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MINERAL COMPOSITION OF *VACCINIUM VITIS-IDAEA* FRUITS ACROSS DIFFERENT MOUNTAIN HABITATS

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Abstract

The present study investigated the mineral composition of Vaccinium vitis-idaea fruits collected from five mountain regions of the Romanian Carpathians, characterized by distinct environmental and geological conditions. Fruit samples were analyzed using portable X-ray fluorescence spectrometry (pXRF) to determine the concentrations of macro-, micro- and trace elements, including K, Ca, Fe, Mn, Zn, Cu, Ni, Sr and Rb. Climatic parameters describing the environmental context of the sampling sites were also considered. The results revealed clear differences in elemental profiles among the studied provenances. Potassium was the dominant macronutrient in all samples, whereas Ca, Mn and Rb exhibited noticeable spatial variability. Multivariate analyses (HCA and PCA) indicated that the elemental profile of lingonberry fruits reflects both environmental gradients and site-specific geochemical characteristics. In particular, rubidium showed the strongest differentiation among sites, suggesting a possible relationship between berry elemental composition and the geological characteristics of the habitats. The results highlight spatial variability in the mineral profile of wild V. vitis-idaea fruits across mountain environments and provide baseline information on their elemental composition in the Romanian Carpathians.

Key words*: Carpathians, environment, mineral composition, Vaccinium vitis-idaea, variability.*

INTRODUCTION

Vaccinium vitis-idaea L. (lingonberry) is a perennial evergreen shrub of the *Ericaceae* family that is widely distributed throughout boreal forests and alpine regions of the Northern Hemisphere. The species typically grows in nutrient-poor, acidic soils and the berries are traditionally harvested from wild populations and are widely consumed in Northern and Central Europe due to their nutritional value and characteristic sour taste (Hjalmarsson & Ortiz, 2001; Nestby et al., 2019). Lingonberry fruits have received considerable scientific attention due to their high content of bioactive constituents, including phenolic acids, flavonoids and anthocyanins, which contribute to their high antioxidant capacity and potential health-promoting properties (Bujor et al., 2018; Kowalska, 2021). In addition to these compounds, lingonberry fruits also contain several mineral elements that contribute to their nutritional value and play important roles in plant physiological processes (Toncheva et al., 2016).

The concentration of mineral elements in wild berries can vary significantly depending on environmental conditions (Ekholm et al., 2007). Soil properties, nutrient availability and geographical location may influence the uptake and accumulation of macro- and micronutrients in plant tissues. Studies on lingonberries have shown that the concentration of elements such as K, Mn, Fe, Zn and Cu can vary between plant organs and sampling sites, reflecting differences in soil chemistry and environmental conditions (Lazarenko et al., 2021).

Environmental factors such as temperature, precipitation and altitude can influence plant physiological processes involved in nutrient uptake, transport and allocation within plant tissues. In mountainous ecosystems, these factors interact with soil characteristics and geological substrate to determine nutrient availability in the rhizosphere. As a result, plants growing under different habitat conditions may exhibit distinct patterns of mineral allocation between vegetative organs and fruits, reflecting both environmental constraints and species-

specific physiological regulation (Broadley et al., 2007; Luginina & Egoshina, 2013).

Despite the increasing interest in the nutritional and biochemical properties of lingonberries, information regarding the variability of mineral elements in fruits collected from different natural populations remains relatively limited. Investigating the mineral composition of lingonberries from different provenances can improve our understanding of spatial variation in element accumulation in wild populations and may also provide useful information on the nutritional value of these fruits. Therefore, the aim of this study was to characterize the mineral composition of *V. vitis-idaea* fruits collected from five mountain habitats in Romania and to examine whether differences in elemental profiles are associated with local environmental and lithological conditions. In this context, the study provides baseline data on the mineral profile of wild lingonberry fruits from the Romanian Carpathians.

MATERIALS AND METHODS

Study area and fruit sampling

Lingonberry (*Vaccinium vitis-idaea* L.) fruits were collected from five natural populations located in mountainous regions of Romania, each representing a distinct geographical area. The sampling sites were Groșii Țibleșului (GT; 47.551° N, 24.136° E), Valea Vinului (VV; 47.513° N, 24.851° E), Bilbor (BI; 47.115° N, 25.496° E), Cheia (CH; 45.427° N, 25.846° E) and Doda Pilii (DP; 46.662° N, 22.861° E). The coordinates represent the central point of the sampled area, while the abbreviations are used throughout the text to indicate the sampling site. Altitude was expressed as the midpoint of the reported elevation range for each sampling site (m a.s.l.).

Lithological information for each location was determined using geological maps and literature describing the geological structure of the Romanian Carpathians (Peters et al., 2018). These locations differ in terms of altitude, lithology and local environmental conditions, as presented in Table 1. Fruit sampling was conducted in August 2023, when berries had reached full ripeness (Figure 1).

The fruits were cleaned to remove visible impurities and dried in a ventilated oven at 60°C until constant weight was reached. The dried material was subsequently ground into a fine homogeneous powder using a laboratory grinder.

Table 1. Geographic and environmental characteristics of the sampling sites

Site	County	Altitude	Lithology
Groșii Țibleșului	Maramureș	1250	Sedimentary
Valea Vinului	Bistrița-Năsăud	1550	Metamorphic
Bilbor	Harghita	1150	Metamorphic
Cheia	Prahova	1450	Sedimentary
Doda Pilii	Cluj	1150	Metamorphic

At each location, three independent composite samples were collected to account for within-site variability. Each sample consisted of berries harvested from several individual plants distributed within an area of approximately 100 m² in order to obtain representative material for the local population. The fruits from each sampling point were pooled to form a composite sample representing that site replicate. After collection, the samples were transported to the laboratory for further processing.



Figure 1. Fully ripe *V. vitis-idaea* berries at the time of sampling

The powdered samples were stored in airtight containers at room temperature until analysis. Elemental analysis was performed using an ElvaX Mobile Portable XRF Spectrometer (Elvatech Ltd., Kyiv, Ukraine). Prior to analysis, the powdered material was loaded into sample cups and analyzed directly using X-ray fluorescence spectroscopy (Sapkota et al., 2019; Zhou et al., 2023). To ensure analytical reliability, all measurements were performed under identical instrumental conditions and each sample was analyzed in replicate scans. The instrument calibration and element quantification were carried out using the

manufacturer's software and factory calibration settings. Detection limits were estimated based on the lowest detectable concentrations recorded during the measurements. Quality control was evaluated through repeated measurements of selected samples, which showed consistent elemental readings within the expected analytical variability of portable XRF measurements.

Climatic variables

Environmental data for each location were obtained from the ERA5-Land reanalysis dataset provided by the Copernicus Climate Data Store (Muñoz-Sabater et al., 2021).

Climatic variables included mean daily air temperature (°C), representing the near-surface air temperature measured at approximately 2 m above ground level; cumulative precipitation (mm), corresponding to the total precipitation reaching the land surface during the analyzed period; mean daily surface solar radiation (MJ m^{-2} day^{-1}), describing the incoming solar energy received at the land surface; and mean volumetric soil water content (m^3 m^{-3}), representing the proportion of water in the upper soil layer (0-7 cm).

These parameters were calculated for the period May-July by averaging daily values (temperature, solar radiation and soil moisture) and summing precipitation totals. This period corresponds to the main fruit development stage of *V. vitis-idaea*, when active nutrient transport and accumulation occur in developing berries, making it particularly relevant for assessing the mineral elements analyzed in this study.

The values were extracted based on the geographic coordinates of each sampling site, and the resulting parameters are presented in Table 2.

Table 2. Climatic variables of the sampling sites

Site	Temperature (°C)	Precipitation (mm)	Solar radiation (MJ m^{-2})	Soil moisture (m^3 m^{-3})
GT	15.6	379.09	20.07	0.35
VV	13.9	291.82	19.73	0.23
BI	14	359.35	19.62	0.29
CH	14.4	482.76	20.1	0.38
DP	15.1	497.83	19.57	0.38

The analyzed elements were grouped into three categories: macronutrients, micronutrients, and trace elements. Elemental concentrations were expressed on a dry weight basis (mg kg^{-1} DW). Values below the detection limit were replaced by half of the detection limit (LOD/2) prior to statistical analysis in order to avoid bias caused by non-detectable values. Detection limits were estimated based on the lowest detectable concentrations obtained during the analytical measurements.

Statistical analysis

Differences in elemental concentrations among sites were evaluated using analysis of variance (ANOVA), followed by a post hoc multiple comparison test to identify statistically significant differences between means ($p < 0.05$).

To explore relationships between mineral composition and environmental variables, multivariate statistical analyses were performed. Principal component analysis (PCA) was used to identify patterns in the distribution of mineral elements and to assess the influence of climatic parameters on elemental variability. In addition, hierarchical cluster analysis (HCA) was applied to evaluate similarities among provenances and to visualize relationships between elements.

All statistical analyses and graphical representations were performed using PAST software - Paleontological Statistics, version 4.17 (Hammer et al., 2001).

RESULTS AND DISCUSSION

Macronutrient concentrations

Potassium (K) was the predominant macronutrient in *V. vitis-idaea* fruits across all sampling sites (Figure 2a). The highest concentrations were recorded at Bilbor and Doda Pilii, exceeding 12,000 mg kg^{-1} DW, while lower values were observed at the other three provenances. Comparable K levels have been reported for lingonberry fruits (approximately 6,400-6,700 mg kg^{-1} FW) (Lazarenko et al., 2021), indicating that potassium represents the dominant mineral component in these berries. This high K content reflects its key physiological role in osmotic regulation and carbohydrate transport during fruit development.

Calcium (Ca) concentrations showed a different pattern among the sampling sites (Figure 2b), with the highest values recorded at Valea Vinului and Cheia (approximately 1,200-1,300 mg kg^{-1} DW), while lower levels were observed

at Groșii Țibleșului, Doda Pilii and Bilbor. Similar Ca levels have been reported in fruits of other *Vaccinium* species, such as *V. oxycoccos* and *V. macrocarpon*, where concentrations range from approximately 480-1,160 mg kg^{-1} DW (Karlsons et al., 2018). Calcium is primarily associated with structural functions in plant tissues, and its relatively moderate concentration in fruits reflects its limited mobility within the plant.

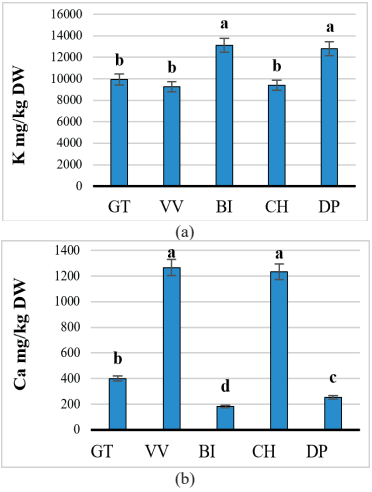


Figure 2. Macronutrient concentrations in *V. vitis-idaea* fruits: (a) potassium (K); (b) calcium (Ca). Mean ± SD (n = 3). Different letters indicate significant differences ($p < 0.05$)

Micronutrient concentrations

Iron (Fe) concentrations in lingonberry fruits were highest at Cheia and Bilbor, reaching approximately 150 mg kg^{-1} DW (Figure 3a), whereas slightly lower values were observed at Doda Pilii and intermediate concentrations at Valea Vinului. The lowest Fe levels were recorded at Groșii Țibleșului, remaining below 100 mg kg^{-1} DW. Although Fe is an essential element involved in electron transport processes and chlorophyll synthesis, its accumulation in fruits is generally limited because iron is preferentially retained in vegetative tissues such as leaves and roots. This pattern is consistent with previous studies on *V. vitis-idaea*, where Fe concentrations were substantially higher in leaves, stems and roots than in reproductive organs, indicating restricted translocation of Fe toward fruits (Kobayashi & Nishizawa, 2012; Kandziora-Ciupa et al., 2017).

Manganese (Mn) concentrations were highest at Valea Vinului and Bilbor, approaching 90-100 mg kg^{-1} DW (Figure 3b), while the lowest values were observed at Groșii Țibleșului (40-50 mg kg^{-1} DW). These concentrations are in line with previously reported Mn levels in lingonberries (Rodushkin et al., 1999) (approximately 87-160 mg kg^{-1} DW). Manganese plays an essential role in photosynthetic processes and is often more strongly accumulated in vegetative organs than in fruits in *V. vitis-idaea*.

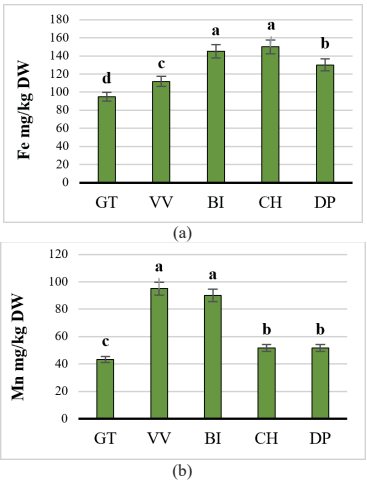


Figure 3. Micronutrient concentrations in *V. vitis-idaea* fruits: (a) iron (Fe); (b) manganese (Mn). Mean ± SD (n = 3). Different letters indicate significant differences ($p < 0.05$)

Zinc (Zn) concentrations ranged between approximately 10 and 18 mg kg^{-1} DW (Figure 4a). The highest levels were recorded at Bilbor and Doda Pilii, while intermediate concentrations were observed at Valea Vinului and Groșii Țibleșului. The lowest Zn content was found at Cheia, with values close to 10 mg kg^{-1} DW. These values fall within the range commonly reported for plant tissues, where adequate Zn concentrations are typically around 15-20 mg kg^{-1} DW (Broadley et al., 2007). Zinc is an essential micronutrient involved in numerous enzymatic and metabolic processes in plants, and differences in its concentration may reflect variations in soil availability and environmental conditions among the sampling sites (Jungová et al., 2021).

Copper (Cu) concentrations ranged from less than 1 to about 6.5 mg kg^{-1} DW (Figure 4b). The

highest value was measured at Bilbor, followed by slightly lower concentrations at Valea Vinului and Cheia, while the lowest Cu content was recorded at Doda Pilii. The generally low concentrations observed are consistent with the limited mobility of Cu in plants and its tendency to accumulate mainly in vegetative tissues rather than fruits. Copper plays an important role in plant metabolism as a cofactor of several redox enzymes involved in photosynthesis and oxidative stress regulation. Comparable Cu levels have been reported for *V. vitis-idaea* fruits, ranging from approximately 0.29 to 1.14 mg kg^{-1} fresh weight (Drózdz et al., 2018).

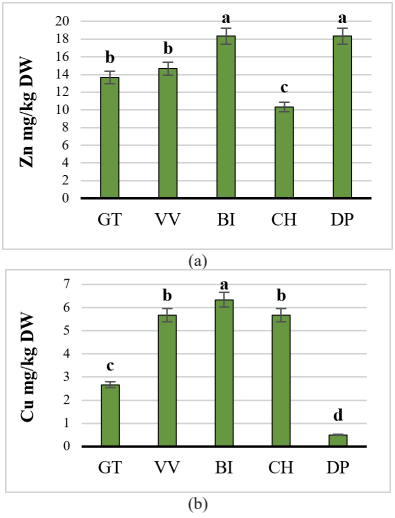


Figure 4. Micronutrient concentrations: (a) zinc (Zn); (b) copper (Cu). Mean ± SD (n = 3). Different letters indicate significant differences ($p < 0.05$)

Trace element content

Nickel (Ni) concentrations were highest at Grosii Țibleșului, reaching approximately 1.1 mg kg^{-1} DW, while lower values were observed at Cheia (Figure 5a). In the other sampling sites, Ni concentrations were below the limit of detection (LOD) of the analytical method. These concentrations are lower than those previously reported for lingonberries, where Ni levels ranged from 1.81 to 12.9 mg kg^{-1} DW (Zeiner & Juranović Cindrić, 2018). Nickel occurs in plants at trace levels and functions as a cofactor of urease involved in nitrogen metabolism, while its accumulation in fruits is generally limited.

Strontium (Sr) concentrations were highest at Valea Vinului, and lower levels were observed at Bilbor while the other locations were below the limit of detection (Figure 5b). Strontium behaves chemically similar to calcium and can enter plant tissues through the same uptake pathways. Consequently, differences in Sr concentrations among sites may reflect variations in the geochemical composition of the underlying substrates and soil mineralogy (Capo et al., 1998; Burger & Lichtscheidl, 2019).

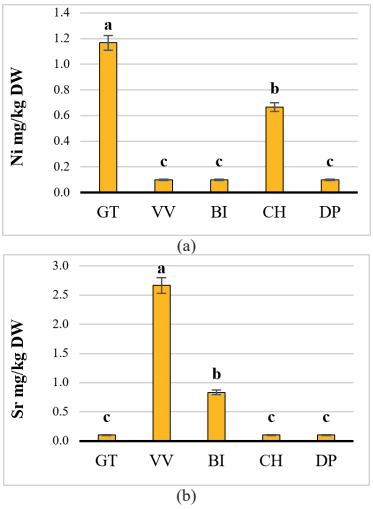


Figure 5. Trace element content in lingonberry fruits: (a) nickel (Ni); (b) strontium (Sr). Mean ± SD (n = 3). Different letters indicate significant differences ($p < 0.05$)

Rubidium (Rb) content was highest at Doda Pilii, reaching approximately 24-25 mg kg^{-1} DW (Figure 6). Lower values were recorded at Grosii Țibleșului, Valea Vinului, and Bilbor, whereas Rb concentrations at Cheia were below the limit of detection (LOD) of the method. Rubidium behaves chemically similar to potassium and can be absorbed by plants through the same transport pathways. Consequently, variations in Rb concentrations among sampling sites are likely influenced primarily by differences in soil geochemistry rather than plant metabolic requirements. Elevated Rb levels, such as those observed at Doda Pilii, may therefore reflect differences in the mineral composition of the underlying substrate, particularly the presence of Rb-bearing silicate minerals in the soil (Kabata-Pendias & Pendias, 1992).

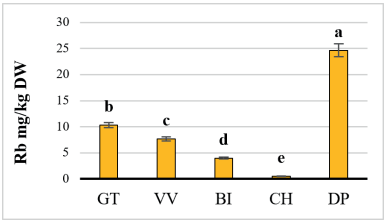


Figure 6. Rubidium (Rb) content in lingonberry fruits. Mean ± SD (n = 3). Different letters indicate significant differences ($p < 0.05$)

Several additional elements were detected sporadically and at very low concentrations. Arsenic and bromine were identified only in samples from Groșii Țibleșului, while molybdenum was detected only in Cheia. Given their rare occurrence and low concentrations, these elements were not included in further analyses.

Multivariate analysis of mineral composition

The hierarchical clustering analysis highlighted clear differences in the mineral profiles of *V. vitis-idaea* fruits among the sampling sites (Figure 7). Bilbor and Doda Pilii formed a closely related cluster characterized by relatively higher levels of K, Zn and Rb, indicating similar elemental signatures in the fruits collected from these locations. In contrast, Valea Vinului was clearly separated from the other sites due to elevated Sr concentrations, suggesting a distinct geochemical influence on the mineral composition of the berries.

A different elemental pattern was observed at Groșii Țibleșului, where comparatively higher Ni levels contributed to its separation from the other sampling sites. These clustering patterns indicate that the mineral composition of lingonberry fruits varies substantially among locations and likely reflects differences in local soil geochemistry and environmental conditions. Similar site-dependent variability in elemental composition of lingonberries has been reported in previous studies (Kabata-Pendias & Pendias, 1992; Rodushkin et al., 1999).

Principal component analysis (PCA) provided further insight into the relationships between mineral elements and environmental variables influencing the composition of *V. vitis-idaea* fruits (Figure 8). The first principal component (PC1), explaining 42.24% of the total variance, was associated with soil moisture, temperature and precipitation, together with higher contributions from K, Zn and Rb, suggesting a potential relationship between environmental conditions and the observed elemental variability. The alignment of these variables may indicate that soil water availability could influence the mobility and transport of these elements in the soil, potentially facilitating their uptake by plants. This relationship is particularly plausible for potassium and rubidium, which are known to be highly mobile in the soil solution and are commonly transported to plant roots through water-driven mass flow processes (Kabata-Pendias & Pendias, 1992). The position of Doda Pilii along this axis reflects higher contributions of these elements, suggesting that both climatic conditions and local substrate characteristics may influence nutrient accumulation in the berries. Differences in altitude among the sampling sites may also contribute indirectly to this pattern by influencing local climatic conditions such as temperature, precipitation and soil moisture.

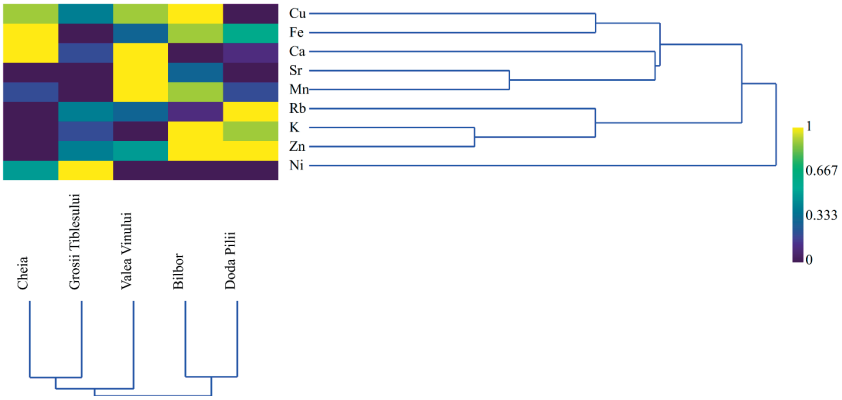


Figure 7. Heatmap and hierarchical clustering of mineral elements in *V. vitis-idaea* fruits from five sampling sites

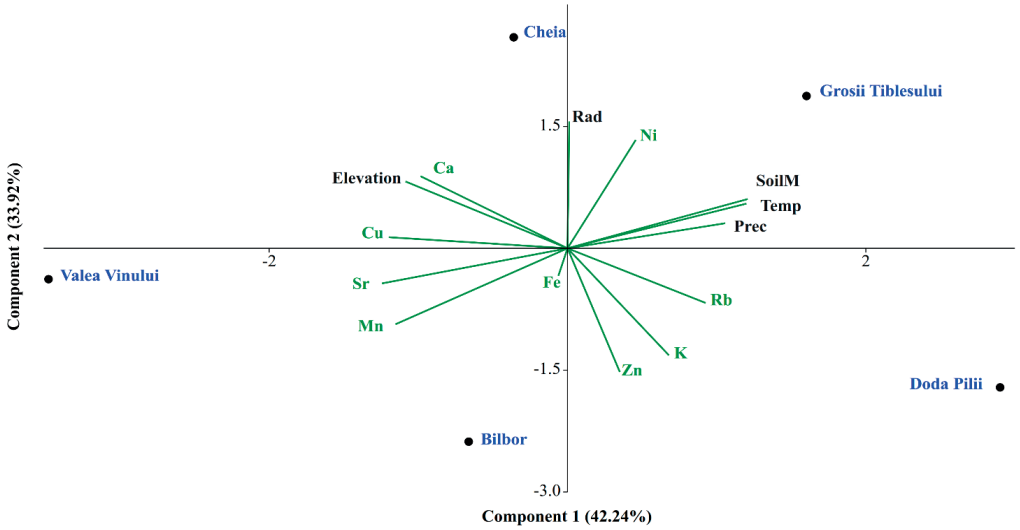


Figure 8. PCA biplot of mineral elements, environmental variables, and sampling sites in lingonberry fruits

The second principal component (PC2), accounting for 28.32% of the variance, was mainly associated with Ni, Ca and solar radiation, while Sr, Mn and Cu were oriented in the opposite direction. The separation of Sr from Ca, despite their chemical similarity, suggests that strontium accumulation in lingonberry fruits may be influenced more strongly by geochemical characteristics of the substrate than by plant physiological regulation. Considering the lithological differences among the studied sites, including sedimentary substrates at Grosii Țibleșului and Cheia and metamorphic substrates at Valea Vinului, Bilbor and Doda Pilii, the observed elemental patterns may partly reflect differences in the mineral composition of soils derived from these parent materials. Sites developed on similar lithological backgrounds may therefore exhibit comparable geochemical influences on element availability, while differences between sedimentary and metamorphic substrates may contribute to the variability observed in the mineral composition of the berries. The PCA indicates that the elemental profile of *V. vitis-idaea* fruits reflects the combined influence of climatic gradients, altitude-related environmental variation and lithological characteristics of the habitats, a pattern also observed in studies investigating mineral composition of wild berries using multivariate approaches (Vilkickyte & Raudone, 2021; Amundsen et al., 2023)

CONCLUSIONS

This study provides new insights into the mineral composition of *V. vitis-idaea* fruits collected from different mountainous regions of Romania and highlights the influence of local environmental conditions on elemental accumulation in berries. The results confirm that potassium is the dominant macronutrient in lingonberry fruits, while other macro- and micronutrients occur in moderate concentrations consistent with previously reported ranges for the genus *Vaccinium*. The observed differences in elemental composition among sites indicate that the mineral profile of lingonberry fruits reflects site-specific environmental and geochemical conditions. In particular, the multivariate analyses revealed distinct elemental signatures associated with certain locations, suggesting that both climatic conditions and local substrate characteristics may influence nutrient accumulation in berries. The study also highlights the relevance of trace elements such as rubidium, strontium and nickel as potential indicators of geochemical variability in lingonberry habitats. Among these, rubidium showed the most pronounced spatial variation, which may be related to differences in soil mineral composition and the chemical similarity between rubidium and potassium during plant uptake. These findings indicate that the mineral composition of *V. vitis-idaea* fruits

reflects the combined influence of climatic gradients and lithological background across mountain habitats. The integration of elemental profiles with environmental variables highlights the potential of wild lingonberries as indicators of local geochemical conditions in natural ecosystems.

Although the present study provides useful baseline information on the mineral composition of wild lingonberry fruits from different mountain habitats, further studies including a larger number of sampling sites and complementary analyses of soil properties would help to better clarify the environmental and geochemical factors influencing elemental accumulation in the berries. Such approaches could contribute to a more comprehensive understanding of the relationships between habitat characteristics and mineral profiles in wild *Vaccinium* populations.

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