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Vanadium fractionation by the BCR procedure from arable soils of the Siedlce upland region

Wanad we frakcjach wydzielonych metodą BCR w glebach uprawnych wysoczyzny siedleckiej

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Abstract
The content of vanadium in exchangeable (F1), reducible (F2), oxidisable (F3) and residual fractions (F4) separated by BCR sequential extraction procedure and their distribution in 11 arable profiles of Eutric Cambisols, Haplic Luvisols and Stagnic Luvisols located on the Siedlce upland was evaluated. In these soils, the varied natural, not contaminated levels of total vanadium content were determined. Chemical analyses revealed that mean contents of vanadium in separated fractions, independently of type of investigated soils, can be arranged in the following decreasing series: F4 > F2 > F3 > F1. The highest amounts of this element in F1 (1.7%) and F4 (83.3%) fractions – in Stagnic Luvisols, and F2 (13.0%) and F3 (7.0%) fraction – in Eutric Cambisols were determined. The highest vanadium percentage share in the F1, F2 and F3 fractions was measured in the humus horizon (Ap) and in the F4 – in the enrichment horizons (B) and parent material horizons (Ck). A statistical analysis revealed the significant impact of selected soil properties (pH, Fe, Al and Mn compounds, CEC, C_{org}, clay fraction ø < 0.002 mm) on vanadium fractionation in investigated soils.

Streszczenie
Badano zawartość i profilowe rozmieszczenia wanadu we frakcji wymiennej (F1), redukowalnej (F2), utlenialnej (F3) i rezydualnej (F4), wydzielonych według sekwencyjnej procedury BCR, w 11 profilach uprawnych gleb brunatnych eutroficznych wyługowanych (Eutric Cambisols), płowych typowych (Haplic Luvisols) i płowych opadowo-glejowych (Stagnic Luvisols) Wysoczyzny Siedleckiej. W glebach tych stwierdzono zróżnicowaną naturalną zawartość ogólną wanadu. Średni procentowy udział wanadu w wydzielonych frakcjach metodą BCR, niezależnie od podtypu badanych gleb, można ułożyć w następujący szereg malejących wartości: F4 > F2 > F3 > F1. Najwięcej tego pierwiastka we frakcji F1(1,7%) i F4(83,3%) zanotowano w glebach płowych opadowo-glejowych, a we frakcji F2 (13,0%) i F3 (7,0%) – w glebach brunatnych eutroficznych wyługowanych. Największy procentowy udział wanadu we frakcji F1, F2 i F3 stwierdzono w poziomach próchnicznych (Ap), a we frakcji F4 – w poziomach wzbogacania (B) i skały macierzystej (Ck) badanych gleb. Obliczenia statystyczne wykazały istotny wpływ wybranych właściwości gleby (pH, związki Fe, Al i Mn, CEC, C_{org}, frakcja iłowa) na zawartość wanadu w wydzielonych frakcjach..

1. INTRODUCTION

Vanadium is a relatively widespread element in the Earth's crust and is mainly found in alkaline magma rocks and clayey formations. The average concentration of vanadium in soil is 10–220 $mg \cdot kg^{-1}$ and depends on the abundance of parent rock and the degree of anthropogenic pressure, whilst its profile distribution results from the soil-forming process. In the soil environment, vanadium is bound to iron oxides, clayey minerals and soil organic matter. The bioavailability of this element is determined by its form, pH values and oxidative–reductive conditions. The natural content of vanadium in soil is usually neutral, although it is sometimes necessary for the proper functioning of organisms (the synthesis of chlorophyll, nitrogen assimilation, the metabolism of lipids and carbohydrates, sodium and potassium transformations). Excessive content of this element may inhibit the growth and development in plants (chloroses and dwarfism) and in humans and animals (pathologies of the nervous, respiratory and digestive systems and of bone formation) [Kabata-Pendias, Pendias 1999, Crans et al. 2004, Aide 2005, Panichev et al. 2006, Gäbler et al. 2009].

The sequential analysis provides an important source of information on mobile and stable forms of the elements in the soil environment. This method permits a qualitative evaluation of the availability and potential toxicity of elements for the biotic components of the trophic chain and facilitates an evaluation of the degree of their translocation into the biological circulation at the stage soil–plant as influenced by the changes of the conditions in the soil environment [Rauret et al. 1999, Ovari et al. 2001, Połedniok, Buhl 2003, Chen, Owens 2008, Jeske, Gworek 2011].

The objective of this paper was to evaluate the content and profile distribution of vanadium in the fractions extracted with the BCR sequential procedure in the selected subtypes of Cambisols and Luvisols with varied properties.

2. MATERIALS AND METHODS OF STUDY

The studies were carried out on arable Eutric Cambisols (four profiles), Haplic Luvisols (three profiles) and Stagnic Luvisols (four profiles) in the Siedlce upland region (central eastern part of the province of Mazowieckie), which dominate in the structure of agricultural soils in Szaruty (52°21'N, 22°3'E), Ruchenka (52°22'N, 22°5'E), Wólka Leśna (52°12'N, 22°25'E), Klimonty

(52°10'N, 22°31'E), Mordy (52°12'N, 22°32'E), Pruszyn (52°10'N, 22°23'E), Krześlin (52°13'N, 22°21'E) and Jartypory (52°25'N, 22°7'E).

The soil samples from particular genetic horizons of 11 soil profiles were air-dried, sieved through a 2-mm mesh and then the following properties were determined: soil texture according to Polish Soil Science Society [Klasyfikacja…2009] – by the areometric method, pH in 1 mol KCl•dm^{-3} – potentiometrically, the amount of organic carbon (C_{org}) – by the oxidation–titration method [Kalembasa, Kalembasa 1992]. The soil cation exchangeable capacity (CEC) values were calculated on the basis of hydrolytic acidity (Hh) determined by Kappen's method and exchangeable cations (Ca^{2+}, Mg^{2+}, K^+ and Na^+) determined in 1 mol CH_3COONH_4•dm^{-3}. The total content of vanadium (V_t) was assessed by ICP-AES method after mineralisation in the mixture of concentrated HCl and HNO_3 (3:1) in a microwave system.

The sequential fractionation of vanadium was determined by the optimised BCR procedure proposed by The European Union's Standards, Measurements and Testing program [Rauret at al. 1999], in which four fractions were separated: F1 – exchangeable, acid extractable (extracted in 0.11 mol CH_3COOH•dm^{-3}; pH = 3); F2 – reducible, bounded to Fe and Mn hydroxides (extracted in 0.5 mol NH_2OH-HCl•dm^{-3}; pH = 2); F3 – oxidisable, bounded to organic matter and sulphides (digest with 8.8 mol H_2O_2 dm^{-3} and extracted in 1 mol CH_3COONH_4·dm^{-3}; pH = 2). Residual fraction F4 was calculated as the difference between the total content of vanadium and the sum of this metal fraction: F1, F2 and F3.

The content of vanadium in particular fractions was determined by ICP-AES method.

The percentage contribution of the separated four fractions of V in relation to its content in aqua regia solution was calculated. The accuracy of the measurements was determined with the addition of a standard into each analysed sample. The analyses included control samples consisting of chemical reagents (corresponding to fractions). For the obtained results, arithmetic means (AM), standard deviation (SD) and relative standard deviation (RSD) were calculated. The statistical relationships between vanadium content in sequenced separated fractions and selected properties of analysed soils were evaluated with linear correlation at two levels of significance $p < 0.05$ and $p < 0.01$, using Statistica 10.1 software (StatSoft, Inc., Tulsa, OK, USA).

3. RESULTS AND DISCUSSION

The examined Cambisols and Luvisols were formed from glacial tills (sandy loam, sandy-clay loam, loam and clay loam that are usually sandy in the surface of the profile) of the Warta glaciation and their selected physical, physical–chemical and chemical properties (Table 1) differentiated by the paedogenetic processes and agricultural activities have been described in the publication by Pakuła and Kalembasa [2013].

It was found that in these soil categories, the content of vanadium was varied between the individual profiles and within a given profile. The average content of this metal was 20.4 mg•kg^{-1} (Eutric Cambisols); 16.9 mg•kg^{-1} (Stagnic Luvisols) and 15.6 mg•kg^{-1} (Haplic Luvisols) (Table 2). The highest concentration of vanadium, regardless of the soil subtype, was detected in the enrichment horizons (Bw and Bt: 23.1–26.8 mg•kg^{-1}), whereas the lowest was in the humus horizon (Ap 12.0–14.1 mg•kg^{-1}), which confirms the impact of soil-forming processes on its profile distribution [Jeske, Gworek 2012] that depends on the proportion of the clay fraction (r = 0.716–0.794; $p < 0.01$) and iron (r = 0.662–0.861; $p < 0.01$), aluminium (r = 0.587–0.823; $p < 0.01$) and manganese compounds (r = 0.357–0.642; $p < 0.01$; Table 3) [Kabata-Pendias, Pendias 1999, Aide 2005]. In the Ap horizon, the content of V_t approximated the value reported for the soil located on the areas with a low degree of anthropogenic pressure [Połedniok, Buhl 2003, Małuszyński, Małuszyńska 2011, Jeske, Gworek 2012]. It was within the range of comparative values proposed for a quality evaluation of the chemical composition in the surface soil horizons in Poland (4–85 mg•kg^{-1}) [Dudka 1992]. Kabata-Pendias and Pendias [1999] reported that the presence of vanadium in the soil profile is a derivative of its content in the parent rocks.

A chemical analysis demonstrated a varied content of vanadium in the sequentially extracted fractions of the investigated soil types (Table 2). The average percentage contribution in the total content of this element can be presented in the following order of decreasing values for the following soil sub-types: Eutric Cambisols F4

Table 1. Some properties (ranges) of investigated soils in the Siedlce upland

Genetic horizon	Mean depth (cm)	pH_{KCl}	CEC* (mmol(+)•kg^{-1})	C_{org} (g•kg^{-1})	Clay fraction, ø < 0.002 mm (%)
		Eutric Cambisols (4 profiles)			
Ap	0–30	5.11–6.34	43.9–80.0	7.79–10.3	11–12
Bw1	30–55	4.78–5.97	114–199	2.45–4.25	33–41
Bw2	55–100	4.90–6.39	127–211	1.38–3.17	24–35
Ck	100–150	7.02–7.75	176–247	0.70–1.33	21–27
		Haplic Luvisols (3 profiles)			
Ap	0–30	4.58–5.68	57.6–79.4	7.40–10.4	7–9
EB	30–45	4.60–6.20	65.4–119	1.70–3.19	18–23
Bt	45–95	4.88–6.52	156–173	0.90–2.20	26–28
Ck	95–150	7.08–7.96	175–187	0.70–1.30	19–23
		Stagnic Luvisols (4 profiles)			
Ap	0–25	4.11–5.19	32.1–77.0	6.05–16.6	7–10
EB	25–40	4.55–5.43	50.7–137	1.30–2.75	11–16
Btg	40–100	4.59–6.31	136–151	0.70–1.60	20–28
Ck	100–150	6.51–7.60	125–192	0.50–1.12	15–24

*Cation exchangeable capacity.

(79.2) > F2 (13.0) > F3 (7.0) > F1 (0.8); Haplic Luvisols F4 (83.0) > F2 (10.0) > F3 (5.7) > F1 (1.3) and Stagnic Luvisols F4 (83.3) > F2 (9.7) > F3 (5.3) > F1 (1.7).

The smallest quantity of vanadium was detected in the bioavailability, exchangeable fraction F1 0.4–1.3% (Eutric Cambisols); 0.5–2.1% (Haplic Luvisols) and 0.5–3.3% (Stagnic Luvisols) (Table 2). The high concentration of vanadium in this fraction in the soil environment may be toxic to plants and animals, especially at a low reaction pH [Kabata-Pendias, Pendias 1999, Crans et al. 2004, Teng et al. 2011]. The significant value ($p < 0.01$) of the correlation coefficient r (from 0.835 to 0.858) indicates a close correlation between the content of vanadium in the fraction F1 and the value of pH in the examined soil types (Table 3). Małuszyński and Małuszyńska [2011] have reported that under reductive conditions the bioavailability of this element increases. The highest amount of vanadium was detected in the residual fraction (F4): 72.8–88.7% (Eutric Cambisols); 74.4–90.2% (Stagnic Luvisols) and 71.4–91.6% (Haplic Luvisols). Ovari et al. [2001], Połedniok and Buhl [2003], Małuszyński and Małuszyńska [2011] and Jeske and Gworek [2012] have highlighted a significant role of very durable organic-mineral and mineral complexes in the speciation of vanadium; these authors detected 57.4–85.5% of this element in the durable complexes of the residual fraction in uncontaminated soil. Aide [2005] and Gäbler et al. [2009] have reported that vanadium is strongly bound by clay minerals and iron oxides/hydroxides and aluminium oxides/hydroxides. In the examined soil types, it was confirmed by significant ($p < 0.01$) correlations between the content of Fe, Al, clay fraction and F4 fraction (Table 3).

The highest average contribution of vanadium in the exchangeable fraction F1, reducible fraction F2 and oxidisable fraction F3 was detected in the surface horizon Ap: 1.1–3.0%; 12.1–14.4% and 10.4–11.1%, whereas the lowest was in the parent rock horizon (Ck): 0.5–0.6% – F1; 11.7% (Cambisols) – F2; 0.7% – F3 and in the fraction F2 in the Bt horizon: 6.4–7.3% (Luvisols). The contribution of this metal in the residual fraction F4 increased with depth amounting to the maximal values in the parent rock of Cambisols (85.7–88.7%) and in the enrichment horizon Bt of Luvisols (88.9–91.6%) (Table 2). Jeske and Gworek [2012] have reported that, in mineral soil, the highest quantity of vanadium is detected in the reducible and residual fractions, which may result from the lithogenic origin of this element and the affinity to iron compounds and clay fraction, and that its profile distribution in Luvisols results from the process of lessivage. Połedniok and Buhl [2003] found that the content of vanadium in the organic complexes was approximately three times higher in the contaminated soil than in the soil with natural content of this element (<15%). This fraction may constitute a potential source of absorbable forms of vanadium and may be used to evaluate the degree of soil contamination.

In the examined soil types, the correlations were detected between the majority of the analysed parameters and they were stronger for Cambisols than for Luvisols (Table 3). Regardless of the subtype of the tested soil, the statistical calculations demonstrated (Table 3) a highly significant and significant positive impact of the total content of vanadium (Vt) on the concentration of this element in the exchangeable fraction F1, reducible fraction F2,

Table 2. The total content of vanadium and percentage contribution (mean* and ranges**) of its fractions in the investigated soils of the Siedlce upland.

Genetic horizon	Mean depth (cm)	V_t (mg•kg^{-1})	Fraction (%)			
			F1	F2	F3	F4
Eutric Cambisols (4 profiles)						
Ap	0–30	14.1* (13.6–14.5**)	1.1 (0.9–1.3)	14.4 (13.8–15.0)	10.5 (9.5–11.2)	74.1 (72.8–75.8)
Bw1	30–55	26.8 (24.9–28.4)	0.9 (0.7–1.1)	13.2 (12.8–13.8)	9.0 (8.5–9.9)	77.0 (75.5–77.8)
Bw2	55–100	25.1 (23.0–26.6)	0.8 (0.6–0.9)	12.6 (12.1–13.2)	8.2 (7.2–9.1)	78.6 (77.0–79.9)
Ck	100–150	15.4 (12.8–18.9)	0.5 (0.4–0.6)	11.7 (10.3–13.1)	0.7 (0.5–0.7)	87.1 (85.7–88.7)
Arithmetic mean		19.2	0.7	12.6	5.6	81.1
SD		5.69	0.24	1.19	4.44	5.65
RSD %		29.7	32.9	9.4	79.9	7.0
Haplic Luvisols (3 profiles)						
Ap	0–30	12.6 (11.8–13.1)	2.0 (1.8–2.1)	12.1 (11.3–12.9)	10.6 (10.3–10.9)	75.3 (74.4–75.9)
EB	30–45	13.2 (12.4–13.8)	1.5 (1.4–1.6)	11.1 (10.1–11.8)	9.6 (9.4–9.8)	77.7 (77.2–78.6)
Bt	45–95	23.1 (22.1–24.2)	0.9 (0.8–1.1)	7.3 (6.3–8.2)	2.3 (2.1–2.6)	89.5 (88.9–90.2)
Ck	95–150	13.5 (12.3–14.1)	0.6 (0.4–0.5)	9.5 (8.8–10.5)	0.7 (0.5–0.7)	89.2 (88.5–90.0)
Arithmetic mean		16.7	1.1	9.5	4.6	84.8
SD		4.87	0.54	1.96	4.26	6.29
RSD %		29.2	48.7	20.7	93.0	7.4
Stagnic Luvisols (4 profiles)						
Ap	0–25	12.0 (11.2–13.0)	3.0 (2.6–3.3)	12.8 (12.1–13.5)	11.1 (10.6–11.9)	73.2 (71.4–74.2)
EB	25–40	16.2 (14.6–18.1)	2.1 (1.9–2.2)	9.8 (9.3–10.3)	7.9 (7.1–8.4)	80.3 (79.9–80.6)
Btg	40–100	23.8 (22.5–25.0)	1.2 (1.1–1.3)	6.4 (5.6–7.5)	1.4 (1.1–1.8)	91.0 (90.3–91.6)
Ck	100–150	15.5 (13.3–17.3)	0.6 (0.5–0.7)	9.6 (9.2–10.1)	0.7 (0.6–0.9)	89.0 (88.8–89.2)
Arithmetic mean		17.4	1.5	9.3	4.3	84.9
SD		4.59	0.90	2.31	4.35	7.03
RSD %		26.5	60.1	24.8	101.7	8.3

SD – standard deviation; RSD – relative standard deviation; Fraction: F1 – exchangeable; F2 – reducible (bound to Fe–Mn oxide; F3 – oxidisable (bound to organic matter); F4 – residual; Vt – total content of vanadium.

Table 3. The coefficient values of correlation between the vanadium fractions and some properties of investigated soils on the Siedlce upland

Parameter	Fraction				V_t
	F1	F2	F3	F4	
Cambisols					
V_t	0.773**	0.966**	0.785**	0.980**	–
Fe	0.456*	0.678**	0.376	0.893**	0.861**
Al	0.411	0.689**	0.354	0.900**	0.823**
Mn	0.234	0.551**	0.271	0.703**	0.642**
pH_{KCl}	0.835**	0.404	0.807**	0.330	0.377
CEC	0.241	0.020	0.360	0.594**	0.152
C_{org}	0.567**	0.031	0.550**	0.335	0.164
ø < 0.002 mm	0.378	0.723**	0.419	0.837**	0.794**
Luvisols					
V_t	0.325*	0.406*	0.377*	0.960**	–
Fe	0.040	0.024	0.313	0.700**	0.662**
Al	0.095	0.057	0.263	0.609**	0.587**
Mn	0.185	0.028	0.190	0.462**	0.357**
pH_{KCl}	0.858**	0.068	0.719**	0.023	0.130
CEC	0.628**	0.182	0.310	0.535**	0.425**
C_{org}	0.466**	0.091	0.440**	0.288	0.316
ø < 0.002 mm	0.284	0.107	0.277	0.772**	0.716**

Significant at $*p < 0.05$ and $**p < 0.01$.
Fraction: F1 – exchangeable; F2 – reducible (bound to Fe–Mn oxide); F3 – oxidisable (bound to organic matter); F4 – residual; V_t – total content of vanadium; CEC – cation exchangeable capacity; ø < 0.002 mm – clay fraction.

oxidisable fraction F3 and residual fraction F4. The fraction F4 significantly ($p < 0.01$) and positively depended on the concentration of iron, aluminium and manganese compounds, clay fraction and cation exchange capacity (CEC). In the brown soil, the complexes of iron, aluminium and manganese and clay fraction had a significant ($p < 0.01$) impact on the content of vanadium in the fraction F2. The content of this element in the fractions F1 and F3 was significantly correlated with carbon in the organic complexes C_{org} (0.466–0.567 and 0.440–0.550) and with the reaction (pH) of soil (0.835 and 0.858; 0.719 and 0.807). Similar relations have been also reported by Ovari et al. [2001], Połedniok and Buhl [2003], Teng et al. [2011] and Jeske and Gworek [2012].

4. CONCLUSIONS

1. In 11 examined cultivated soils located on Wysoczyzna Siedlecka, the content of vanadium was classified as natural and its profile distribution resulted from the type and degree of the advancement of the soil-forming process.
2. Regardless of the subtype of tested soil, the average percentage contribution of vanadium in the fractions sequenced with BCR method is arranged in the following order of decreasing values: F4 > F2 > F3 > F1. The highest quantity of vanadium in the exchangeable fraction F1 and the residual fraction F4 was detected in the Stagnic Luvisols, whereas in the reducible fraction F2 and oxidisable fraction F3 the highest level was in Eutric Cambisols.
3. In the surface humus horizons of the examined soil types where the pH value was lowest and the content of carbon in the organic complexes was the highest, the highest quantity of vanadium was detected in the exchangeable fraction F1 and the oxidisable fraction F3. The contribution of these fractions decreased with soil depth.
4. In the reducible fraction F2, the highest contribution of vanadium was detected in the humus horizon, whereas the lowest was in the parent rock horizon in Cambisols and in the enrichment horizon in Luvisols. The contribution of this element in the residual fraction F4 increased, in general, with soil depth, which was associated with the profile distribution and accumulation of mineral colloids under the influence of soil-forming processes.
5. The statistical calculations demonstrated the significant impact of the selected properties of the investigated soil types on the content of vanadium in the extracted fractions.

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