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Impact of drying, freezing and re-wetting events soil leachate in acidic *versus* calcareous soils

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Abstract: This study investigates the impact of drying-rewetting and freezing--rewetting events on soil leachate ion composition across two contrasting geochemical settings through a series of controlled laboratory experiments. Dissolution of ions (Na^+, K^+, Ca^{2+}, Mg^{2+}, Al^{3+}, Fe^{3+}, Mn^{2+}, F^-, Cl^-, NO_2^-, SO_4^{2-}, NO_3^-, PO_4^{3-}) in soil leachate was analysed following rewetting cycles after drying and freezing treatments. The results indicate that variations in leachate ion concentrations are primarily influenced by bedrock type, while drying-rewetting and freezing-rewetting treatments did not significantly impact overall variance. However, some inconsistent differences were observed: higher K^+ concentrations in calcareous soils and Al^{3+}, Fe^{3+} and Mn^{2+} in acidic soils after drying, higher anion concentrations in calcareous soils in both treatment leachates compared to controls. The findings highlight that the effects of drying, freezing, and rewetting are inherently linked to treatment type, ion characteristics and geochemical conditions.

Keywords: ions; forest soil; laboratory experiment; thermal conditions; limestone; sandstone.

INTRODUCTION

Analysing the composition of soil leachate is important for understanding stress factors affecting forest ecosystems.[1] Soil solution unravels climate change impacts, observed for instance in leaching losses of N.[2] The leaching of nitrate and sulphate ions is associated with the losses of soil base cations like Ca^{2+} and Mg^{2+} affected by increases in soil acidity, which causes potential decrease in forest productivity. Forest disturbance in relation to the climate changes can have significant effect on soil chemical properties, and on a relatively short-term it may

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affect soils' solution.[3] The sediment and nutrient losses increase after freeze-thaw conditions.[4] The similarly of ion availability is known to be affected by seasonal soil warming.[5] The nutrient depletion from soil poses an environmental challenge for tree species, and its underlying factors are not yet fully understood.

Drying, freezing and re-wetting soil processes are controlled by meteorological conditions. Both processes, successional drying and wetting, or wetting, freezing and melting, are on-site processes controlled by the factor of soil temperature change. Soil temperature change accelerates soil dispersion forces and looseness of soil particles, and dissolvability of loosely bonded soil ions which are accordingly leached from soil. How specific ions, regarding different types of soil, site and regional level meteorology, react to alternations in temperature dynamic have not been sufficiently documented.

Limestone (with 61 %) and quartz sandstone (with 30 %) are among the most abundant bedrock types in Bosnia and Herzegovina.[6] Two the most common forest soil types Calcic Cambisol and Dystric Cambisol are formed on these bedrock materials.[7] While soils formed on limestone and dolomite are either calcic, calcareous and eutric, often not containing active carbonates, those formed on quartz reddish Werfen sandstone are highly acidic and dystric. Substantial distinctions are evident concerning their chemical and mineralogical properties. Calcic Cambisol formed on clear limestone (~99 % $CaCO_3$) have clay-like texture and contain montmorillonites, quartz, kaolinite and small amount of hematite.[8] Dystric Cambisol formed on sandstones have sandy texture and they inherit quartz, mica and clay fraction.[9] Although the most of the forest soils in the Dinaric region are not attributed to significant microclimate changes due to the applied close-to-nature silvicultural practices, their thermal stability is moderated by the multi-layered forest canopy.[10] However, this buffering effect may be compromised by the anticipated increase in natural hazards causing large-scale forest disturbances.[11] The ability of soils to retain nutrients is anticipated to change. Hence, it is important to attain deeper understanding about the effect of increased soil temperature fluctuations on the ion dynamic behaviour.

In order to achieve better understanding of factors controlling leachate composition, simulation of soil temperature fluctuations was performed in laboratory conditions. The main hypothesis was that the thermal conditions determine soil leachate composition relative to soil geochemistry. Three main objectives were a) to determine an average leachate composition assuming light rain; b) to identify the main factor of soil leachate variation and c) to test the effect of treatments.

EXPERIMENTAL

Soil sample origin and pretreatment measurements

To analyse soil leachate composition, samples were collected from two contrasting soil and geological types. The soil samples originated from the Dinaric mountain beech and fir forests, developed on clear Triassic limestone on Mt. Bjelašnica (43.72735°N; 18.27205°E) and

Triassic quartz sandstone on Mt. Trebević (43.8332°N; 18.46439°E). The chosen bedrock types represent the most common geological settings in Bosnia and Herzegovina.[12] Soils were field described and named according to World Reference Base from 2022 as Calcaric Cambisol (Humic, Episceletic, Episiltic) with Oligomull humus on limestone (Calcareous soil) and as Dystric Cambisol (Chromic) with Hemimor humus on Werfenic sandstone (acidic sandstone soil, Table I).[13] Four soil samples were collected from soil profile pits of calcareous and acidic sandstone soil at fixed depths from 0 to 10 cm and 10 to 20 cm. Approximately 3 kg of soil was taken from each layer, or 12 kg in total. Total number of composite samples was four: two calcareous and two sandstone soil.

TABLE I. Soil properties of calcareous and acidic sandstone soil (*N* – number of samples, depth of sample in cm, bulk density – *BD* (g cm^{-3}), water field capacity – *FC* (vol. %), texture (ClL – clay loam, S - sandy loam), pH value, carbonates as $CaCO_3$ (%), soil organic carbon (*SOC*), total nitrogen (*TN*), C to N ratio – *C*/*N*; Bedrock, soil type and humus form were determined during site survey and compared with soil map

No.	Soil type	*N*	Depth cm	*BD* g cm^{-3}	*FC* vol. %	Texture US Soil	pH ($CaCl_2$)	$CaCO_3$ %	*SOC* %	*TN* %	*C*/*N*
					Calcareous soil						
1.	Calcaric Cambisol	1	0–10	0.40	44.0	ClL	5.98	3.50	12.1	0.80	15.1
2.	(Limestone)	1	10–20	0.51	40.0	ClL	6.12	3.50	11.3	0.40	28.3
					Acidic sandstone soil						
3.	Dystric Cambisol	1	0–10	1.30	31.8	SL	3.53	0.00	8.01	0.37	21.6
4.	(Sandstone)	1	10–20	1.42	21.5	SL	3.93	0.00	4.90	0.23	21.3

After the transportation to the laboratory, soil samples were manually cleaned, homogenized, and air-dried in preparation for subsequent drying, freezing, and re-wetting treatments. The natural aggregate structure was preserved; samples were not milled, but instead gently ground by hand and sieved through a 4 mm mesh. A portion of each sample was set aside for pre-treatment analysis of soil properties.

Pre-treatment measurements included determining following properties: bulk density (*BD*, gravimetrically), field capacity (*FC*, gravimetrically), soil texture (pipet method with 0.2M Na pyrophosphate), pH value (in 0.01M $CaCl_2$ dilution), amount of $CaCO_3$ (volumetrically, ISO 10693:1995),[14] soil organic carbon (*SOC*, combustion with $K_2Cr_2O_7$, ISO 14235:1998)[15] and total nitrogen (*TN*, Kjeldahl method, ISO 11261:1995)[16] were determined per fixed layer depth prior to further analysis (Table I) at the laboratory of University of Sarajevo – Faculty of Forestry, Sarajevo (Bosnia and Herzegovina).

Experimental design

The experiment was designed to test the impact of drying–rewetting and freezing–rewetting cycles on the composition of soil leachate (Fig. 1). Applying a light drizzle with an intensity of approximately 0.25 mm h^{-1}, the concentrations of leached ions were quantified for each treatment after four rewetting cycles. Equal masses (~110 g) of air-dried calcareous soil (CS) and acidic sandstone soil (AS) were used to fill the porous plastic containers, six per soil type (twelve in total), for subsequent treatments.

The control soil (CS control 0-10, CS control 10-20, AS control 0-10, AS control 10-20) was soil in porous plastic containers left with approximate 40 % of water, corresponding to

average field capacity content for both soils. The soil was left at room temperature approximately 22–23 °C, overnight and re-wetting cycles were performed with the rain intensity of 122 ml during 30 min, *i.e.*, ~0.25 mm h^{-1}.

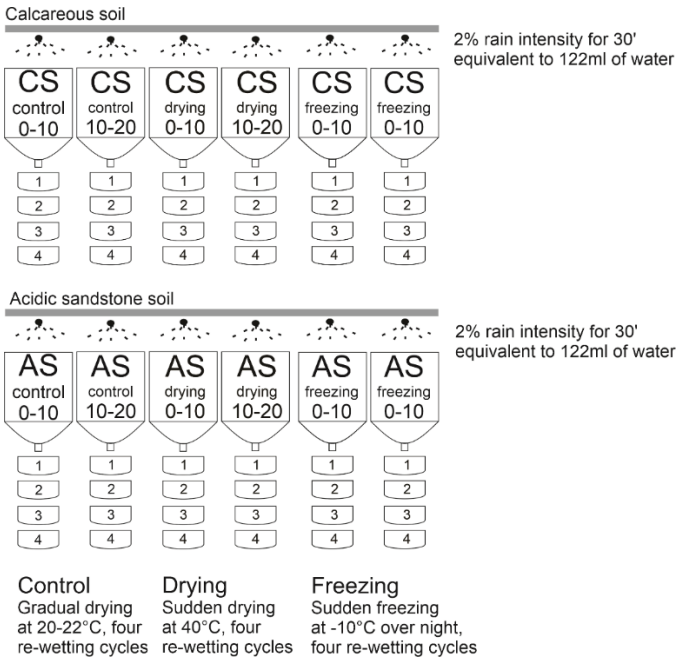

Fig. 1. Experimental design of drying-re-wetting and freezing–re-wetting treatment impact on soil leachate composition. Soil samples are marked as control calcareous (CS control 0-10, CS control 10-20, CS drying 0-10, CS drying 10-20, CS freezing 0-10, CS freezing 10-20) and acidic sandstone soil (AS control 0-10, AS control 10-20, AS drying 0-10, AS drying 10-20, AS freezing 0-10, CS freezing 10-20).

The drying treatment involved sudden drying event of soil sample (CS drying 0-10, CS drying 10-20, AS drying 0-10, AS drying 10-20) in the oven at 35 °C for 4 h, cooling at room temperature and re-wetting with simulated rain intensity of 122 ml in 30 min which corresponded to light rain intensity of ~0.25 mm h^{-1}.

The freezing treatment involved freezing of soil sample (CS freezing 0-10, CS freezing 10-20, AS freezing 0-10, CS freezing 10-20) at –10 °C over night, leaving at room temperature for one hour and re-wetting with the same rainfall intensity of 122ml in 30 min which corresponded to light rain intensity of ~0.25 mm h^{-1}.

Experiment was set up at the Institute of Chemistry, Technology and Metallurgy (ICTM), University of Belgrade (Serbia). All samples in plastic containers were connected to rain-event simulator with funnel so that no water is lost. Soil water leaching from plastic containers was collected and used for further measurements.

Leachate measurements

Leachate (soil water) volume, pH value and electric conductivity (*EC*, µS cm^{-1}) captured after each treatment and control were measured (electrometrically). Concentrations of soil

leached cations (K^+, Ca^{2+}, Mg^{2+}, Na^+, Al^{3+}, Fe^{3+}, Mn^{2+}) were measured on Inductively coupled plasma optical emission spectrometer with axial view (Thermo scientific iCAP 6000 series ICP spectrometer, USA). Concentrations of anions (F^-, Cl^-, NO_2^-, SO_4^{2-}, NO_3^-, PO_4^{3-}) were determined on Dionex ICS 3000, *i.e.*, ion chromatography with conductometric detection. The method refers to is EPA 314.7 (this method applies to anions in water) but at Institute of Chemistry, Technology and Metallurgy in Belgrade it is validated and verified it for anions in soil. Concentrations of cations and anions in soil leachate are given in mg L^{-1}. Ion measurements were made in the ICTM laboratories

Statistical analysis

Descriptive statistical methods were used to present the average composition of the leachate concentrations based on the control ($N = 16$), treatment drying ($N = 16$) and treatment freezing ($N = 16$). Total number of observations was $N = 48$. Soil leachate composition, means and standard deviations were calculated for calcareaus (CS control 0-10, CS control 10-20) and acidic sandstone soil (AS control 0-10, AS control 10-20). Leachate concentrations of ions and anions represent means of two rain cycles. Principal component analysis (PCA) was used to address cumulative variance among variables and the effect of bedrock and treatment. The "prcomp" function from the base R package "stats" was employed for this purpose. Variables were scaled prior to model fit.[17] Analysis were performed in RStudio version 4.3.1.[18]

RESULTS AND DISCUSSION

Soil leachate composition

Determined values of leachate pH, EC and ion concentrations were generally corresponded to those reported in the literature, although some deviations were observed (Table II). The only exception were the pH values in the calcareous soil,

TABLE II. Means and standard deviations (in brackets) of pH values, electrical conductivity (*EC*, μS cm^{-1}) and ion concentrations (mg L^{-1}) in leachate of control calcareous and acidic sandstone soils (combined M10 and M20) compared to typical forest soil ranges according to Blume *et al*. (2016)[19]

Feature	Calcareous soil (M10 and M20)	Acidic sandstone soil (M10 and M20)	Typical range for forest soils
pH	7.07 (0.25)	4.31 (0.09)	3.00–4.50
EC	747 (475)	97.7 (9.48)	200–1600
Al	0.16 (0.05)	1.71 (0.11)	0.20–3.00
Ca	127 (0.35)	5.35 (0.16)	<1.00–180
Mg	6.80 (0.39)	1.47 (0.06)	<0.01–30.0
Na	18.5 (14.7)	1.43 (0.14)	<1.00–40.0
K	1.51 (0.02)	0.58 (0.44)	0.30–50.0
Mn	0.11 (0.07)	2.35 (0.05)	0.02–3.00
Fe	0.13 (0.02)	0.50 (0.05)	0.00–10.0
F	0.01 (0.00)	0.00 (0.00)	0.20–30.0
Cl	0.96 (0.06)	0.89 (0.17)	3.00–170
NO_2	0.01 (0.00)	0.00 (0.00)	<1.00
SO_4	0.78 (0.21)	1.62 (0.02)	5.00–350
NO_3	3.86 (2.37)	0.58 (0.05)	5.00–450
PO_4	0.04 (0.03)	0.12 (0.02)	<0.05–12.0

and concentrations of Cl^-, SO_4^{2-} and NO_3^- in the acidic soil applying light rainfall of 0.25 mm h^{-1} the leaching rates may vary in respect to natural soil heterogeneity. Soil leachate composition was imparted by geochemical characteristic of the two soil sample sets, the acidic sandstone and the calcareous soil.

Soil leachate composition in relation to bedrock

The variation in ion concentrations of leachate are linked to soil pH, buffering capacity, and the presence of organic matter influenced by bedrock. This was revealed by the cumulative variance in PCA which indicates sample bedrock type as the main discriminating factor, while drying–re-wetting and freezing–re-wetting treatment did not significantly impact variance (Fig. 2).

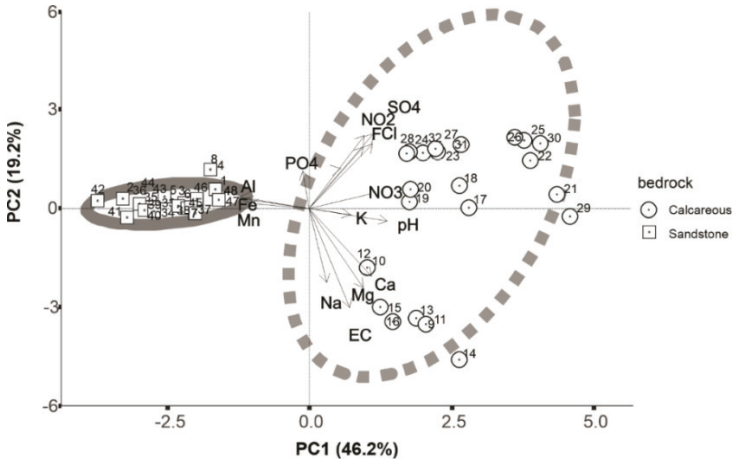

Fig. 2. Principal component analysis (PCA) plot of drying–re-wetting and freezing–re-wetting experiment showing variance in soil water composition impacted by soil geochemistry on two different soils calcareous soil (dashed line) and sandstone soil (solid line). The first two principal components (*PC*s) are plotted and colored according to bedrock. PCA was performed using all data (48 observations). Percentage of variation accounted for by each principal component is shown in brackets with the axis label.

The first two PCs account for approximately 65.4 % of the total variance. This suggests that these components effectively capture the essential patterns within the data. The eigenvalues associated with each PCs reveal the amount of variance explained by that component. Higher eigenvalues, *PC1* = 6.91, *PC2* = 2.58 explain the most variance. Variables Al^{3+}, Fe^{3+} and Mn^{2+} were negatively correlated with *PC1* and pH, *EC*, all anions concentrations, as well as Ca^{2+}, Mg^{2+}, Na^+ and K^+ were positively correlated with *PC1*. The anion concentrations were corelated with the increase of P*C2*, *EC*, Ca^{2+} and Mg^{2+} were corelated with decrease of *PC2*, and there was no correlation to pH values and nitrite concentrations.

The primary outcome illustrates the influence of soil geochemistry on the ion leachate composition and ions concentrations (Fig. 2). The leachate ion concentrations varied according to *PC1* (eigenvalue, 6.91), followed by *PC2* (eigenvalue, 2.58). The strong correlation between Al^{3+}, Fe^{3+} and Mn^{2+} was specific for soil formed on acidic sandstone. Anions except nitrites corelated to each other and basic ions, Na^{+}, K^{+}, Ca^{2+} and Mg^{2+}, specific for calcareous soil formed on limestone. Many interrelated properties different for the two types of soil, like texture.[20] Also, the soil water holding capacity explain variation in soil ion diffusion.[21]

Effect of treatments

To assess the cumulative ion release and show overall treatment effects, concentrations from all four rewetting cycles were summed for each sample (Table III).

TABLE III. Summed ion concentrations (mg L^{-1}) in leachate from control, drying–rewetting (drying) and freezing–rewetting (freezing) treatments in calcareous and acidic sandstone soils (M10 and M20 layers); values of pH and electric conductivity (*EC*, μS cm^{-1}) are mean values

Feature	Calcareous soil						Acidic sandstone soil					
	Control		Drying		Freezing		Control		Drying		Freezing	
	M10	M20	M10	M20	M10	M20	M10	M20	M10	M20	M10	M20
pH	6.90	7.25	7.10	7.77	6.93	7.47	4.24	4.37	4.33	4.57	4.16	4.16
EC	411	1083	157	232	244	235	104	91.0	151	95.4	117	106
Al	0.76	0.48	0.76	0.24	0.93	0.39	7.14	6.51	17.1	5.02	9.53	5.60
Ca	506	508	142	308	226	290	20.9	21.8	39.6	25.7	21.9	17.6
Mg	28.3	26.1	10.3	13.2	20.2	13.0	5.73	6.07	12.1	7.39	7.88	5.56
Na	32.4	60.1	5.70	7.00	50.0	6.60	5.34	6.11	9.12	7.06	7.51	9.39
K	6.00	6.10	7.60	5.00	3.53	3.12	3.57	1.07	9.00	2.48	1.54	0.04
Mn	0.64	0.25	0.37	0.11	0.63	0.12	9.56	9.26	23.4	10.6	11.9	6.43
Fe	0.58	0.46	0.47	0.32	0.63	0.34	2.13	1.87	5.31	0.95	4.61	2.92
F	0.04	0.02	0.65	0.34	0.47	0.38	0.01	0.02	0.01	0.01	0.04	0.01
Cl	3.68	4.03	10.6	12.6	7.68	11.5	4.03	3.09	3.63	2.61	2.56	0.75
NO_2	0.02	0.02	0.97	1.03	0.84	1.00	0.02	0.01	0.03	0.01	0.00	0.00
SO_4	3.73	2.52	58.6	63.8	44.1	63.7	6.55	6.44	4.69	3.53	3.67	1.66
NO_3	22.1	8.72	19.2	25.9	9.92	25.8	2.21	2.44	2.07	0.49	0.76	0.81
PO_4	0.05	0.23	0.24	0.25	0.24	0.25	0.51	0.42	0.18	0.33	0.10	0.02

The leachate pH values, *EC* and the ion concentrations differed between the two bedrock types: calcareous and acidic sandstone (Figs. 3 and 4).

In calcareous soils, K^{+} concentrations increased after the drying–rewetting treatment, while Na^{+} concentrations were higher following the freezing–rewetting treatment (Fig. 3). No increase in the cation concentrations was observed in the calcareous soil following the treatments. In the acidic sandstone soils, the drying––rewetting treatment led to elevated concentrations of Al^{3+}, K^{+}, Mg^{2+}, Mn^{2+} and Fe^{3+} in the layer M10 compared to the control leachate. No increase in the cation concentrations was observed in the acidic sandstone soil following the freezing–

–rewetting treatment. These effects were not observed in the M20 horizon of both soil types. Consequently, drying-rewetting vs freezing–rewetting treatments did not reveal a consistent and uniform effect with respect to the analysed variables of soil leachate in acidic sandstone and calcareous soil.

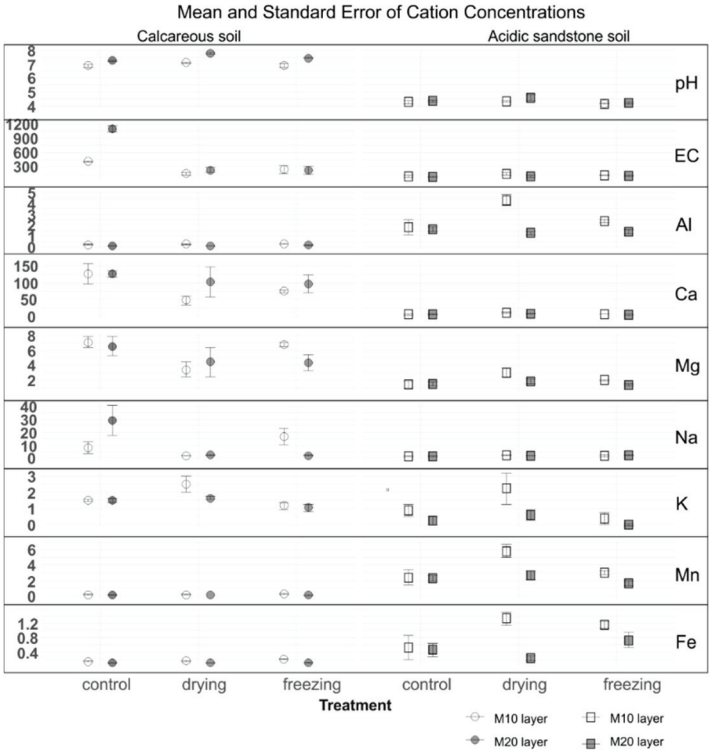

Fig. 3. Mean and standard error for leachate pH values, electric conductivity *EC* (μS cm^{-1}) and cation concentrations (mg L^{-1}) concentrations relative to bedrock, treatment and soil layer (0–10 cm M10; 10–20 cm M20).

The effect of drying–rewetting and freezing–rewetting treatments was evident in the concentrations of analysed anions in calcareous soil (Fig. 4). Anion concentrations were higher after both treatments compared to the control. In contrast, no significant differences in anion concentrations were observed in acidic sandstone soil.

No clear differences between treatments (drying–rewetting and freezing––rewetting) were observed.

The potassium concentration increased with higher temperature in calcareous soil was previously explained by higher K transmission factor with higher temperature in regard to water viscosity.[22] Also in nutrient rich soils, drying–rewetting and freezing–rewetting treatments influenced increases in leached concentration

of anions, which are most likely linked to easily soluble ions in soil organic matter. Drying induces N and P mobilization which may influence their leaching from soils.[23] Drying and rewetting probably caused dissolution of the organic N in this soil organic matter rich soil.[24] Nitrogen availability in natural soils is predominantly linked to soil organic matter dynamic and microbial processes which are sensitive to drying and rewetting. Increase in SO_4^{2-} and Cl^- are linked to the increase in temperature and higher moisture content as highly susceptible to dissolution.[25] In calcareous soils ions, Ca^{2+}, Mg^{2+}, K^+ and Na^+, stand in equilibrium with negatively charged surfaces of clay minerals and organic matter. Dissolved available ions, Al^{3+}, Fe^{3+} and Mn^{2+}, in calcareous soil did not show significant difference from control leachate, indicating their stability in such kinetic state.

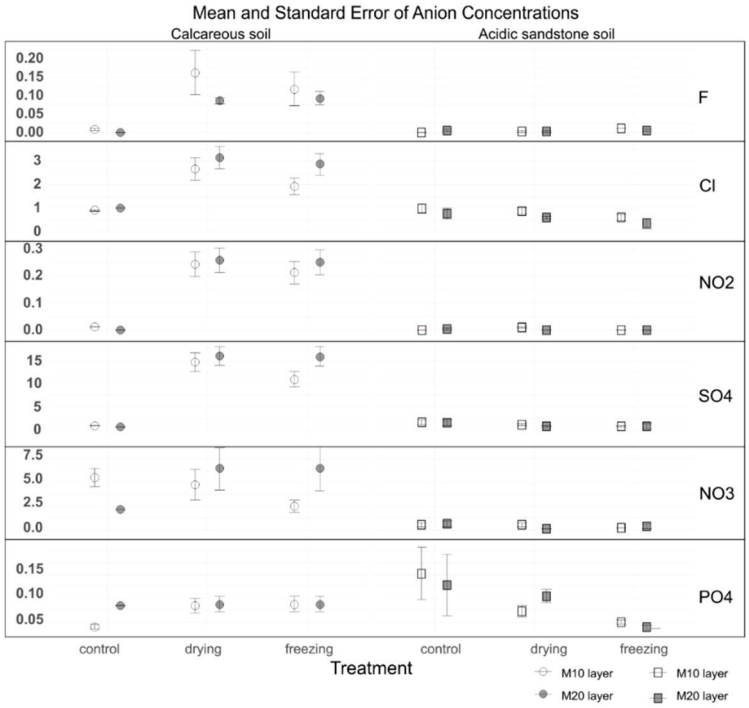

Fig. 4. Mean and standard error for leachate anion concentrations (mg L^{-1}) concentrations relative to bedrock, treatment and soil layer (0–10 cm M10; 10–20 cm M20).

In acidic soil leachate unclear treatment effect was found, observed in similar values of *EC*, pH values and ion concentrations among treatments. Only concentrations of Al^{3+}, Mn^{2+} were higher after drying–rewetting events, or Fe^{3+} also after freezing–rewetting treatment. Freezing-–rewetting stimulated substantially higher Fe^{3+} concentrations in the acidic soil. Organic acids and rewetting may be linked

to metal leaching in the form of Al^{3+} and Mn^{2+} chelates. It was found that a solubilization of Al^{3+}, Fe^{3+} and Mn^{2+} is higher in low pH soil.[26] Most probably Fe chelated with organic acids or the non-chelating acidic agents is prone to leaching processes in acidic sandstone soil. It was found in field conditions that soil rewetting after dry periods can cause low pH and high amounts of dissolved metals in the water.[27] Depending on disturbance level acidic sandstone soil may cause periodically higher metal concentrations in water. The anion concentrations differed between treatments, but generally they were in the same range as control.

The dissolution of ions should be considered both thermodynamically and kinetically, meaning that changes in energy and kinetics may be favourable or not for ion dissolution.

It is known that implications of changes of thermodynamic conditions may be more intense in acidic soil on sandstone due to higher concentrations of Al^{3+}, Fe^{3+} and Mn^{2+}.[28] The acidification also affects calcareous soil, but due to the buffering effect of $CaCO_3$ these soils are not having increase in Al^{3+}, Fe^{3+} and Mn^{2+} concentrations in leachate. The ion concentrations variability in the soil leachate can be attributed to the inherent differences in ion dissolution rates, which are influenced by soil properties.[29]

Additionally, the sample size and the number of cycles of repeating treatments in this study, in order to fully capture variability in leaching dynamics should be enlarged in further studies.

CONCLUSION

The results of this study indicate that the thermodynamic conditions significantly influence the composition of leachate in relation to the geochemical properties of the soil. The observed differences in ion concentrations between the drying–rewetting and the freezing–rewetting conditions suggest that ion solubility in agreement between both the type of ion and the prevailing thermodynamic conditions.

SUPPLEMENTARY MATERIAL

Additional data and information are available electronically at the pages of journal website: https://www.shd-pub.org.rs/index.php/JSCS/article/view/13287, or from the corresponding author on request.

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ИЗВОД

УТИЦАЈ СУШЕЊА, ЗАМРЗАВАЊА И ПОНОВНОГ ОВЛАЖИВАЊА НА ФИЛТРАТ ЗЕМЉИШТА У КИСЕЛИМ И КАЛЦИТНИМ ЗЕМЉИШТИМА

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Истраживање проучава утицај догађаја сушења-поновног овлаживања и замрзавања–поновног овлаживања на састав јона у филтрату земљишта кроз серију контролисаних лабораторијских експеримената. реакције растворености јона (Na^+, K^+, Ca^{2+}, Mg^{2+}, Al^{3+}, Fe^{3+}, Mn^{2+}, F^-, Cl^-, NO_2^-, SO_4^{2-}, NO_3^-, PO_4^{3-}) у филтрату земљишта су анализиране након циклуса поновног овлаживања под различитим условима. Резултати указују да састав филтрата зависи од специфичног процеса поновног овлаживања (сушење у односу на замрзавање) и геохемијских својстава земљишта. Циклуси сушења–поновног овлаживања и замрзавања–поновног овлаживања су имали већи утицај на састав филтрата у калцитним земљиштима. Тако су циклуси замрзавања-поновног овлаживања снажно подстакли исцурење K^+, Al^{3+} и Mn^{2+}, док су циклуси сушења-поновног овлаживања подстакли исцурење Ca^{2+} и Mg^{2+} и, за оба третмана, повећане концентрације аниона (F^-, Cl^-, NO_2^-, SO_4^{2-}, NO_3^-, PO_4^{3-}). Сушење и поновно овлаживање су утицали на веће концентрације Mn^{2+} и Fe^{3+} и мало веће концентрације аниона у киселом земљишту. Ова истраживања истичу да су ефекти сушења, замрзавања и поновног овлаживања неодвојиво повезани са геохемијским карактеристикама земљишта.

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