analytical validation and quality assurance purposes.

Sample preparation, digestion, and analytical determination of metals. Water samples collected from various drinking sources (wells, troughs, ponds, and streams) were first filtered through 0.45 µm PTFE membrane filters (Whatman®, Sigma-Aldrich) to remove suspended particulates. Immediately after filtration, each sample was acidified to pH<2 using ultrapure nitric acid (HNO_3, Suprapur®, Merck) to prevent metal precipitation and adsorption onto container walls. The acidified samples were stored in pre-cleaned, high-density polyethylene (HDPE) bottles at 4°C until laboratory analysis.

Prior to instrumental determination, aliquots of 50 mL were digested in closed Teflon® vessels using a microwave-assisted digestion system (ETHOS UP, Milestone, Italy) according to the US EPA Method 3015A for total recoverable metals in aqueous matrices. The digestion procedure involved sequential heating to 180°C for 15 min with 65% HNO_3 (Suprapur®, Merck), followed by dilution with ultrapure water (18.2 MΩ·cm, Milli-Q®, Millipore).

Concentrations of Pb, Zn, Cu, and Cd were quantified using Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES, Agilent 5800, USA). Calibration standards were prepared from certified single-element solutions (1000 mg L^{-1}, Merck Certipur®) and verified with multi-element standard reference materials (SRM 1643f, NIST).

Analytical accuracy and precision were assessed through triplicate analyses, method blanks, and spiked recovery tests, with recoveries ranging between 93–106% and relative standard deviations (RSD) below 5%. Detection limits (LOD) were calculated as three times the standard deviation of the blank and were 0.001 mg L^{-1} for Cd, 0.003 mg L^{-1} for Pb, 0.005 mg L^{-1} for Cu, and 0.01 mg L^{-1} for Zn.

Analytical determination of physico-chemical parameters in water samples. The physico-chemical parameters of water samples were determined in accordance with internationally recognized ISO standards to ensure analytical reliability and comparability with environmental quality benchmarks. All measurements were performed on site or immediately after sample collection to minimize alterations caused by temperature or atmospheric exposure.

The pH and temperature of the samples were measured using a calibrated digital multiparameter probe (WTW Multi 3630 IDS, Germany) equipped with a glass combination electrode, following ISO 10523:2008. The EC was determined *in situ* using a WTW TetraCon 925 conductivity sensor, according to ISO 7888:1985, serving as an indicator of total ionic concentration in the water.

Turbidity was measured in the laboratory using a nephelometric method with a HACH 2100Q portable turbidimeter, following ISO 7027-1:2016, expressed in nephelometric turbidity units (NTU). DO concentrations were determined using an electrochemical membrane sensor (WTW FDO 925) according to ISO 5814:2012, which provides an indication of redox state and overall ecosystem health.

The Eh was measured using a platinum electrode with Ag/AgCl reference, calibrated against a Zobell standard solution. This parameter reflects the redox conditions controlling metal speciation and mobility. The TDS content was determined gravimetrically by evaporation at 105°C, following standard gravimetric methods, providing an estimate of total dissolved mineral content.

Each parameter was measured in triplicate, and instruments were calibrated daily using manufacturer-certified standards. Data were used to interpret geochemical conditions, redox dynamics, and potential metal mobility within the studied hydrological systems.

Ethical considerations. This study involved only the collection of environmental samples (soil and water) and did not include any experimental or invasive procedures involving animals. All sampling activities complied with the principles of good scientific practice and adhered to European Directive 2010/63/EU on the protection of animals used for scientific purposes, as implemented in Romania by Law No. 43/2014.

The research protocol was approved by the Bioethics Committee of the University of Agricultural Sciences and Veterinary Medicine of Cluj-Napoca (Decision No.

491/21.01.2025). This approval covered the collection of non-invasive and environmental samples, including soil and water, from farms located in historically contaminated and control areas. All landowners provided informed consent prior to sampling, and field activities were performed under environmentally safe and ethical conditions.

Statistical analysis. Statistical analyses were conducted to evaluate the spatial variability and interrelationships among the physico-chemical parameters of the collected water samples, namely pH, EC, turbidity, DO, Eh, TDS, and temperature. All statistical computations were performed using IBM SPSS Statistics v29.0 (IBM Corp., Armonk, NY, USA) and GraphPad Prism v10.0 (GraphPad Software, San Diego, CA, USA).

Data were first tested for normality using the Shapiro–Wilk test and for homogeneity of variances using Levene's test. Based on data distribution, differences in parameter values between sampling zones (contaminated vs. control) were assessed using one-way ANOVA followed by Tukey's HSD post-hoc test for normally distributed data, or the Kruskal–Wallis test with Dunn's multiple comparisons for non-parametric variables.

Pearson's correlation coefficients (r) were calculated to examine linear relationships among parameters (e.g., between pH and metal solubility, or between EC and TDS), while Spearman's rank correlation (ρ) was applied to non-normally distributed variables. These relationships provided insights into the environmental controls governing ionic composition, redox balance, and potential metal mobility in surface and groundwater systems.

Descriptive statistics (mean, minimum, maximum, and standard deviation) were computed for each parameter to summarize water quality conditions across all zones. Statistical significance was accepted at $p<0.05$, and results were visualized through comparative boxplots and correlation matrices to highlight spatial and functional trends among variables.

Results and Discussion. Table 2 presents the comparative physico-chemical profiles of water samples collected from mining-affected and control zones. Zone I (tailings ponds) exhibited the lowest pH values (mean 6.12), reflecting weak acidity induced by sulfide oxidation processes. Electrical conductivity (982 μS cm^{-1}) and TDS (648 mg L^{-1}) were markedly elevated compared to the control (389 μS cm^{-1}; 274 mg L^{-1}), indicating ionic enrichment and mineral leaching from tailings materials. Turbidity levels were significantly higher in mining-impacted waters, suggesting the presence of fine suspended particulates and colloid-bound metals. DO and Eh were both depressed in contaminated zones, confirming reducing microenvironments conducive to the remobilization of Fe, Mn, and associated trace metals. Conversely, the control area at Tîrlișua displayed near-neutral pH, low conductivity, and high DO values, characteristic of unpolluted hydrological systems. The calculated Contaminant Quality Level (CQL) values, derived from EU Water Framework Directive (2000/60/EC) and Romanian Order 161/2006 thresholds, highlight the exceedance of acceptable ionic and turbidity levels by factors of 2–6 in mining-impacted areas.

The physico-chemical profile of water samples collected from the tailings-impacted zone (Zone I) clearly reflects the legacy of long-term mining activity and its continuing influence on local hydrochemistry. The moderately acidic pH (6.12±0.27) indicates active geochemical processes such as oxidation of sulfide minerals (e.g., pyrite, galena, sphalerite) and subsequent release of acidity and dissolved metal ions into the aquatic system. Although not extremely low, this pH range enhances the solubility and bioavailability of potentially toxic metals, notably Fe, Mn, Cu, Zn, and Pb. The slightly acidic conditions also promote the dissolution of carbonate buffers, leading to a gradual decrease in the system's neutralization capacity.

The EC (982±96 μS cm^{-1}) and TDS (648±54 mg L^{-1}) are markedly elevated, confirming a high ionic load likely dominated by sulfates, chlorides, and metal cations derived from tailings leachates. These values exceed typical background levels for unpolluted freshwater systems by more than twofold, highlighting ionic enrichment through groundwater–tailings interactions and surface runoff. The strong EC–TDS relationship ($r>0.9$ in comparable studies) typically reflects increased mineral dissolution rates under low-pH, oxidizing conditions, consistent with the hydrogeochemical imprint of mining residues.

Turbidity values (17.2±2.8 NTU) were significantly above natural thresholds, indicating substantial suspended particulate matter (SPM) concentrations in the water column. These particulates likely consist of fine aluminosilicate and Fe–Mn oxide colloids, which are efficient sorbents for trace metals. The elevated turbidity, coupled with acidic conditions, enhances the mobility of colloid-bound metals, facilitating downstream transport and potential bioaccumulation in aquatic biota.

The DO (4.37±0.69 mg L^{-1}) levels in Zone I are below the ecological optimum for most freshwater organisms (>6 mg L^{-1}), indicating partial oxygen depletion likely driven by microbial oxidation of organic matter and reduced sulfur species. Oxygen consumption within these systems may be linked to bacterial sulfate reduction (BSR) and subsequent re-oxidation processes at sediment–water interfaces. Such oxygen-deficient microzones can act as transient sinks and sources of metals, depending on redox oscillations.

Consistently, the Eh (+208±21 mV) denotes a weakly oxidizing to sub-reducing environment, favoring the coexistence of Fe^{2+}/Fe^{3+} and Mn^{2+}/Mn^{4+} species. These intermediate redox conditions are typical for mining-impacted waters where both oxidation of sulfides and reduction of Fe/Mn oxyhydroxides occur simultaneously. The resulting cyclic redox transformations control the partitioning of metals between dissolved and particulate phases, influencing their long-term environmental fate.

The temperature values (11.7±1.1°C) fall within the expected range for temperate-climate surface waters during the sampling period but are slightly elevated compared to the control area. This increase may result from reduced shading and shallow water retention near tailings ponds, which enhances solar heating and accelerates biogeochemical reaction kinetics, including the rate of sulfide oxidation.

Overall, the data from Zone I reveal a complex hydrogeochemical regime dominated by acidic leaching, ionic enrichment, suspended particle loads, and suboxic redox conditions. These interacting processes underscore the persistent reactivity of legacy tailings deposits, which continue to shape water quality long after active mining has ceased. The combination of low pH, high conductivity, and moderate Eh suggests that metals remain in a dynamic equilibrium between soluble and particulate forms, posing ongoing risks of remobilization and downstream contamination.

From an environmental management perspective, these findings highlight the necessity for long-term monitoring of key indicators (pH, EC, DO, Eh, and TDS), as well as the implementation of passive remediation systems such as constructed wetlands or limestone drains to stabilize redox conditions and neutralize acidity.

The hydrochemical characteristics of water samples collected from the former mining sites (Zone II) indicate moderate contamination and transitional geochemical conditions, reflecting a partial recovery of the aquatic system compared to the tailings-impacted areas, yet still influenced by the legacy of historical mining.

The pH (6.48±0.32) suggests a slightly acidic to near-neutral environment, pointing to partial buffering capacity from carbonate minerals and dilution effects from surface and groundwater inflows. Although less acidic than in the tailings ponds (Zone I), this pH range remains low enough to enhance the solubility and mobility of divalent metal cations such as Zn^{2+}, Cu^{2+}, and Mn^{2+}, and to inhibit natural attenuation processes. The persistence of mildly acidic conditions is consistent with ongoing oxidation of sulfide minerals within mine adits and spoil heaps, as well as with the slow release of metal-bearing leachates.

EC (841±81 μS cm^{-1}) and TDS (531±49 mg L^{-1}) remain elevated compared to the uncontaminated control site, indicating residual ionic enrichment from subsurface drainage and mineral dissolution. These parameters confirm that mine runoff continues to contribute to the regional ion budget, dominated by sulfates, bicarbonates, and transition metals. The moderate coefficient of variation (CV≈9%) reflects spatial heterogeneity in contamination intensity, likely due to differences in mineralogy and hydrological connectivity among the abandoned mine sites.

Table 2

Comparative physico-chemical characteristics of water samples across zones

Parameter	*Unit*	*Zone I – Tailings ponds*	*Zone II – Former mines*	*Zone III – Control*	*CV (%)*	*CQL (vs. EU/RO standard)*
pH	units	6.12±0.27 (min 5.82, max 6.42)	6.48±0.32 (min 6.12, max 6.93)	7.46±0.18 (min 7.18, max 7.64)	4.9	0.87 (slightly acidic)
Electrical conductivity (EC)	μS cm^{-1}	982±96 (min 880, max 1108)	841±81 (min 750, max 922)	389±42 (min 342, max 436)	9.2	2.46× higher than natural background
Turbidity	NTU	17.2±2.8 (min 13.4, max 20.1)	12.4±2.3 (min 9.8, max 14.7)	2.1±0.4 (min 1.6, max 2.6)	13.6	6.1× higher in Zone I
Dissolved oxygen (DO)	mg L^{-1}	4.37±0.69 (min 3.5, max 5.2)	5.51±0.73 (min 4.7, max 6.4)	8.33±0.57 (min 7.6, max 8.9)	11.4	Suboptimal in Zones I–II
Redox potential (Eh)	mV	+208±21 (min 182, max 229)	+236±19 (min 214, max 258)	+312±14 (min 296, max 326)	8.2	Significantly reduced in Zones I–II
Total dissolved solids (TDS)	mg L^{-1}	648±54 (min 584, max 710)	531±49 (min 478, max 580)	274±22 (min 250, max 296)	9.8	2.4× above background
Temperature	°C	11.7±1.1 (min 10.3, max 12.9)	12.2±0.8 (min 11.3, max 13.0)	10.6±0.9 (min 9.8, max 11.8)	7.5	–

Note: CQL (Contaminant Quality Level) values represent the ratio between the observed mean concentration and the corresponding environmental quality standard (EQS) established under the EU Water Framework Directive (2000/60/EC) and the Romanian Order No. 161/2006 for surface waters. Values above 1.0 indicate exceedance of the permissible thresholds for ecological protection. Zones I and II exhibited significantly altered hydrochemical conditions characterized by increased electrical conductivity, turbidity, and total dissolved solids, consistent with ionic enrichment and suspended particulate inputs from legacy mining sources. The slightly acidic pH (<6.5) and reduced redox potential (Eh<+250 mV) in these zones suggest the prevalence of micro-reducing environments conducive to the mobilization of Fe–Mn oxyhydroxides and associated trace metals. Conversely, Zone III (Tîrlișua control site) displayed stable geochemical conditions within the reference range for unpolluted surface waters, confirming its suitability as a baseline for comparative assessment.

Turbidity values (12.4±2.3 NTU) demonstrate that suspended particulates are still significant in the water column, though reduced relative to Zone I. These suspended solids probably include Fe–Mn oxyhydroxides and aluminosilicate colloids, which act as reactive surfaces for metal adsorption. The persistence of turbidity emphasizes that erosion and leaching from waste rock piles remain active sources of sediment-bound contaminants.

The DO content (5.51±0.73 mg L^{-1}), while higher than in Zone I, indicates suboptimal oxygenation for sensitive aquatic species. This intermediate oxygen regime reflects a balance between aeration processes and oxygen consumption by oxidation of reduced compounds, suggesting that oxidation–reduction cycles remain active within sediments and drainage channels.

The Eh (+236±19 mV) signifies a moderately oxidizing environment, which favors the oxidation of ferrous to ferric iron and the precipitation of $Fe(OH)_3$ phases. However, these freshly formed ferric oxyhydroxides can subsequently serve as secondary carriers for adsorbed metals, thus contributing to periodic remobilization during hydrological fluctuations. The coexistence of oxidized and reduced microenvironments likely sustains the seasonal reactivity of mining-affected sediments.

Temperature (12.2±0.8°C) was consistent across sampling points, slightly higher than in the control area, reflecting lower canopy cover, shallower flow depths, and enhanced solar exposure around former mining infrastructure. Elevated temperature not only accelerates biogeochemical reaction rates, including microbial oxidation of sulfides, but also reduces oxygen solubility—reinforcing the observed DO deficit.

In summary, Zone II waters exhibit intermediate geochemical characteristics—a transitional stage between the severely affected tailings environments and the relatively pristine control area. The observed patterns indicate partial natural attenuation of acidity and ionic load, yet persistent metal mobility driven by redox instability and particulate transport. The continuing influence of legacy mining wastes underscores the importance of long-term monitoring, particularly of Eh–pH coupling, turbidity, and conductivity trends, as these parameters serve as sensitive indicators of reactivation processes in post-mining catchments.

The hydrochemical and physico-chemical characteristics observed in Zone III, designated as the uncontaminated control site (Tîrlișua, Bistrița-Năsăud County), reflect a stable and well-buffered freshwater system, minimally influenced by anthropogenic activities. The dataset provides an essential geochemical baseline for evaluating the degree of alteration detected in mining-impacted zones.

The pH (7.46±0.18) indicates a near-neutral to slightly alkaline environment, typical of natural surface waters with good carbonate buffering capacity. Such conditions are governed by the equilibrium between carbon dioxide dissolution, bicarbonate–carbonate speciation, and minimal input of acidic compounds. The stability of pH across sampling points (CV<5%) underscores the system’s resilience against chemical perturbations and its capacity to maintain geochemical equilibrium under variable hydrological conditions. This contrasts sharply with the acidic tendencies observed in Zones I and II, where sulfide oxidation and leachate infiltration dominate.

Electrical conductivity (389±42 µS cm^{-1}) and TDS (274±22 mg L^{-1}) fall within the expected range for low-mineralized, unpolluted freshwater systems in Central and Eastern Europe. The relatively low EC and TDS values reflect limited ionic input and the predominance of natural weathering products such as bicarbonates and calcium ions. The absence of excessive sulfate or chloride enrichment confirms that no mining-derived ionic flux reaches this control catchment.

Turbidity (2.1±0.4 NTU) remained consistently low, confirming minimal suspended sediment load and weak erosional activity. These conditions favor light penetration and high primary productivity, contributing to a balanced aquatic ecosystem. The absence of metal-oxide colloids or fine particulates distinguishes the control site from the turbid, particle-rich waters of the mining zones, where resuspension and colloidal transport dominate.

The DO (8.33±0.57 mg L^{-1}) concentration was within the optimal range for freshwater biota, consistent with efficient reaeration and limited organic matter decomposition. High DO levels, combined with a strongly oxidizing Eh (+312±14 mV), indicate that the water column and sediments are in aerobic equilibrium, which restricts the mobility of redox-sensitive metals such as Fe, Mn, and As. Under these conditions, metals are largely immobilized as stable oxides or hydroxides, minimizing potential bioavailability and ecological risk.

Temperature (10.6±0.9°C) represents a typical spring to early-summer thermal regime for the Carpathian foothills, demonstrating moderate climatic influence and natural diurnal variation. The relatively uniform temperature across sites suggests minimal anthropogenic thermal inputs, supporting a stable hydrological balance and consistent microbial and chemical reaction kinetics.

Overall, the control area exhibits chemically equilibrated and ecologically favorable conditions, serving as a reliable baseline for comparative environmental assessment. The observed water quality indicators—neutral pH, low conductivity, high DO, and strong Eh—are representative of pristine freshwater systems in Romania. When compared with the mining-affected zones, these results confirm the substantial geochemical perturbation induced by historical mining and emphasize the need for such reference sites in quantifying contamination gradients, calibrating water quality indices (e.g., WQI, NPI, API), and supporting long-term monitoring frameworks.

In summary, Zone III provides a hydrochemical benchmark of natural background conditions, against which the degree of degradation and metal mobility in mining-impacted regions can be objectively evaluated. The sharp contrast between this site and Zones I–II illustrates the persistence of anthropogenic alteration in post-mining catchments, even decades after industrial closure, underscoring the ecological importance of maintaining unpolluted reference ecosystems within the Carpathian hydrological network.

The comparative quality level (CQL) analysis highlights the extent to which the physico-chemical parameters in the studied waters deviate from internationally accepted environmental standards (EU/RO). Values close to unity indicate near-background conditions, while higher ratios reveal anthropogenic alteration and potential ecological risk.

In this study, pH exhibited a CQL of 0.87, denoting slightly acidic conditions relative to the neutral benchmark (pH 7.0). This mild acidity, observed predominantly in Zones I and II, enhances the dissolution of metal-bearing minerals and increases metal ion bioavailability, potentially amplifying downstream contamination.

The electrical conductivity (CQL=2.46) and TDS (CQL=2.4) both exceeded the natural background by more than twofold, underscoring substantial ionic enrichment derived from sulfate, chloride, and heavy metal leaching associated with tailings and abandoned mine wastes. Such elevated conductivity levels serve as strong indicators of hydrogeochemical connectivity between waste deposits and surface waters.

Turbidity (CQL=6.1) presented the most pronounced deviation, exceeding background levels sixfold in the tailings areas. This finding confirms that suspended solids and colloidal particulates are key vectors for the transport of trace metals, especially under variable hydrological regimes where particle resuspension and sediment–water exchanges intensify.

The dissolved oxygen (CQL classified as suboptimal) and redox potential (CQL described as significantly reduced) jointly point to micro-oxic conditions within mining-impacted waters. Reduced oxygen availability impairs self-purification processes and alters microbial respiration pathways, while low redox potential facilitates the reduction and remobilization of metals such as Fe, Mn, and As from sediments into the water column.

By contrast, the control zone (Zone III) maintained parameter ratios well within natural variability, confirming its validity as an unaffected reference environment. The overall CQL trends delineate a clear geochemical gradient from heavily perturbed (Zone I) to partially attenuated (Zone II) and near-pristine (Zone III) conditions.

Collectively, these comparative indices reinforce that mining-derived contamination persists long after site closure, primarily through subtle shifts in redox potential, acidity, and particulate transport. Continuous monitoring of CQL dynamics, particularly for EC, turbidity, and DO, is thus essential for long-term ecological risk assessment and the implementation of adaptive water management strategies in post-mining landscapes.

The statistical summary and correlation matrix (Table 3) reveal clear interdependencies among the measured physico-chemical parameters, reflecting the hydrogeochemical processes governing water quality in mining-impacted and reference sites. These correlations not only validate the analytical coherence of the dataset but also provide insight into the geochemical coupling of redox dynamics, ionic load, and particulate transport within the aquatic system.

Table 3

Statistical summary and correlation matrix of physico-chemical parameters

Parameter	*CV (%)*	*Min–Max range*	*Correlation (r)*	*Significance (p)*	*Interpretation*
pH	5.6	6.2–8.1	r = -0.78 (with Eh)	<0.01	Inverse correlation: acidic waters reductive conditions
EC	15.5	490–1260	r = 0.91 (with TDS)	<0.001	Strong positive correlation between ion content and salinity
Turbidity	28.6	2.1–13.8	r = 0.67 (with DO)	<0.05	High turbidity reduces oxygen diffusion
DO	13.1	6.1–9.8	r = -0.72 (with TDS)	<0.01	Lower oxygen linked to increased dissolved load
Eh	11.3	180–298	r = 0.69 (with DO)	<0.05	Positive link between redox and oxygen levels

Note: EC – electrical conductivity; DO – dissolved oxygen; Eh – redox potential.

Statistical analysis revealed significant correlations among redox-sensitive parameters. The strong EC–TDS relationship (r=0.91, p<0.001) confirms that ionic strength governs conductivity. Negative correlations between pH and Eh indicate that acidified environments enhance reduction potential, favoring the mobilization of trace metals from sediments into the water column.

The pH exhibited a moderate coefficient of variation (CV=5.6%), indicating relatively stable acidity across sampling zones. Its inverse correlation with redox potential (r=-0.78, p<0.01) confirms the classical geochemical relationship where acidic waters promote reductive conditions, typically observed in environments affected by sulfide oxidation and organic matter degradation. In such systems, low pH enhances the solubility of transition metals (Fe, Mn, Cu, Zn), while the concurrent drop in Eh signifies oxygen depletion and dominance of reduced species. This strong inverse coupling thus serves as a diagnostic indicator of post-mining acid generation and subsequent redox imbalance.

Electrical conductivity (EC) displayed the highest correlation strength in the matrix (r=0.91, p<0.001 with TDS), demonstrating a linear dependency between ionic strength and dissolved mineral content. This robust association confirms that EC serves as a reliable proxy for salinity and mineralization in these systems. The high variability (CV=15.5%) across sampling sites suggests heterogeneous contamination sources, likely influenced by the degree of tailings leaching, dilution by rainfall, and connectivity with groundwater inflows. Such strong EC–TDS coupling is characteristic of catchments dominated by evaporative concentration, ion exchange, and metal-sulfate release from weathering residues.

The turbidity parameter showed considerable spatial fluctuation (CV=28.6%), highlighting dynamic sediment–water interactions. Its positive correlation with DO (r=0.67, p<0.05) may initially appear counterintuitive, but can be interpreted as a result of hydraulic mixing events that enhance both suspended particulate concentration and aeration. During high-flow conditions or physical disturbance, resuspension of sediments increases turbidity while simultaneously facilitating oxygen diffusion, particularly in shallow, fast-flowing streams. Nevertheless, in stagnant or stratified zones, this relationship may reverse, as excessive turbidity attenuates light penetration and promotes oxygen depletion in the benthic layer.

The negative association between DO and TDS ($r=-0.72$, $p<0.01$) reinforces the notion that oxygen solubility decreases in mineral-rich or organically loaded waters. Elevated TDS concentrations correspond to higher ionic strength, which reduces gas solubility, while oxygen is further consumed through oxidation of reduced ions and organic substrates. This pattern delineates a coupled redox-ionic stress scenario typical for post-mining aquatic systems, where chemical oxygen demand competes with biological processes for available oxygen.

Finally, the positive correlation between Eh and DO ($r=0.69$, $p<0.05$) validates the mutual control between these parameters, as both depend on oxidation–reduction equilibria and oxygen-driven reactions. High Eh values coincide with oxic conditions that stabilize Fe^{3+} and Mn^{4+} as insoluble oxides, thereby immobilizing trace metals; conversely, reduced Eh conditions favor their dissolution and mobility. This linkage emphasizes the critical role of oxygenation in maintaining metal speciation stability and the self-purification potential of aquatic ecosystems.

Conclusions. The results of this study demonstrate that the physico-chemical properties of water in mining-impacted zones of northwestern Romania remain significantly altered decades after the cessation of mining operations. Zones I and II, corresponding to former tailings ponds and abandoned mines, exhibit clear signatures of acidic leaching, ionic enrichment, and redox instability, which collectively influence the mobility, speciation, and persistence of dissolved and particulate contaminants.

The moderately acidic pH values (<6.5) and depressed Eh (<+250 mV) observed in these areas promote the solubilization and remobilization of Fe, Mn, and trace metals, sustaining a dynamic geochemical regime dominated by oxidation–reduction cycling. Elevated EC and TDS further indicate persistent ionic input from sulfates and chlorides associated with tailings leachates. Turbidity remains an important indicator of suspended particulate transport, acting as a key vector for metal dispersion under variable hydrological conditions.

In contrast, the control area (Zone III, Tîrlișua) maintains stable hydrochemical conditions with neutral pH, low conductivity, and high DO, reflecting a well-buffered, oxidizing, and ecologically balanced aquatic environment. These parameters represent the natural geochemical background of the region and serve as a reliable reference for assessing anthropogenic impact.

Correlation analyses highlight strong EC–TDS coupling ($r=0.91$), indicative of the control exerted by ionic strength on salinity and mineralization, and significant inverse relationships between pH and Eh ($r=-0.78$), confirming that acidification enhances reductive processes. Such relationships reinforce the interconnected nature of hydrogeochemical processes governing contaminant dynamics in post-mining systems.

Overall, the integration of CQL indices, statistical evaluation, and ISO-standard physico-chemical parameters provides a robust framework for diagnosing the long-term environmental legacy of mining activities. The results emphasize the necessity of continuous monitoring of redox-sensitive indicators (pH, DO, Eh) and the application of passive remediation strategies (e.g., constructed wetlands, reactive barriers) to mitigate residual contamination.

This study contributes to the growing body of evidence that post-mining aquatic systems require sustained, adaptive management to ensure ecological recovery and compliance with the objectives of the EU Water Framework Directive (2000/60/EC). The established hydrochemical baseline presented here can serve as a reference for future assessments of contaminant behavior, ecosystem resilience, and restoration effectiveness across the Carpathian mining landscapes.

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