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SOIL GEOCHEMISTRY ACROSS LAND USES IN THE LAKE ȚAGA MARE CATCHMENT, TRANSYLVANIA: INSIGHTS FOR FUTURE SEDIMENT SOURCE TRACKING USING LAKE SEDIMENTS

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Abstract: This study investigates geochemical variability in the Lake Ţaga Mare catchment located in central Transylvania, Romania, to separate sediment sources contributing to the lake sediment archive. Geochemical analyses of parent material, soils and subaerial sediments were conducted across diverse land-use types. A Principal Component Analysis (PCA) was employed to differentiate sediment sources. Results indicate that parent material is characterized by high concentrations of carbonate-related elements (Ca > 1 wt.%, Mg > 0.65 wt.%, Sr > 100 $mg \cdot kg^{-1}$), while agricultural soils exhibit elevated Si (> 23 wt.%) and Ti (> 0.45 wt.%) and lower Zr (< 180 $mg \cdot kg^{-1}$). Baseline geochemical ranges for other natural elements such as Pb (15 - 25 $mg \cdot kg^{-1}$), S (maximum catchment values of 380 $mg \cdot kg^{-1}$), P (300 - 600 $mg \cdot kg^{-1}$), Mn (400 - 1000 $mg \cdot kg^{-1}$) and Fe (2.5 - 4.5 wt.%) further allow identification of deviations linked to historical and modern anthropogenic pollution or lacustrine redox processes. The principal components effectively differentiate between carbonate-rich parent material, weathered soils, and surface vs. subsurface erosion sources. This novel approach, the first such attempt at a national level, provides a geochemical framework for historical sediment source tracking and palaeoenvironmental interpretation of Lake Ţaga Mare sediment archive. Specifically, it enables the identification of natural erosion phases, anthropogenic disturbances associated with historical land use changes, and abrupt events such as floods, landslides or other forms of deeper soil disturbance. Finally, results could inform locally-adapted strategies to mitigate long-term soil degradation and guide sustainable land-use planning.

Keywords: sediment fingerprinting, XRF, PCA, soil erosion, land-use, lowlands, marl-clay lithology, Romania

1. INTRODUCTION

Soil is recognized as one of the most complex ecosystems on Earth, playing a fundamental role in supporting terrestrial life (Kopittke et al., 2019). However, soil quality is increasingly threatened by processes such as erosion, which is the leading contributor to land degradation, and a major constraint on agricultural productivity (Eekhout & de Vente, 2022). Soil erosion rates are driven by a combination of climatic conditions, land use patterns, and agro-environmental policies, with climate change further exacerbating erosion risks through increased rainfall and drought intensity and distribution (Regnier et al., 2013; Eekhout & de Vente, 2022; Arias-Navarro et al., 2024). Currently, around 24 % of soils in the European Union (EU) are affected by water erosion, predominantly in croplands, with projections indicating a potential increase of 13 - 25 % by 2050, mainly due to an increase in future rainfall intensity (Panagos et al., 2021; Arias-Navarro et al., 2024). Additionally, average land susceptibility to wind erosion across the EU shows moderate and high levels, ranging from 13 to 25.8 million hectares, where soil erosion mostly affects arable land (Borrelli et al., 2016). Under the RCP8.5 climate change scenario, soil loss is expected to increase in up to 84 % of the European Union territory. In Romania, average annual soil loss rates amount to almost one tone per hectare, increasing nearly threefold in lowland areas (Borrelli et al., 2017a), and are projected to rise by 10 - 20 % under the RCP4.5 scenario, and by up to 50 % under the RCP8.5 scenario (Panagos et al., 2021).

While the major factors influencing soil erosion operate at large spatial scales, their effects often vary considerably at local scales due to site-specific conditions such as topography, soil type, distribution of vegetation cover, localized human activities and farmers' decisions (Borrelli et al., 2017b; Kavelidze & Kalandadze, 2023; Mihajlović et al., 2024). Reconstructing past soil erosion at a local (i.e., catchment) scale is critical for understanding how these factors interact in specific contexts, and over a longer timeframe, enabling the development of adaptive land management strategies to address increasing erosion risk (Collins et al., 2020).

Natural sediment archives, such as lake sediments and peat deposits, have long been used as records of past soil erosion dynamics, integrating information over centuries to millennia (e.g., Engstrom & Wright, 1984; Boyle, 2002; Hutchinson et al., 2016, 2024; Panait et al., 2019; Florescu et al., 2024). However, most of these studies focus on identifying palaeoenvironmental processes and conditions solely based on the properties of the sediment archives, with limited research tracing sediment sources and transport pathways at the local (catchment) scale, constraining our ability to fully reconstruct and understand past erosion dynamics (e.g., Boyle, 2002; Arnaud et al., 2012). In Romania, sediment source tracking (i.e., fingerprinting) has been attempted in only two case studies, both reconstructing palaeoenvironmental changes in mid-elevation mountain areas (Florescu et al., 2017; Haliuc et al., 2020). No such attempts exist in the country's vast lowland agricultural areas, which are more exposed to the effects of soil erosion. Furthermore, both studies referenced above relied on volume magnetic susceptibility as the sediment source tracker – a method likely unsuitable for lowlands, where intense pedogenesis and long-term human activities alter soil magnetic properties (e.g., Liu et al., 2012). Soil geochemistry, in contrast, offers a promising alternative for sediment source fingerprinting in such environments (Smith & Blake, 2014; Collins et al., 2017).

In Romania, soil geochemistry has traditionally been determined using wet chemical analysis and spectroscopic techniques, and has primarily focused on nutrient levels and heavy metal concentrations to assess soil quality and ecological risks (Sârbu-Rădășanu & Buzgar, 2013; Huzum et al., 2015; Ungureanu et al., 2017; Damian et al., 2019; Maftei et al., 2019; Sur et al., 2022; Pascu et al., 2024). Recent advancements, such as portable X-ray fluorescence (XRF) spectroscopy that allows for in situ multi-elemental quantification, was used to determine major and trace elements and soil contamination/pollution (Weindorf et al., 2013; Paulette et al., 2015). However, integrating soil geochemical data with sediment archives to investigate changes in sediment sources over time at a catchment scale has yet to be applied, leaving an important gap in understanding how climate and land-use changes have influenced geochemical signatures in lake sediments.

The Țaga Mare Lake catchment in central Transylvania offers an ideal natural laboratory to address these challenges. This catchment features a relatively homogeneous geology, dominated by marly clays, with predominantly agricultural land-use, simplifying the analysis of soil geochemical variability. This area, characterized by a long history of human habitation (National Archaeological Database, www.ran.cimec.ro), is also ecologically vulnerable, with widespread landslides and sheet erosion processes threatening agricultural land productivity, but providing ample sediment sources (Rădoane et al., 2014). The lake itself, situated in a tectonic micro-depression, has historically served as a natural buffer for floodwaters and a resource for fishing, while also functioning as an archive of sediment inputs from the catchment (Fizeș Basin ROSCI-ROSPA Management Plan - 2005).

This study investigates the geochemical composition of soils across different land uses, soil types, and depths within the Țaga Mare Lake catchment. Non-destructive XRF analysis and multivariate statistical analysis are used to: (1) identify the primary characteristics of soil geochemistry at the catchment scale; and (2) establish methodological guidelines for sediment source tracking using the lake's sediment archive.

By applying sediment source fingerprinting to geochemical data, this research represents a first step into reconstructing the relationship between soil erosion, climate and land use practices over centuries to millennia in the country's agricultural lowlands.

Such reconstructions would further inform locally-adapted strategies to mitigate soil degradation, and guide sustainable land-use planning. Moreover, this study contributes to future research on catchment-level sediment dynamics, with implications for addressing larger-scale challenges in land and water resource management.

2. STUDY AREA

The catchment of Lake Ţaga Mare is located in the central part of the Transylvanian Plain, within the Fizeș River basin, and spans approx. 6500 ha (Figure 1). The lake itself (46°55'38"N, 24°4'36"E) covers approximately 101 hectares, with a maximum depth of 3.4 m (Mârza, 2009). It is part of a chain of shallow lakes along the Fizeș River, which collectively cover 439 hectares and account for 31.4 % of the fishing areas in the Transylvanian Plain (Sorocovschi, 2008). The lake has an elongated shape and is located at an altitude of 285 m asl. Its water is classified as mixed sulfate-bicarbonate type (Table 1), a characteristic influenced by the carbonate- and sulfate-rich lithology of the catchment, as well as episodic soil salinization during dry periods (Gâștescu & Parichi, 1963; Floca et al., 1998; Sorocovschi, 2009; Mihăescu et al., 2010).

The region's landscape consists of gently undulating hills separated by wide valleys, has an average elevation of 350 m and slopes ranging from 0 - 24° (Irimuș, 1998). The underlying geology of the area comprises Badenian, Sarmatian and Pannonian marly clays and marls, with occasional intercalations of tufa and sand. The climate is humid continental (Dfb in the Köppen-Geiger classification system). Mean annual temperature is 8.2 °C and average annual precipitation ca. 620 mm, based on the 1865 - 2022 data recorded at the Cluj-Napoca meteorological station. Precipitation exhibits strong seasonal variability, peaking in late spring and early summer, and reaching a minimum in winter. The Fizeș River, which traverses the catchment, has a pluvio-nival hydrological regime, characterized by high flows during spring snowmelt and rainfall, followed by low flows from late summer to early autumn (Sorocovschi, 2005). The river has a multiannual average flow of 1.3 m^3/s at the Fizeșu Gherlii hydrometric station. Vegetation includes patches of deciduous and coniferous forests, transitional zones with grasses and shrubs, and extensive pastures (Figure 1).

The geomorphological, hydrological and vegetation history of the Fizeș River basin also reflects long-term human influence. Archaeological

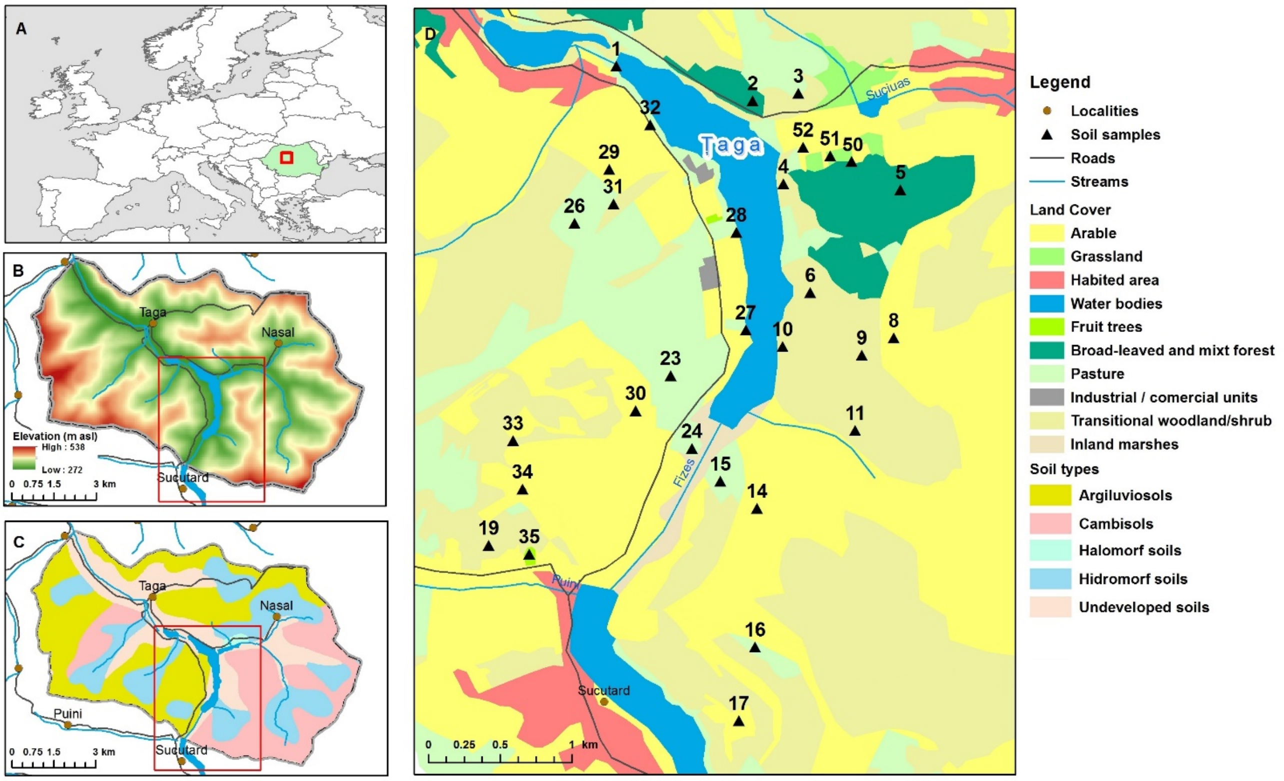

Figure 1. Characteristics of the study area: a) location of the Lake Ţaga Mare catchment in Romania and Europe; b) digital elevation model of Lake Ţaga Mare catchment; c) distribution of soil types in the lake's catchment; d) distribution of land cover/use types. Black triangles mark soil sampling locations, and numbers are sample denominations - see Table 2 for additional details.

evidence (available in the National Archaeological Database, www.ran.cimec.ro) indicates that the region has been continuously inhabited since the Neolithic period (6000 - 8000 years before present). Early settlements, concentrated around Lake Țaga Mare, reveal a history of agriculture, livestock rearing, and deforestation to expand arable land. By the Bronze Age, cereal cultivation, viticulture, and orchard farming had become widespread, with settlement patterns further intensifying during Roman and medieval times (Mârza, 2009).

Table 1. Main characteristics of water in Lake Țaga Mare (extracted from Gâștescu & Parichi, 1963; Floca et al., 1998; Sorocovschi, 2009).

Variable (mg/L)	**1963**	**July 1997**	**2004**	
			Lake surface	**Lake bottom**
Cl^-	42.5	100	90	92
$SO4^{2-}$	341.8	108	233.6	228.1
HCO^{3-}	597.9	335.5	202	178
$Na^+ + K^+$	176.6	150.3	92	91.6
Ca^{2+}	94.6	89.25	44.9	80.2
Mg^{2+}	69.5	0	53.2	33.1
Mineralization	1322.9	783.1	715.7	703.0

Lakes along the river, including Lake Țaga Mare, were documented as early as the 14th century. Historical sources dating back as early as AD 1326 describe the Fizeș (Szarvas) River floodplain as a continuous chain of lakes, some up to 1000 meters wide, resembling a "fluvius" that extended across the entire current Fizeș Valley, from the Cămăraș area to the "Moara Roșie" (Mârza, 2009). This depiction is supported by Schrembl and Wenzeli's 1789 map, the first cartographic record to illustrate this "fluvius" and what is now Lake Țaga Mare. These lakes were historically managed for water storage and mill operations beginning in Roman times and continuing through the Middle Ages (Fizeș Basin ROSCI-ROSPA Management Plan - 2005). A complete sediment core extracted from Lake Țaga Mare was radiocarbon-dated as part of the Young Teams project "HoLFloods - Mid-late Holocene flood events in south-eastern and central Romanian lowlands". Eighteen radiocarbon dates along the core confirm that the lake formed around 6200 years ago and that it had a continuous sedimentation (Bădăluță et al., 2023). Consequently, the sedimentary archive of Lake Țaga Mare holds the potential to provide insights into the evolution of the natural environment under human influence since the Neolithic, as well as the consequences of intensified human impact on erosional processes and hydrological events.

3. MATERIAL AND METHODS

3.1. Sample collection, pre-processing and cartographic analysis

In May 2022, we conducted soil sampling in the Lake Taga Mare catchment. Sampling locations (n = 30), shown in Figure 1, encompassed all major agricultural land types (arable land, abandoned arable land, orchard, grassland and pasture, degraded grassland/pasture, shrubland, forest), subaerial sediment deposits upstream and downstream of the lake and parent material from the lake shore and outcrops (Table 2). This strategy was used to identify potential sediment sources contributing to the lake and the areas most susceptible to erosion. At each location, we collected surface samples (0 - 5 cm) and deeper samples (25 - 30 cm). For each depth, three samples were taken within a 5-meter radius under the same land-use conditions to account for micro-variability in soil properties. In total, 186 samples were collected and processed in the laboratory. Processing involved oven drying the samples at a maximum temperature of 37 °C for 1 - 2 days until completely dry, followed by gentle manual disaggregation and sieving through a 1 mm mesh to remove coarse components. Subsequently, the three surface samples from each location were combined, homogenized, and a representative subsample was taken for geochemical analysis. The same procedure was applied to the deeper samples. Overall, 60 samples (30 surface and 30 deeper samples) were prepared and subjected to geochemical analysis.

To characterize the study area and location of the sampling points, cartographic tools were employed. The digital elevation model of Lake Țaga Mare catchment was derived from the Shuttle Radar Topography Mission data set (SRTM; http://www2.jpl.nasa.gov/srtm/). Land cover/use classes were extracted from the Corine Land Cover (CLC) 2018 dataset (copernicus_r_3035_100_m_clc-2018_p_2017-2018_v20_r01) and corrected based on the 2024 satellite imagery available on Google Earth and field observations. To assess major changes in the spatial distribution of land-use classes over the last decades, that may have altered soil composition, we digitized data from the 1984 topographic map 1:25000 (Direcția Topografică Militară - 1984) and the 2024 satellite imagery. All cartographic analyses were carried out in ArcMap 10.7 (ESRI, 2011).

3.2. Geochemistry via X-ray fluorescence

The 60 surface and deeper soil samples were analyzed non-destructively at Goethe University (Frankfurt am Main, Germany) using a Niton XL3t

Table 2. Location and main characteristics of the sampling points shown in Figure 1.

Sample number (as in Figure 1)	**Land-cover and use**	**Latitude**	**Longitude**	**Elevation (m asl)**	**Observations**
1	Downstream sediment	46°56'16.14"	24° 4'6.40"	289	
2	Forest	46°56'04.8"	24°'4'54.9"	294	Pine plantation
3	Grassland/pasture with shrubs	46°56'05"	24°'5'02.6"	330	Only scattered shrubs
4	Parent material	46°55'47.1"	24°'5'0.09"	263	Lake shore
5	Forest	46°55'47.7'	24°4'22.5"	368	Transect (mixed forest)
50	Forest	46°55'55.6"	24°'5'19.8"	335	Transect (forest margin)
51	Grassland/pasture	46°55'53.2"	24°'5'17.9"	323	Transect
52	Arable	46°55'56.3"	24°'5'15.4"	302	Transect
6	Parent material	46°55'23.5"	24°'5'12"	311	Outcrop
8	Abandoned arable	46°55'14.4"	24°'5'08.7"	310	Abandoned for over 3 years
9	Pasture	46°55'08"	24°'5'07.1"	309	Compact shrub encroachment uphill of the sampling point
10	Parent material	46°55'11"	24°'5'1.4"	283	Lake shore
11	Degraded grassland/pasture	46°54'32.5"	24°'5'8.5"	303	Eroded slope; scattered shrubs
14	Arable	46°54'32.9"	24°'4'48.4"	360	
15	Grassland/pasture	46°54'29.8"	24°'4'48.1"	364	
16	Parent material	46°54'02.9"	24°'4'55.5"	426	Landslide
17	Arable	46°53'50.2"	24°'4'44.2"	327	
32	Parent material	46°56'03.9"	24°'4'16.8"	316	From a road embankment under construction
26	Grassland/pasture	46°55'44.6"	24°'4'6.8"	356	
31	Shrub encroachment	46°55'43"	24°'4'6.9"	361	
23	Degraded grassland/pasture	46°54'52.7"	24°'4'23.6"	315	Scattered shrubs
30	Arable	46°54'52.2"	24°'4'23.9"	314	Maize field on a slope
24	Upstream sediment	46°54'49.4"	24°'4'30.3"	282	
27	Grassland/pasture	46°55'14.9"	24°'4'51.1"	300	On the lake’s shore
28	Grassland/pasture	46°55'35.6"	24°'4'48.4"	294	On the lake’s shore
29	Arable	46°55'54"	24°'4'13.5"	337	Maize field
33	Grassland/pasture with shrubs	46°54'38.3"	24°'3'24.3"	387	Transect (upper terrace, T1)
34	Arable	46°54'36.6"	24°'3'48.5"	398	Transect (maize field; middle terrace, T2)
35	Orchard	46°54'24.9"	24°'3'23.3"	302	Transect (lowest terrace, T3)
19	Degraded grassland/pasture	46°54'25.7"	24°'3'23.9"	325	

GOLDD+ X-ray fluorescence handheld analyzer. A reference soil sample with known elemental concentrations was used to assess the measurement accuracy. Measurements were conducted under helium flow to increase the detection of light elements such as Mg, Al, Si, P, S, and Cl (Adams et al., 2020), using the factory-calibrated soil setting. Each sample was measured twice, and the results were averaged. In this study, the concentrations of the most common detrital elements (e.g., Ti, Zr, Rb, K, Al, Si, V, Fe, Mn, As), heavy metals (e.g., Pb, Zn, Cu, Cr), carbonate-related elements (Ca, Mg, Sr) and nutrient-related elements (P, S) were used.

3.3. Statistical analysis

We used XY scatterplots to describe elemental concentrations for samples grouped by sample and land-use type. Then, we used a Principal Component Analysis (PCA) to visualize the relationships between samples based on their geochemical properties. Both analyses were performed in the PAST software (Hammer et al., 2001). PCA was conducted on the correlation matrix of the data, and the significance of the principal components was tested via the Broken stick model (Bennett, 1996). The PCA outputs are presented as both a minimum spanning tree (to identify sample-to-sample distances, gradients and outliers) and a biplot (to show the variables, i.e., geochemical elements, and their contributions to the principal components).

4. RESULTS AND DISCUSSION

4.1. Geochemical variability across sample types and land uses

Shifts in contrasting land-use types (e.g., from forests to agricultural land) affect soil geochemistry through alterations in vegetation cover, soil disturbance, and fertilization practices (e.g., Richter & Markewitz, 2001). These shifts can also influence the rates of soil erosion and degradation, which have intensified in Romania over the last decades, in both natural and agricultural environments (e.g., Begy et al., 2016; Hutchinson et al., 2016; Florescu et al., 2019). We explored land-use changes in the Lake Țaga Mare catchment over the last four decades to contextualize geochemical signatures of soils, outlier samples and ultimately sediment source dynamics.

A spatial comparison of land-use in the Lake Țaga Mare catchment between 1984 and 2024 (Figure 2) reveals relatively stable areas occupied by arable land and broadleaved forests. However, some notable changes were observed, including an increase in the coniferous forest cover due to a recent pine plantation (sampling point 2), an increase in transitional woodlands, as well as an expansion of industrial activity areas which were excluded from sampling. Grasslands and pastures show a reduction in area likely due to shrub encroachment resulting from decreased grazing pressure, as documented in other parts of the Fizeș River Basin (Feurdean et al., 2017). To account for potential geochemical differences associated with this encroachment, the sample categories “Grassland/pasture” and “Grassland/pasture with shrubs” were included in Table 2 and Figures 3 and 4. Despite these changes, the overall spatial distribution of land-use types suggests that recent alterations have been minor, with limited impact on the catchment’s soil geochemical properties at the sampling locations. This stability supports the interpretation that current geochemical signatures primarily reflect longer-term land-use and environmental dynamics, rather than recent shifts.

The geochemical analysis reveals distinct elemental patterns between parent material, subaerial sediment deposits and degraded grasslands on one hand, and agricultural land on the other. In contrast, the differences between land-use types (forest, grassland/pasture, arable, orchards, encroached grasslands) are much smaller (Figure 3). Parent material, sediment deposits upstream and downstream of the lake, and degraded grasslands exhibit lower Zr and Si concentrations (generally below 180 mg·kg^{-1} and 23 wt.% respectively) compared to agricultural land. Thus, a combination of low Si and Zr concentrations may differentiate between these potential sources of eroded material, as opposed to Rb concentrations, which are more mixed, making this element less effective for this purpose.

Spatial differences are apparent in the case of Si concentrations: soil samples from the left side of the catchment exhibit higher Si concentrations (above 24 wt.%) compared to those from the right side. Notably, the left side is dominated by pastures and cultivated land on Argiluvisols, whereas the right side shows large areas covered by forest and shrub-encroached grassland, mostly on underdeveloped soils and Cambisols (Figure 1). Ti concentrations are above 0.45 wt.% in cultivated soils but lower in eroded and parent material, while K shows no discernible patterns across sediment sources (Figure 3).

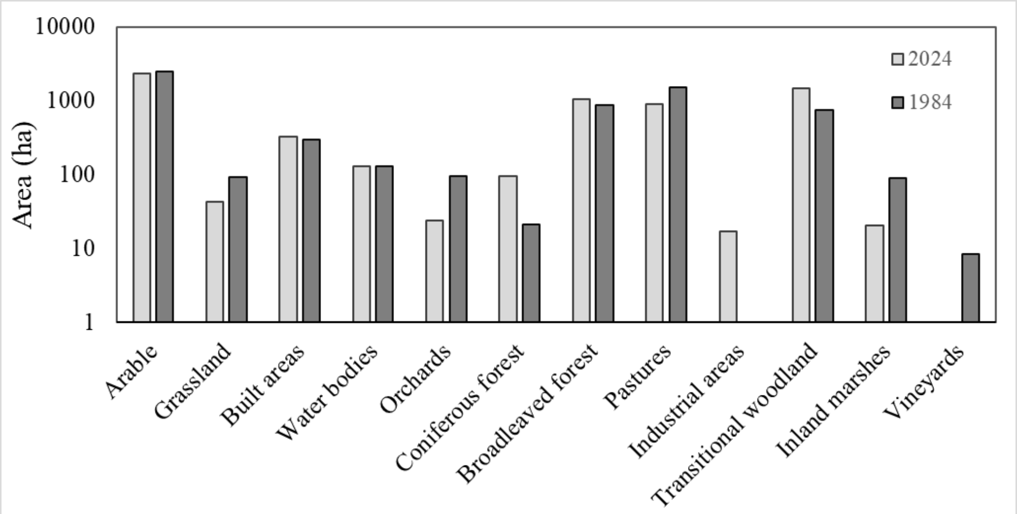

Figure 2. Changes in the spatial distribution of land-uses from 1984 to 2024 in the Lake Țaga Mare catchment.

Shrub-encroached grasslands resemble orchards in their geochemical profile, characterized by higher Rb concentrations (over 110 mg·kg^{-1}) and lower Zr (below 200 mg·kg^{-1}) and Si (below 23 wt.%), compared to open grasslands/pastures.

The underlying lithology, predominantly marly clays, suggests elevated concentrations of carbonate-related elements such as Ca, Sr, and Mg in parent material, sediment deposits and deeper soil layers. As expected, parent material and sediment deposits exhibit the highest concentrations of these elements (Ca > 1 wt.%, Sr > 100 mg·kg^{-1}, Mg > 0.65 wt.%), but these elements do not appear to differentiate between surface and deeper samples (Figure 3).

In agricultural soils, Ca concentrations are typically below 1 wt.%, while Si concentrations exceed 23 wt.%, and this elemental combination may serve to differentiate parent material from soils. Encroached grasslands and orchards display slightly higher concentrations of Ca, Sr, and Mg compared to open grasslands/pastures. This difference may result from lower soil disturbance in encroached grasslands

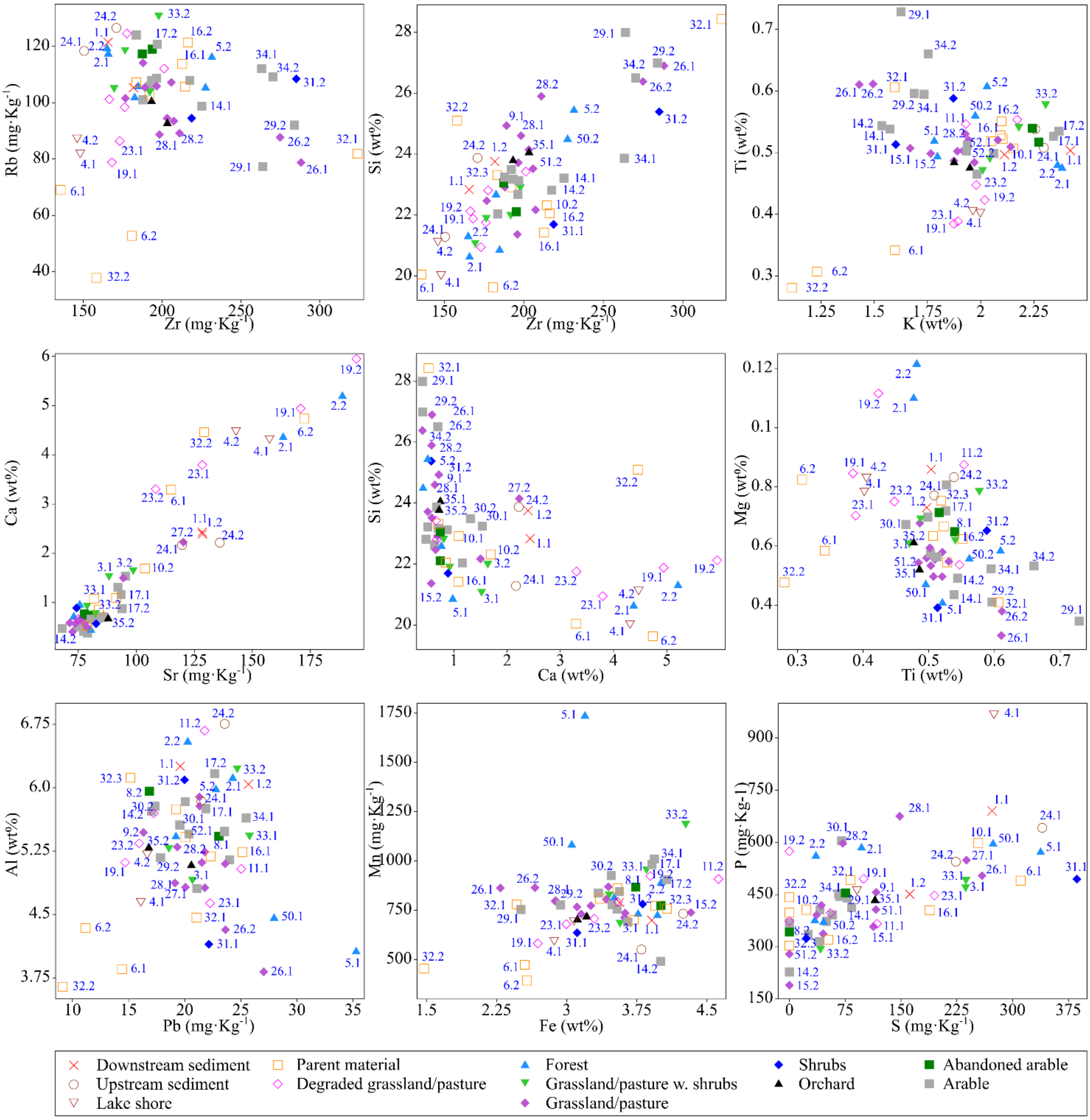

Figure 3. Scatterplots describing elemental concentrations for samples grouped by sample type and land-use in the Lake Țaga Mare catchment. Empty symbols are used for subaerial (upstream, downstream) sediment, parent material (incl. lake shore) and degraded grassland, while filled symbols denote land-uses. Sample numbers represent soil sample locations as shown in Figure 1 and Table 2. For each sample location, the digit “1” after the decimal point denotes surface sample, while the digit “2” after the decimal point indicates deeper sample.

and orchards, allowing these elements to remain closer to their natural concentration as derived from the parent material. Interestingly, most forests samples have among the lowest concentrations of Ca, Mg and Sr in the dataset, similar to cultivated land. This can be explained by a combination of factors related to nutrient cycling, leaching, and reduced soil disturbance under forest cover (e.g., Labaz et al., 2022). Supporting this interpretation, the two outlier forest soil samples (sampling point 2) from a recent pine plantation on the right lake shore align geochemically with degraded land and parent material, rather than with other forest samples. These outlier samples exhibit high concentrations of carbonate-related elements, as well as maximum K and Al, likely due to soil cover disturbance during plantation establishment.

Deeper soil samples show elevated Al concentrations (over 5.5 wt.%) compared to surface samples, making Al a potential marker for distinguishing between these depths. This difference in Al concentrations between the two sampling depths may be attributed to the aluminosilicate minerals (e.g., kaolinite, smectite, or illite) in the marl-clays remaining less weathered at deeper levels due to reduced exposure to surface conditions such as acid rain, organic acids, and fluctuating temperatures (White, 1995). In contrast, other soluble elements such as K do not show higher concentrations in deeper samples, indicating that leaching is not likely a significant factor in the observed Al enrichment.

Pb concentrations in the catchment soils and rocks range from 15 - 25 mg·kg^{-1}. Values considerably larger than this range could potentially discriminate historical atmospheric pollution in the lake sediment archive (e.g., Hutchinson et al., 2016; Mîndrescu et al., 2022). Natural Mn concentrations range from 400 - 1000 mg·kg^{-1}, while those of Fe are from 2.5 - 4.5 wt.%, offering a baseline for assessing the impact of redox-driven processes on the stratigraphy of redox-sensitive elements in the lacustrine archive (e.g., Florescu et al., 2017). Phosphorus (P) concentrations, ranging from 200 - 700 mg·kg^{-1}, are slightly higher in parent material and sedimentary deposits but lower in agricultural land, likely due to biotic uptake. Sulphur (S) content is more elevated in surface samples (above 100 mg·kg^{-1}), and, along with Al, may help identify these sources of material.

4.2. Geochemical fingerprints and potential for sediment source tracking

A Principal Component Analysis (PCA) was applied to geochemical data to explore the spatial distribution/clustering of the samples based on their cumulated geochemical properties and show which chemical elements are the most important in differentiating the samples. The Broken Stick model revealed that the first three Principal Components are statistically significant, collectively explaining 76 % of the variance in the entire geochemical dataset (Figure 4). The first Component (37.3 %) is positively associated with Rb, Fe, V, Al, K, with large loadings, and negatively associated with Sr, Ca and P. This pattern likely reflects weathering and soil formation processes, and the distinction between weathered and less weathered soils is the carbonate-dominated material. Specifically, the strong positive associations with Al, Fe, K, and Rb indicate the dominance of aluminosilicate minerals (e.g., clays like illite or kaolinite) and Fe oxides. Rb may be associated with K-bearing minerals such as micas, which are retained in less leached soils (Négrel et al., 2018). Negative associations highlight depletion of Sr, Ca, and P in weathered soils, as Sr and Ca are markers of carbonate-rich parent material (Figure 3) and P may reflect the loss of apatite mineral during weathering (Négrel et al., 2018). The minimum spanning tree illustrates this interpretation, as samples taken from agricultural land cluster together towards the positive side of Axis 1, while samples from the parent material, subaerial sediment and degraded grasslands appear as outliers from the main cluster (Figure 4). Therefore, Axis 1 helps identify the intervals where clay-rich, weathered material dominated in the lacustrine archive, potentially linked to enhanced surface erosion.

Component 2 (26 %) groups most of the variance related to Sr, Ca, Mg, K (positive loadings) and Zr, Ti, Si (negative). It likely distinguishes carbonate-rich soils and parent material (positive side) from silicate-dominated or heavy mineral-enriched soils (negative side). On the positive side, Sr, Ca, and Mg reflect carbonate minerals, which are abundant in the marl-clay lithology. On the negative side, Zr, Ti, and Si indicate enrichment in heavy minerals (e.g., zircon, rutile) and silicates, which are resistant to weathering. Their strong negative loadings suggest they are most prevalent in areas where carbonate material is heavily weathered. The clustering of parent material, sediment and degraded grasslands on the positive side of Axis 2 in the minimum spanning tree is in accordance with this interpretation. In the lacustrine core, Axis 2 may therefore reflect inputs from carbonate-rich soils, possibly linked to marl-clay parent material during periods of minimal chemical weathering or intensification of physical erosion.

On Component 3 (12.7 %), Pb, Zn, Mn, S and P align on the positive side with the highest loadings, while As and Al are positioned on the negative side. It highlights the distinction between surface (0 - 5 cm) and deeper (ca. 30 cm) samples. Surface samples' geochemistry (positive side) is influenced by heavy metals such as Pb and Zn – which may be also associated with anthropogenic contamination (Kumpiene et al., 2008), nutrient inputs (P), and redox-sensitive elements (Mn, S). Deeper samples' geochemistry (negative side) is dominated by lithological signatures, with stable aluminosilicate

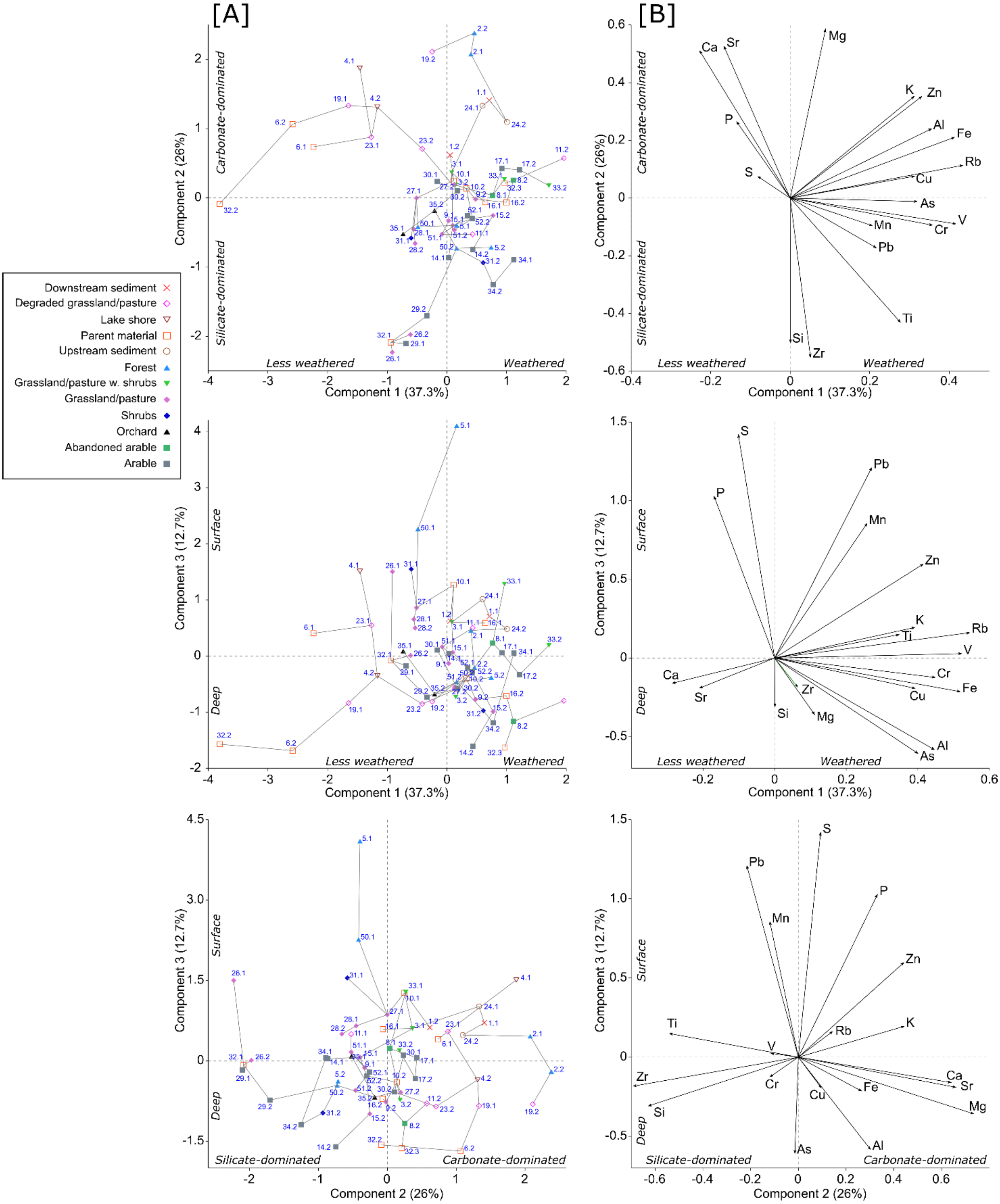

Figure 4. Principal Component Analysis of soil samples in the Lake Ţaga Mare catchment: A) Minimum Spanning Tree for the Components 1-3, showing the proximity of samples based on their geochemical properties; B) PCA biplot for the Components 1-3, showing vector orientations along the three statistically significant axes/components. Empty symbols are used for subaerial (upstream, downstream) sediment, parent material (incl. lake shore) and degraded grassland, while filled symbols denote land-uses. Sample numbers on the minimum spanning tree plots represent soil sample locations.

minerals (Al) and naturally occurring elements like As. The strong differentiation between surface and deeper samples on Component 3 emphasizes the role of surface processes (e.g., nutrient cycling, human activity) in modifying soil geochemistry, compared to the more stable, lithology-driven conditions at depth. The separation of surface and deeper samples is evident in the minimum spanning tree, with surface samples clustering positively on Axis 3. Component 3 can therefore indicate whether sediments in the lacustrine archive were primarily derived from surface erosion or deeper soil disturbance (e.g., due to landslides, channel erosion or intensive agriculture).

5. CONCLUSIONS

This study employs a geochemical approach to differentiate sediment sources in the Lake Țaga Mare catchment contributing to the lake sediment archive. By analyzing the geochemical characteristics of catchment samples and their spatial variability, we provide guidelines for interpreting the lake's sediment archive in future investigations. The geochemical analysis of catchment samples shows that parent material is characterized by high carbonate-related elemental concentrations (Ca > 1 wt.%, Sr > 100 mg·kg^{-1}, Mg > 0.65 wt.%), typical for the marl-clay lithology, and low Zr (< 180 mg·kg^{-1}) and Si (<23 wt.%) concentrations. Conversely, agricultural land is enriched in Si, Zr and Ti (> 0.45 wt.%). At the catchment level, Zr and Si are indicators of silicate and heavy mineral content, elevated in agricultural land and lower in degraded soils and parent material, while Ca, Sr, and Mg are markers of carbonate-rich parent material and sediment deposits. Ti is elevated in cultivated soils, distinguishing them from degraded or less weathered sources. Al is elevated in deeper soils, distinguishing surface and subsurface material. Baseline geochemical ranges for other natural elements such as Pb (15 - 25 mg·kg^{-1}), S (maximum catchment values of 380 mg·kg^{-1}), P (300 - 600 mg·kg^{-1}), Mn (400 - 1000 mg·kg^{-1}) and Fe (2.5 - 4.5 wt.%) can be used to identify deviations linked to historical and modern anthropogenic pollution or lacustrine redox processes.

The PCA separates the geochemical dataset into components that represent distinct processes and source materials. It is therefore possible, in this particular case, to differentiate between clay-dominated, weathered soils and carbonate-dominated parent material, and between surface and subsurface material, attributable to surface and deeper erosion respectively. By combining individual elemental concentrations, this approach has created a geochemical "fingerprint" for each sediment source and thus enables distinguishing surface processes (pedogenesis, anthropogenic inputs, nutrient cycling, redox activity) from deeper, lithology-driven geochemistry. The geochemical composition of each sediment layer in the lacustrine core can then be projected onto these PCA axes, allowing to identify natural erosion phases, anthropogenic disturbance linked to historical land use changes, and abrupt events such as floods or landslides. This approach also accounts for the effect of lacustrine diagenetic and biogenic processes on sediment properties. However, sampling density and reliance only on geochemical tracers may limit the generalization of our findings. Future studies should incorporate mineralogical analysis, as well as isotopic and organic geochemical markers for improved source attribution, and also extend the geochemical analysis to other lowland catchments.

The geochemical patterns identified in this study lay the groundwork for understanding sediment contributions to the lake archive over time. By integrating these findings into catchment management practices, stakeholders will be able to make more informed decisions to address soil degradation and mitigate erosion, ensuring the long-term sustainability of soil resources, and maintaining the ecological and hydrological functions of the catchment-lake system.

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