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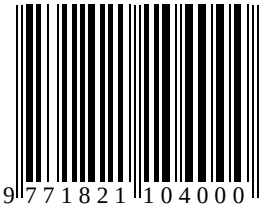

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SPATIAL DISTRIBUTION AND CONCENTRATION OF NICKEL (NI) IN ORGANIC HORIZON AND SURFACE MINERAL SOIL OF FOREST ECOSYSTEMS IN THE REPUBLIC OF SERBIA

Jelena BOŽOVIĆ[1], Jovana CVETKOVIĆ[1], Bojan KONATAR[1], Vanja STOJANOVIĆ[1], Snežana KONATAR[2], Aleksandar VEMIĆ[1], Dragana ŽIVOJINOVIĆ[3]*

Abstract: *This scientific paper investigates the spatial distribution and concentration of nickel (Ni) in the organic layer and surface soil (0-10 cm) of forest ecosystems in the Republic of Serbia. The aim of the research is to determine the influence of regional affiliation (Eastern, Western, Northern, Southern, and Central Serbia) and altitude on nickel accumulation in these forest soil components. Samples of organic layer and soil were collected from representative locations across Serbia. Nickel content was determined after digestion of the samples with aqua regia, and concentrations were analysed using inductively coupled plasma mass spectrometry (ICP-MS). It is expected that the results will show significant variations in nickel content depending on geographical location and altitude, which may be related to the geological substrate, specific pedogenetic processes, climatic conditions, and anthropogenic influences. The significance of the research lies in assessing the potential risk of nickel toxicity to flora, microorganisms, and other ecosystem components, as well as contributing to the understanding of metal biogeochemical cycles in forest soils. The obtained data will serve as a basis for future research and monitoring of soil quality in forest ecosystems in Serbia.*

Keywords: Forest soil, Organic horizon, Nickel, Regional distribution, ICP-MS

PROSTORNA DISTRIBUCIJA I KONCENTRACIJA NIKLA (NI) U ORGANSKOJ PROSTIRCI I POVRŠINSKOM SLOJU ZEMLJIŠTA ŠUMSKIH EKOSISTEMA REPUBLIKE SRBIJE

Apstrakt: *Ovaj naučni rad istražuje prostornu distribuciju i koncentraciju nikla (Ni) u organskoj prostirci i površinskom sloju zemljišta (0-10 cm) šumskih ekosistema u Republici Srbiji. Cilj istraživanja je utvrditi uticaj regionalne pripadnosti (Istočna, Zapadna, Severna, Južna i Centralna Srbija) i nadmorske visine na akumulaciju nikla u ovim komponentama šumskog zemljišta. Uzorci organske prostirke i zemljišta sakupljeni su sa reprezentativnih lokacija širom Srbije. Sadržaj nikla određen je nakon digestije uzoraka carskom vodom, a koncentracije su analizirane korišćenjem induktivno kuplovane plazme sa*

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masenom spektrometrijom (ICP-MS). Očekuje se da će rezultati pokazati značajne varijacije u sadržaju nikla u zavisnosti od geografskog položaja i nadmorske visine, što može biti povezano sa geološkom podlogom, specifičnim pedogenetskim procesima, klimatskim uslovima i antropogenim uticajima. Značaj istraživanja leži u proceni potencijalnog rizika od toksičnosti nikla za biljni svet, mikroorganizme i druge komponente ekosistema, kao i u doprinosu razumevanju biogeohemijskih ciklusa metala u šumskim zemljištima. Dobijeni podaci će služiti kao osnova za buduća istraživanja i monitoring kvaliteta zemljišta u šumskim ekosistemima Srbije

Ključne reči: Šumsko zemljište, Organska prostirka, Nikl, Regionalna distribucija, ICP-MS

1. INTRODUCTION

Soil is a fundamental component of forest ecosystems, providing physical support for plants, acting as a reservoir for water and nutrients, and serving as habitat for numerous organisms. The quality of soil directly influences forest productivity and health. Among the many chemical elements present in soil, heavy metals including nickel (Ni) draw significant attention due to their potential toxicity to living organisms and their capacity for accumulation within various ecosystem components. Nickel is an element found in the Earth's crust, and its soil content depends on the geological substrate (Kabata-Pendias & Pendias, 2001). However, anthropogenic activities such as industry (notably metallurgy), transportation, agriculture (use of fertilizers and pesticides), and fossil fuel combustion can substantially increase nickel concentrations in the environment, including forest soils (Alloway, 2013).

The organic horizon, as the first surface layer of soil (comprising the Ol, Of, and Oh subhorizons according to forestry pedology classification), plays a vital role in biogeochemical processes, including the cycling of heavy metals. It acts as a reservoir for metals deposited from the atmosphere (dry and wet deposition) or leached from tree canopies. Through the decomposition of organic matter, heavy metals are gradually released into the mineral soil fraction, increasing their bioavailability to plants and microbes. The surface soil layer (0–10 cm) is the most active part of the soil profile, rich in organic matter and microorganisms, and consequently most susceptible to heavy metal accumulation due to intense sorption, complexation, and biological transformation processes (Sparks, 2003).

Geographical location and altitude significantly influence the distribution and accumulation of heavy metals in soil. Different geological substrates across Serbia naturally contain varying levels of nickel (Manojlović et al., 2012). Climate conditions—varying with geographic position and elevation affect pedogenic processes, leaching, and element accumulation in soils (Buol et al., 2011). Additionally, population density and industrial activity vary regionally, leading to differences in anthropogenic nickel input (Saljnikov et al., 2009).

Previous studies have shown that soil metal content can vary considerably depending on region and altitude (Alloway, 2013). Specific research in Serbia indicates variability in metal concentrations in forest soils based on location and elevation (Saljnikov et al., 2009). However, a comprehensive analysis of nickel content in the organic horizon and surface mineral soil across Serbian forest

ecosystems – focusing on regional differences and elevation effects has not yet been systematically conducted to enable thorough assessments and identify potential risk areas.

The aim of this study is to determine the total nickel content in the organic horizon and surface mineral soil (0–10 cm) of forest ecosystems in Serbia, analysing the influence of regional affiliation (Eastern, Western, Northern, Southern, Central Serbia) and altitude on its concentration. The results will improve understanding of the biogeochemical cycle of nickel in Serbian forests and provide a basis for ecological risk assessment related to elevated nickel levels, supporting informed decision-making in environmental protection and sustainable forest management.

Nickel is an essential trace element for plants at very low concentrations, involved in activating specific enzymes such as urease (Marschner, 2012). However, at elevated levels, nickel becomes toxic. High soil nickel concentrations can inhibit plant growth, cause chlorosis, necrosis, and other toxicity symptoms, as well as disrupt biochemical processes in plants (Kabata-Pendias & Pendias, 2001). Nickel can also accumulate in plant tissues and enter the food chain, posing potential risks to animals and humans. Furthermore, elevated nickel levels can adversely affect soil microbial activity, which is crucial for organic matter decomposition, nutrient cycling, and soil structure formation (Giller et al., 1998). Therefore, monitoring and understanding the distribution of nickel in soils are vital for ecosystem health assessment and sustainable forest management, especially in the context of climate change and increasing anthropogenic pressures.

2. MATERIAL AND METHODS

Samples of organic matter and soil were collected from representative sites across Serbian forest ecosystems. Locations were carefully selected to cover five major geographic regions (Eastern, Western, Northern, Southern, Central Serbia) and a broad range of altitudes (approximately 100 m to over 1500 m above sea level). At each site, samples of humified organic horizon (Oh) and organic-mineral soil layer (0–10 cm) were collected. Multiple sub-samples were taken within a radius of 10–20 meters around a central point at each site, then homogenized to produce a representative composite sample. In total, 89 organic matter samples and 115 soil samples were collected.

Collected samples were transported to the laboratory, where they were air-dried until reaching a constant weight. Soil samples were sieved through a 2 mm mesh to remove coarse fragments and visible debris. Organic samples were ground in a plant material mill and sieved through a 0.25 mm mesh. Both soil and organic samples were then milled into fine powder (<100 μm) using an agate mortar, ensuring complete homogenization for chemical analysis.

Total nickel content was determined after acid digestion. Approximately 0.2 g of dried, ground sample was digested in a mixture of concentrated nitric acid and hydrochloric acid (aqua regia) in a 1:3 ratio, using a Milestone Ethos Easy microwave digestion system in closed Teflon vessels. After digestion, the solutions were filtered and diluted to 100 ml with deionized water. Nickel concentrations were measured via inductively coupled plasma mass spectrometry (ICP-MS, PerkinElmer NexION 1000G). Multi-element standard solutions of known concentration were

used for calibration. Results are expressed in milligrams per kilogram of dry weight (mg/kg).

Data were processed using Microsoft Excel, enabling distribution analysis of nickel in the organic horizon and soil across Serbia and by individual regions.

3. RESULTS AND DISCUSION

The findings reveal significant spatial variability in nickel concentrations in both the organic horizon and the surface mineral soil of Serbian forest ecosystems. Graphical representations (Figures 1 and 2) illustrate the heterogeneity of nickel distribution across regions.

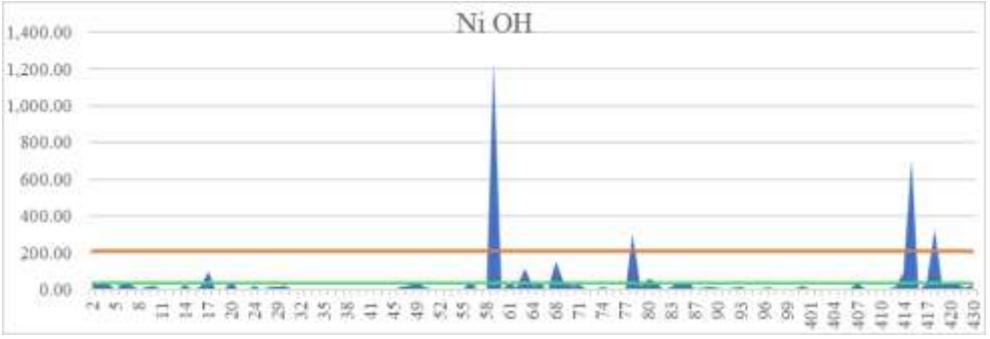

Figure 1. *Ni Content in the Organic Horizon across Serbia*

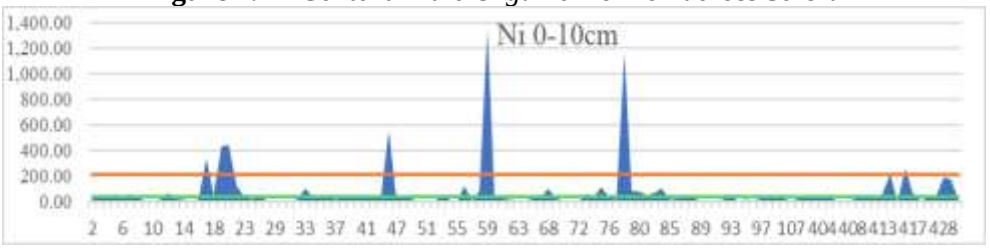

Figure 2. *Ni Content in the Surface Soil Layer (0–10 cm) across Serbia*

Comparison of nickel levels in the organic horizon and mineral soil highlights the higher sorption capacity of the organic layer, attributable to organic matter's ability to form complexes with metal ions. Consequently, nickel concentrations are generally higher in the organic horizon, especially at sites with significant anthropogenic input or geologically rich in nickel-bearing ultramafic rocks. The ratio of nickel in organic matter to mineral soil indicates the metal's mobility and transport dynamics within the soil profile.

In the organic horizon, nickel concentrations ranged from below detection limits to extremely high values. Spatial distribution maps (Figures 3–5) demonstrate elevated concentrations in Western and Southern Serbia. Western Serbia exhibits the most extreme values, with some points reaching 1235 mg/kg (Point 59), 711 mg/kg (Point 415), 328 mg/kg (Point 418), and 99 mg/kg (Point 17), indicating local sources of nickel accumulation, likely from ultramafic geology.

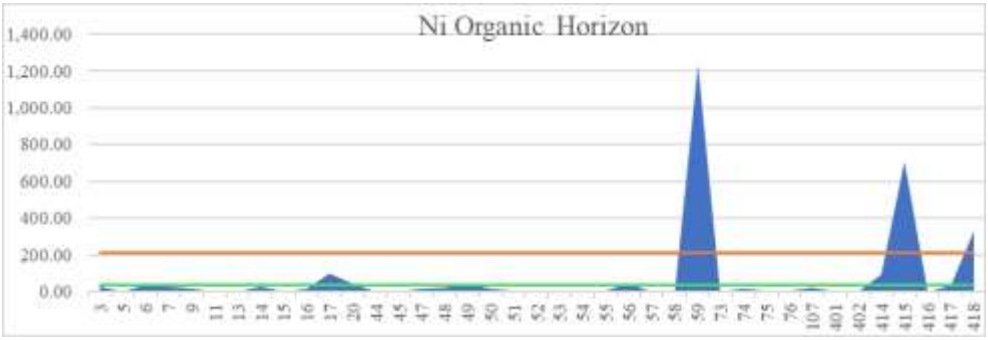

Figure 3. *Ni Content in Organic Horizon in western Serbia*

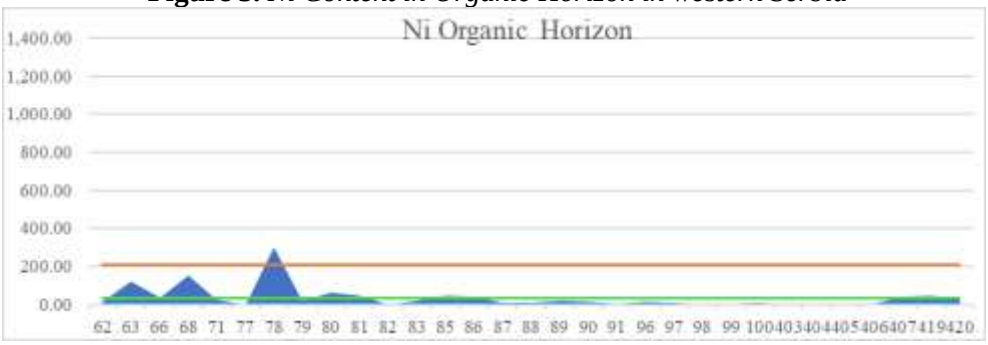

Figure 4. *Ni Content in Organic Horizon in southern Serbia and Content in the Surface Soil Layer (0–10 cm) across Serbia*

Southern Serbia also shows locations with elevated nickel concentrations (Point 78 with 301.43 mg/kg, Point 68 with 152.99 mg/kg, Point 63 with 119.03 mg/kg). Northern and Central Serbia generally show lower nickel concentrations in the organic horizon, although some variability exists. Lower nickel levels are associated with the geological substrate, which is predominantly composed of sedimentary rocks. Additionally, average nickel concentrations in the organic horizon significantly vary between regions, with a tendency toward higher values in western and southern Serbia.

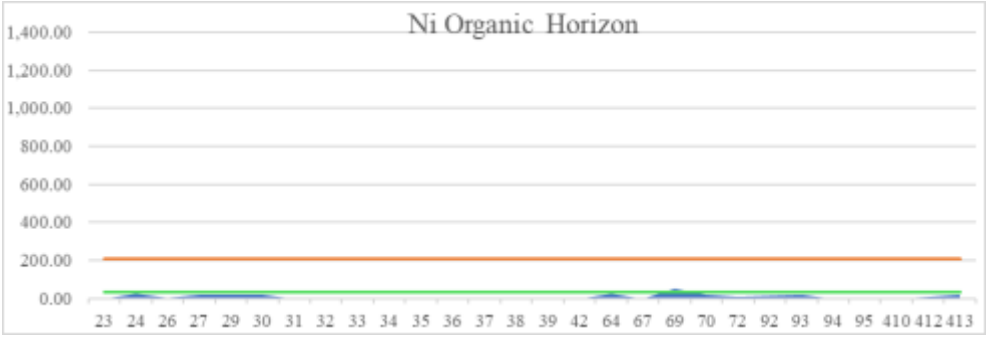

Figure 5. *Ni Content in Organic Horizon in eastern Serbia*

High nickel concentrations in the organic horizon at certain sites in Western and Southern Serbia are primarily associated to the geological substrate. It is known that parts of these regions are rich in ultramafic rocks (serpentinites and peridotites),

which naturally contain high levels of nickel. As these rocks weather, they release increased amounts of nickel into the soil. The organic horizon, due to its ability to bind metals through ion exchange and complex formation with organic molecules, can accumulate substantial quantities of nickel. This process is especially pronounced in forest ecosystems, where large amounts of organic material accumulate on the soil surface.

Furthermore, atmospheric deposition should also be considered. Although forest ecosystems in remote areas may be less exposed to direct industrial pollution, the transport of pollutants through the air from distant or local sources (e.g., industrial facilities, mines, traffic) can contribute to nickel accumulation in the organic horizon.

It is also noteworthy points where nickel concentration in the organic horizon is below the detection limit (Points 53, 58, 402 in western Serbia, Points 33, 34, 36, 37, 38, 39, 410 in eastern Serbia, Point 98 and 403 in southern Serbia). Such low Ni values may result from the absence of nickel in the geological substrate from which the soil develops. In some cases, nickel may be present in the soil but in insoluble forms that are inaccessible to plants, preventing uptake. Under these circumstances, nickel does not participate in the material cycling within the ecosystem, as forest trees do not transport it to the soil surface via leaf litter, and thus, it does not accumulate in the topsoil.

Concentration of Nickel in the Surface Soil Layer (0–10 cm)

Nickel concentrations in the surface soil layer also exhibit considerable variability. As shown in the maps depicting the spatial distribution of nickel in the topsoil, western and southern Serbia stand out due to the presence of sites with exceptionally high concentrations. At these locations, values significantly exceed the stipulated remediation threshold of 210 mg/kg such as Point 59 (1346.80 mg/kg), Point 78 (1167.60 mg/kg), Point 45 (560.19 mg/kg), Point 20 (438.67 mg/kg), and Point 19 (431.32 mg/kg). These extremely high values indicate that the fundamental functions of the soil concerning nickel are severely disrupted at these sites, necessitating urgent remediation, reclamation, and other measures.

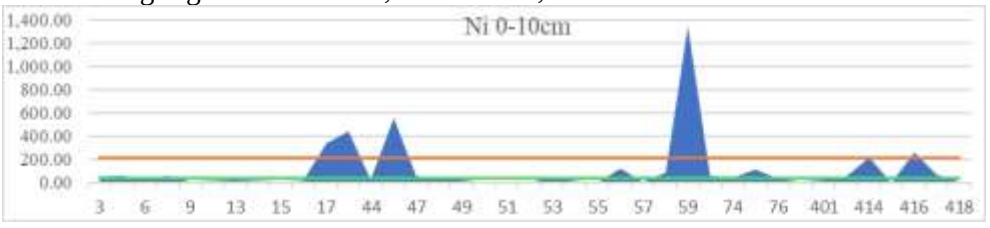

Figure 6. *Ni Content in the surface soil layer in the area of western Serbia*

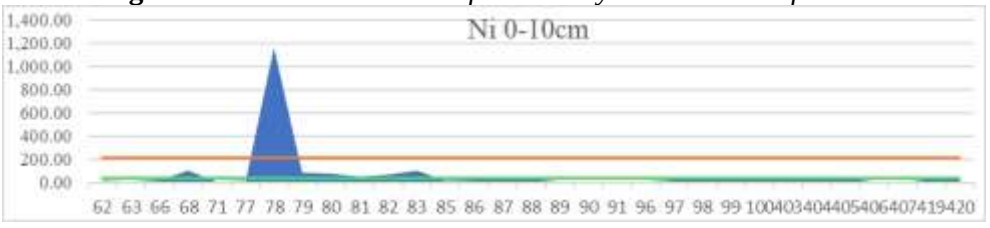

Figure 7. *Ni Content in the surface soil layer in the area of southern Serbia*

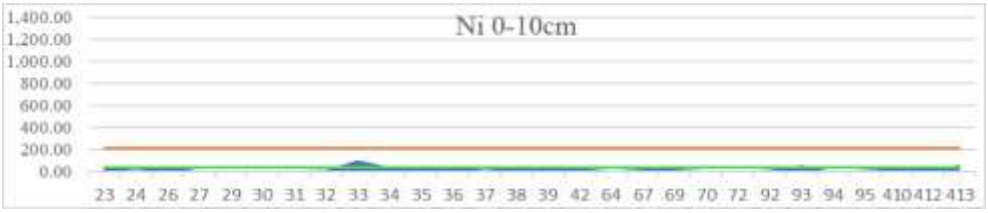

Figure 8. *Ni Content in the surface soil layer in the area of eastern Serbia*

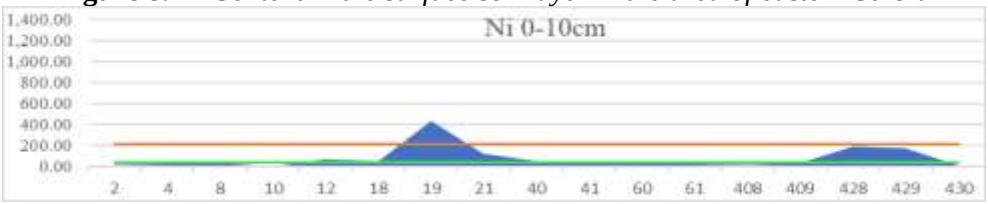

Figure 9. *Ni content in the surface layer of the soil in the area of central Serbia*

Comparing nickel concentrations in the organic horizon and the 0–10 cm mineral soil layer at the same locations can provide insights into the biogeochemical cycle of nickel. In some sites with high levels in the organic horizon, the soil also shows high nickel values (e.g., Points 59, 78, 20), suggesting that nickel from organic matter is being transported into the mineral soil layer through organic mineralization and leaching. However, at other sites, the relationship between concentrations in the organic horizon and the mineral soil can differ, which may be due to specific pedogenetic processes, soil pH (which affects nickel mobility and availability lower pH increases mobility), organic matter content in the mineral layer (which can bind nickel if humus is present or convert it into soluble forms if humic substances are abundant), and other factors influencing nickel mobility and retention in the soil.

The analysis shows that average nickel concentrations in the surface soil layer are also highest in western and southern Serbia, although intra-regional variability is significant. It is already evident that western and southern Serbia contain the largest number of sites with notably elevated nickel levels, likely due to the dominant influence of geological substrates, particularly ultramafic rocks.

Regarding the minimum threshold value of 35 mg/kg, many sites across all regions exceed this limit in both the organic horizon and the surface mineral soil. Based on Maps 1 and 2, it is clear that many points indicate an exceedance of this value. This is a positive indicator, as it signifies that the functional properties of the soil its sustainable quality have been fully achieved. Elevated nickel concentrations, even below remediation thresholds, can negatively impact sensitive plant species, soil microorganisms, and biogeochemical cycles, potentially threatening forest ecosystem health.

The effect of altitude on nickel content is complex and depends on the interaction of multiple factors. At lower elevations, anthropogenic influences such as emissions from industry and transportation are more pronounced, potentially leading to higher nickel accumulation. As elevation increases, the influence of anthropogenic sources diminishes; however, climatic conditions (temperature, precipitation) and soil types change, affecting leaching, accumulation, and nickel binding processes. In higher mountainous areas, where pedogenic processes are slower and organic matter decomposes more gradually, nickel tends to remain longer

in the organic horizon. Additionally, vegetation type and soil pH both of which vary with altitude also influence nickel mobility and bioavailability.

5. CONCLUSION

Based on the analysis of the obtained results, it can be concluded that the spatial distribution and concentration of nickel in the organic horizon and surface mineral soil of Serbian forest ecosystems show significant variability. Maps clearly illustrate regional differences, with the highest concentrations recorded in western and southern Serbia. These elevated levels are primarily related to naturally high nickel content in the geological substrate, especially ultramafic rocks.

Regional analysis of nickel content revealed notable differences. Areas with geological formations rich in nickel such as serpentinites and peridotites exhibit higher soil nickel concentrations. The parts of western and southern Serbia, known for these geological features, show higher nickel levels compared to other regions. Conversely, eastern Serbia, with predominantly sedimentary and acidic silicate rocks, exhibits lower nickel concentrations. Anthropogenic factors, such as proximity to industrial zones or busy roads, also contribute to increased nickel levels in certain regions.

A considerable number of sites in western and southern Serbia show surface soil nickel concentrations exceeding the remediation threshold, indicating potential serious disruption of soil functions and the need for risk assessment and possible remediation measures. Many sites across all regions also surpass the limit value for nickel, suggesting that soil quality at these locations may not meet regulatory standards and could affect sensitive ecosystem components.

Besides the geological substrate, pedogenetic processes, soil properties (pH, chemical nature of organic matter), and atmospheric deposition influence the spatial distribution of nickel. Further analysis incorporating correlations with altitude and other soil parameters, as well as detailed spatial modelling will provide deeper insights into the factors controlling nickel distribution.

High nickel concentrations in the organic horizon and surface soil layers of forest ecosystems pose potential ecological risks. The migration of nickel into deeper soil layers and groundwater also represents a broader environmental concern.

Identifying sites with elevated nickel levels is crucial for directing future research and monitoring efforts. Additional studies should focus on assessing the bioavailability of nickel at these locations and analysing its impacts on plant life and soil microorganisms within Serbian forest ecosystems.

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SPATIAL DISTRIBUTION AND CONCENTRATION OF NICKEL (NI) IN ORGANIC HORIZON AND SURFACE MINERAL SOIL OF FOREST ECOSYSTEMS IN THE REPUBLIC OF SERBIA

Jelena BOŽOVIĆ, Jovana CVETKOVIC, Bojan KONATAR, Vanja STOJANOVIĆ, Snežana KONATAR, Aleksandar VEMIĆ, Dragana ŽIVOJINOVIĆ

Summary

This scientific study investigated the spatial distribution and concentration of nickel (Ni) in the organic layer and the surface mineral soil layer (0–10 cm) of forest ecosystems across the Republic of Serbia. The aim was to determine the influences of regional differences, including eastern, western, northern, wouthern, and central Serbia, as well as differences in altitude, on nickel accumulation in these soil layers. Samples were collected from representative locations throughout the country, with nickel content determined through digestion with aqua regia and using ICP-MS (Inductively Coupled Plasma Mass Spectrometry), a method that offers high precision and sensitivity in element detection.

The results revealed significant regional variability in nickel concentrations. The highest levels were recorded in western and southern Serbia, consistent with the geological characteristics of these areas, known for rich ultramafic rocks such as serpentinites and peridotites, which naturally contain high levels of nickel. These regions are particularly susceptible to elevated nickel levels due to geological sources, but anthropogenic factors such as industrial activities, transportation, urbanization, and atmospheric deposition also contribute to these concentrations. In the organic soil layer, nickel levels were generally higher than in the mineral layer, owing to its ability to bind with organic matter through complexation and ion exchange, leading to increased accumulation in surface soils. Some locations exhibited nickel levels exceeding regulatory thresholds, indicating potential risks to soil quality, plant life, and microbial communities.

Across Serbia, nickel levels in soils depend on various factors, including the underlying geology, pedogenetic processes, soil properties (such as pH and organic matter content), and atmospheric influences. The complex relationship between altitude and these

factors further affects nickel mobility and accumulation, with different distribution patterns observed at varying elevations. High levels of nickel in surface soil layers can pose serious ecological risks, disrupting plant and microbial communities. Additionally, there is a potential for nickel to migrate into deeper soil layers and groundwater, representing a further environmental threat to underground water sources and overall ecosystem health. These findings underscore the importance of continuous monitoring and analysis of nickel levels in forest soils of Serbia, especially in ultramafic regions at higher risk of contamination.

In conclusion, this research contributes to a better understanding of the biogeochemical cycles of nickel in Serbia and provides a basis for developing strategies for sustainable land management and soil protection. Identified locations with elevated nickel concentrations require further studies and remediation measures to preserve soil functionality and ecosystem health. The data obtained contribute to an improved understanding of the biogeochemical cycling of nickel in Serbia and serve as a foundation for future monitoring efforts and the sustainable management of forest resources.

PROSTORNA DISTRIBUCIJA I KONCENTRACIJA NIKLA (NI) U ORGANSKOJ PROSTIRCI I POVRŠINSKOM SLOJU ZEMLJIŠTA ŠUMSKIH EKOSISTEMA REPUBLIKE SRBIJE

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Rezime

U ovom radu istražena je prostorna distribucija i koncentracija nikla (Ni) u organskom sloju i površinskom mineralnom sloju zemljišta (0–10 cm) šumskih ekosistema u Republici Srbiji. Cilj je bio da se utvrde uticaji regionalnih razlika (Istočna, Zapadna, Severa, Južna i Centralna Srbija) i nadmorske visine na akumulaciju nikla u ovim slojevima zemljišta. Uzorci su prikupljeni sa reprezentativnih lokacija širom zemlje, a sadržaj nikla određen je digestijom uzoraka carskom vodom i analizom pomoću ICP-MS metode.

Rezultati pokazuju značajnu regionalnu varijabilnost, pri čemu su najviši nivoi nikla zabeleženi u Zapadnoj i Južnoj Srbiji, oblastima poznatim po bogatim ultramafičnim stijenama (serpentinitima i peridotitima) koje prirodno sadrže visok nivo nikla. Visoke koncentracije nikla u ovim regionima uglavnom su posledica geoloških izvora, ali na njih utiču i antropogeni faktori, poput industrijskih aktivnosti i atmosferskog depozicije. U organskom sloju, nivo nikla je uglavnom viši nego u mineralnom sloju, zbog njegove sposobnosti da se vezuje za organsku materiju putem kompleksacije i jona razmene. Neke lokacije pokazuju nivo nikla koji premašuje zakonske pragove, što ukazuje na potencijalne rizike po kvalitet zemljišta, biljni svet i mikroorganizme.

Na nivou Srbije, nivo nikla u zemljištu zavisi od geološke podloge, pedogenetskih procesa, svojstava zemljišta (pH, sadržaj organske materije) i atmosferskih uticaja, pri čemu složen odnos između nadmorske visine i ovih faktora dodatno utiče na mobilnost i akumulaciju nikla. Visoki nivoi nikla u površinskom sloju zemljišta mogu ugroziti zdravlje ekosistema, oštećujući biljni i mikrobiološki svet, kao i mogućnost transporta nikla na dublje slojeve i u podzemne vode.

Zaključno, istraživanje ističe važnost praćenja i analize nivoa nikla u zemljištima šumskih ekosistema Srbije, naročito u oblastima sa ultramafičnom geologijom. Identifikovane lokacije sa visokim koncentracijama nikla zahtevaju dodatne studije i moguće mere sanacije, kako bi se zaštitila funkcionalnost zemljišta i zdravlje celokupnog ekosistema. Dobijeni podaci doprinose boljem razumevanju biogeohemijskih ciklusa nikla u Srbiji i predstavljaju osnovu za buduće monitoring aktivnosti i održivo upravljanje šumskim resursima.