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Potassium availability and soil extraction tests in agricultural soils with low exchangeable potassium content

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ABSTRACT

Low application of fertilizer potassium (K) in intensive agriculture leads to a gradual decrease and, afterwards, to a steady state of soil K determined by tests which are based on soil exchangeable K (K_{exch}) extraction. In this situation, non-exchangeable K ($K_{non-exch}$) is being released and therefore real plant availability does not necessarily correspond to soil test K. This incoherency was investigated in 14 agricultural soils with low K as determined by the Mehlich 3 method. Plant-available K released by exhaustive cropping of perennial ryegrass and K determined by the Neubauer seedling test were compared with 8 soil K extraction methods, with the total K content and with the relative content of soil K-bearing minerals. K determined by the ryegrass test (314 mg/kg on average) was more than 3-times higher than K based on K_{exch} extraction methods, but was from 2 to 3 times lower than K determined by $K_{non-exch}$ extracting methods. The relative content of mixed-layer phyllosilicates was significantly related to K extracted by soil tests. The relative content of orthoclase correlated only with total K and mica-group minerals with none of the extractions. The best prediction of plant-available K in investigated soils was obtained with sodium tetraphenylboron and StepK methods.

Keywords: essential nutrient; soil testing; plant K availability; X-ray diffraction; clay minerals

Potassium (K) fertilizer recommendations are usually based on soil exchangeable K (K_{exch}) measurement which gives reasonable results in many soils and situations (Slaton et al. 2010, Zörb et al. 2014, Bar-Yosef et al. 2015). However, the K_{exch} testing might be inadequate in case of soils with high proportion of non-exchangeable K (Khan et al. 2014). In such soils, tests based on non-exchangeable K ($K_{non-exch}$) can substantially improve prediction of plant K availability (Richards and Bates 1988, Cox et al. 1999, Moody and Bell 2006). The need for $K_{non-exch}$ testing increases also in regions where application rates of K fertilizers dropped substantially in last decades, as it is the case of many European countries (Öborn et al. 2005, Torma 2006, Čermák et al. 2007, Grzebisz et al. 2010, FAO 2011). In intensive cropping systems with imbalanced K fertilization, plant nutrition relies largely on $K_{non-exch}$. While K_{exch} can reach steady state at a low level when K balance is negative for a long time (Blake et al. 1999, Madaras and Lipavský 2009), a decrease of K reserves is clearly measurable in $K_{non-exch}$ pool (Carey and Metherell 2003, Andrist-Rangel et al. 2007, Madaras et al. 2014). K_{exch} monitoring therefore might not give a true impression about the development of soil K reserves (Madaras 2014).

Methods determining $K_{non-exch}$ were reviewed by Martin and Sparks (1985) and include exhaustive cropping, extraction with acids, electro-ultrafiltration, exchange resins and extraction using sodium tetraphenylboron (TPB) with ethylenediaminetetraacetic acid. The latter method is based on K precipitation, which decreases K concentration in solution so as to facilitate a release of $K_{non-exch}$ (Carey et al. 2011). Exhaustive cropping is often

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used for determination of plant-available K and for evaluation of suitability of faster chemical methods of $K_{non\text{-}exch}$ determination (MacLean 1961, Richards and Bates 1988, Cox et al. 1999).

In our research, we were concerned with agricultural arable soils that showed steady-state of K_{exch} at low level (measured by the Mehlich 3 soil test), which could indicate that plant K demand was fulfilled from $K_{non\text{-}exch}$ pool. The aim of this paper is to investigate the relation between true plant K availability (determined by exhaustive cropping) and soil test K determined by various methods of K_{exch} and $K_{non\text{-}exch}$ extraction for such soils.

MATERIAL AND METHODS

Sites for soil sampling (Table 1) were selected from the database of soil testing which is regularly (in a 6-year period) performed in the Czech Republic by the Central Institute for Testing and Supervising in Agriculture. Selection criteria of arable land soils were: (1) low level of K in Mehlich 3 soil test (K_{M3} < 100 mg/kg) in the last two testing periods; (2) considering the most common soil types of agricultural land, that are Chernozems, Luvisols, Cambisols and Fluvisols; (3) considering variability of climatic conditions and geological substrates. 14 soil profiles were sampled in spring 2010. At each site, soil samples (0–25 cm) were mixed from 3 sampling points. Soil samples were homogenized, dried at room temperature and sieved through 2 mm mesh prior to further analyses.

Mineralogical composition of soil samples was determined on the basis of the X-ray diffraction (XRD) patterns which were obtained using the CuK radiation with the powder difractometer X'Pert System Philips with a graphite monochromator. The relative presence of main soil minerals was evaluated on a 5-point scale in a range from the limit of detectability (1) to dominant mineral (5).

Soil reaction, texture and organic carbon content were determined by common methods (Pansu and Gautheyrou 2006). Determination of soil K was performed by several chemical extraction meth-

Table 1. Characterization of sites and topsoil properties

Site	Altitude (m a.s.l.)	Soil type	Geology	pH	C_{org} (%)	CEC ($mmol^+$/100 g)	Texture	MLP type
Vlačice	220	Chernozem	Pleistocene fluvial sands and grits	7.9	2.0	26.8	silt loam	I/S
Moravská Nová Ves	156	Mollic Fluvisol	Neogene fluvial sands and grits	5.6	1.5	24.6	clay loam	I/S
Čistá (district Mladá Boleslav)	287	Luvisol	Quaternary loess/loessial clays	6.2	1.1	13.0	silt loam	I/S
Bynovec	392	Cambisol	Cretaceous quartz sandstones	5.2	1.8	10.0	sandy loam	I/V
Vojkovice	164	Fluvisol	Holocene fluvial sands	7.9	1.2	15.2	loam	C/S
Tuhaň	304	Luvisol	Cretaceous quartz sandstones	5.5	1.2	13.3	silt loam	I/S
Podkost	303	Luvisol	Quaternary loess/loessial clays	5.0	1.2	13.9	loam	I/S
Všelibice	415	Cambisol	Cretaceous quartz sandstones	5.3	0.8	5.54	loamy sand	I/S
Radovesnice II	228	Chernozem	Cretaceous calc. clay/marl-stones	5.1	1.1	8.94	sandy loam	I/S
Oblanov	451	Cambisol	Permian sandstones	6.1	1.0	8.81	sandy loam	I/V
Škvořetice	466	Cambisol	Paleozoic mica-schist	5.6	1.5	12.7	loam	I/S
Čejč	185	Chernozem	Neogene calc. clays	7.7	1.1	9.49	sandy loam	I/S
Velký Ratmírov	511	Cambisol	Paleozoic paragneiss	4.7	1.6	8.04	sandy loam	I/S + C/S
Vápno	399	Luvisol	Cretaceous quartz sandstones	4.2	1.0	9.89	silt loam	I/S

CEC – cation exchange capacity; MLP – mixed-layer phyllosilicates; C – chlorite; S – smectite; I – illite; V – vermiculite

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Table 2. Overview of potassium (K) extraction methods and plant tests

Abbreviation	Extractant/Procedure description	K pool	Reference
K_{Neub}	rye (*Secale cereale*) seedlings grown for 17 days in quartz sand-soil mixture (1:2)	plant-available K	Neubauer (1924)
$K_{exhaust}$	exhaustive cropping of perennial ryegrass (*Lolium perenne*) grown for 10 months in quartz sand-soil mixture (9:1)	plant-available K	
K_{M3}	Mehlich 3 combined solution	exchangeable K ('plant-available' K)	Mehlich (1984)
K_{NH_4Ac}	1 mol/L ammonium acetate	exchangeable K	Pansu and Gautheyrou (2006)
K_{BaCl_2}	$BaCl_2$ – triethanolamine, pH 8.1	exchangeable K	ISO 13536
$K_{0.1\ mol/L\ HNO_3}$	0.1 mol/L nitric acid	exchangeable K	Richards and Bates (1988)
K_{HCl}	1 mol/L hydrochloric acid (20 h at 50°C)	exchangeable plus non-exchangeable K	Scheffer and Schachtschabel (1976)
K_{TPB}	0.1 mol/L sodium tetraphenylboron	exchangeable plus non-exchangeable K	Carey et al. (2011)
K_{StepK}	1 mol/L nitric acid; 7 consecutive extractions (StepK – acronym of the method)	non-exchangeable K	Richards and Bates (1988)
K_{tot}	HF-$HClO_4$ digestion	total K	Potts (1995)

ods and two plant tests (Table 2). As a reference for plant-available K content in soil, exhaustive cropping with perennial ryegrass was chosen. The ryegrass was grown in a glasshouse in 2 L plastic pots filled with the homogeneous mixture of 150 g of soil and 1350 g of fine quartz sand. The purity of the sand was checked by XRD. The sand contained 7 mg K_{HCl}/kg. Pots were irrigated by K-free Hoagland solution including all other essential nutrients to keep the water content at approximately field capacity. After six weeks, the above-ground biomass was cut for the first time, dried and analysed for K content. Plants were grown until the substantial decrease of the vitality in all treatments. Altogether eight cuts were performed. At the end of the experiment, roots were carefully taken out of pots, washed by distilled water, dried and analyzed for K content. K content in plants of the control treatment (only quartz sand) was subtracted. After the ryegrass cropping, K_{M3} and K_{HCl} were determined in the pot substrate (a mixture of soil and quartz sand).

Each test was performed in two replicates. In plant tests, the biomass was mineralized by microwave digestion. All extracts were analyzed by ICP EOS, Thermo Jarrell Ash (Trace Scan, Franklin, USA). Statistical evaluation of the results was performed using the Statistica 12 software (Statsoft, Tulsa, USA).

Table 3. Descriptive statistics of different potassium extractions and XRD semiquantitative analysis of K-bearing minerals

	Unit	Average	Min	Max	SD
$K_{exhaust}$	(mg/kg soil)	314	46	875	241
K_{Neub}		118	31	291	83
K_{NH_4Ac}		65	30	148	36
K_{BaCl_2}		79	31	156	37
K_{M3}		91	32	176	41
$K_{0.1\ mol/L\ HNO_3}$		103	31	214	56
K_{StepK}		1034	319	2024	512
K_{HCl}		590	110	1630	406
K_{TPB}		768	163	1844	496
K_{tot}		15 700	3980	27 200	7000
Orthoclase rel.	relative scale 1–5	2.1	1.0	3.0	0.6
Mica minerals rel.		1.3	1.0	2.0	0.4
MLP rel.		1.8	1.0	3.0	0.5

Relative scale: 1 – mineral present at the limit of detectability; 5 – dominant mineral; SD – standard deviation; MLP – mixed-layer phyllosilicates

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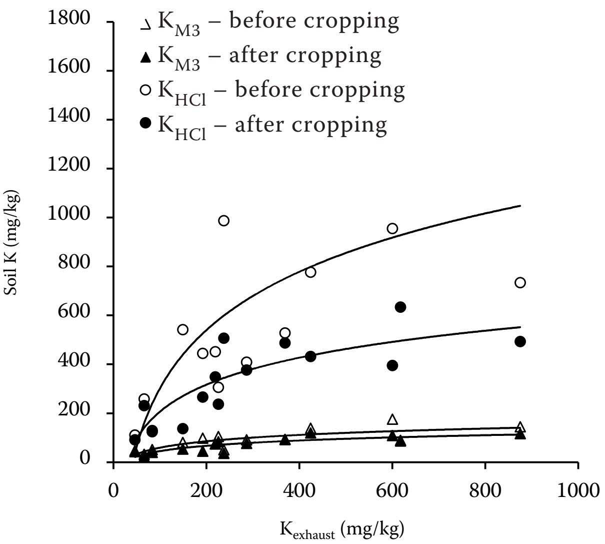

Figure 1. Soil potassium (K) extracted by the Mehlich 3 method and by 1 mol/L hydrochloric acid before and after exhaustive cropping, related to the total K plant uptake

RESULTS AND DISCUSSION

Plant-available K determined by exhaustive cropping of ryegrass released from 3 to 5-times more K than chemical methods oriented to K_{exch} extraction (Table 3). The range of $K_{exhaust}$ was also much wider than the range of K_{NH_4Ac}, K_{BaCl_2}, K_{M3} and $K_{0.1\ mol/L\ HNO_3}$; the latter four were related the closest to K_{Neub} by both absolute value and by the range. It was evident that $K_{non\text{-}exch}$ significantly contributed to plant nutrition, as was found also for other sites (MacLean 1961, Cox et al. 1999). After the ryegrass test, K_{HCl} decreased by 250 mg/kg (32% of the starting value) on average, whereas K_{M3} decreased only by 19 mg/kg (20% of the starting value) on average (Figure 1). It can be seen that especially in high $K_{exhaust}$ soils there was still a significant amount of plant-available K after the exhaustive cropping; even the biomass production after the 7th cut was very low in all treatments. The incomplete K exhaustion can explain why all three methods extracting $K_{non\text{-}exch}$ released more than twice as much K compared to exhaustive cropping. The highest K contents were obtained by consecutive extractions with 1 mol/L nitric acid (StepK method) which overestimated K contents especially in samples with low $K_{exhaust}$.

With exception of K_{tot}, K contents determined by all other chemical extractions correlated significantly with $K_{exhaust}$ (Table 4). The best prediction of $K_{exhaust}$ was given by TPB extraction ($P = 8.3 \times 10^{-8}$), followed by StepK method ($P = 2.1 \times 10^{-5}$). K contents based on K_{exch} extraction were strongly positively correlated among each other and also with K_{Neub},

Table 4. Correlation (*R*) between plant-available K, extraction tests and selected soil properties

	$K_{exhaust}$	K_{Neub}	K_{NH_4Ac}	K_{BaCl_2}	K_{M3}	$K_{0.1\ mol/L\ HNO_3}$	K_{StepK}	K_{HCl}	K_{TPB}	K_{tot}	Orthoclase rel.	Micas rel.	MLP rel.
K_{Neub}	0.92***												
K_{NH_4Ac}	0.82***	0.90***											
K_{BaCl_2}	0.77**	0.86***	0.93***										
K_{M3}	0.77**	0.85***	0.88***	0.89***									
$K_{0.1\ mol/L\ HNO_3}$	0.83***	0.88***	0.96***	0.88***	0.91***								
K_{StepK}	0.89***	0.76**	0.57*	0.53	0.63*	0.67**							
K_{HCl}	0.69**	0.52	0.24	0.21	0.39	0.32	0.89***						
K_{TPB}	0.96***	0.86***	0.78**	0.76**	0.76**	0.84***	0.90***	0.65*					
K_{tot}	0.29	0.21	0.13	0.02	0.05	0.26	0.53	0.41	0.43				
Orthoclase rel.	0.24	0.10	0.04	–0.05	0.00	0.22	0.51	0.41	0.38	0.94***			
Mica min. rel.	0.52	0.43	0.28	0.28	0.15	0.23	0.53*	0.50	0.41	0.23	0.15		
MLP rel.	0.81***	0.67**	0.67**	0.57*	0.56*	0.70**	0.72**	0.48	0.82***	0.23	0.17	0.39	
% clay	0.85***	0.75**	0.83***	0.83***	0.74**	0.78**	0.61*	0.37	0.84***	0.07	0.00	0.28	0.81***

MLP – mixed-layer phyllosilicates; **P* < 0.05; ***P* < 0.01; ****P* < 0.001

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Table 5. Relationships between plant-available potassium (K) determined by exhaustive cropping and selected K extraction tests ($K_{exhaust}$ = A × X + B)

Variable X	Full sample set (*n* = 14)			Sample 'Vojkovice' excluded (*n* = 13)			
	A	B	*P*	A	B	*P*	*R*
K_{NH_4Ac}	5.529	–43.4	3.73×10^{-4}	6.000	–105	1.71×10^{-6}	0.94
K_{BaCl_2}	4.998	–79.7	1.23×10^{-3}	5.629	–163	1.10×10^{-5}	0.92
K_{M3}	4.541	–98.2	1.36×10^{-3}	4.613	–130	3.72×10^{-4}	0.84
$K_{0.1\ mol/L\ HNO_3}$	3.592	–56.5	2.10×10^{-4}	3.675	–91.0	1.21×10^{-5}	0.91
K_{StepK}	0.4185	–119	2.15×10^{-5}	0.4684	–158	5.27×10^{-5}	0.89
K_{HCl}	0.4081	73.2	6.67×10^{-3}	0.5465	11.8	1.29×10^{-2}	0.67
K_{TPB}	0.4655	–43.7	8.30×10^{-8}	0.4550	–40.1	4.90×10^{-7}	0.95

P – probability

as was reported previously by Matula (2009) who used a 21-day barley test. The relation of extractions based on K_{exch} to K extracted by strong acids (K_{StepK} and K_{HCl}) was none or weak, whereas K_{exch} tests significantly correlated with K_{TPB}. K_{tot} was not related to any other K extraction, contrary to the findings of Carey et al. (2011); the closest relation of K_{tot} was to K_{StepK} (P = 0.051).

The orthoclase content was significantly related only to K_{tot}, but had no other relation neither to soil K extraction nor to plant-available K. Results confirmed that orthoclase can be a dominant K phase of soil K in some soils (Andrist-Rangel et al. 2010), but it is rather inert in context of plant K uptake as K is bound mainly in structural form (Huang 2005). Mica group minerals contribute to K plant uptake (Madaras et al. 2013); however the relation to $K_{exhaust}$ was found weak in this study (P = 0.059).

For plant K nutrition, mixed-layer phyllosilicates appeared to be the most important K-bearing mineral phase. Relative contents of mixed-layer phyllosilicates (MLP) significantly correlated with both plant tests and also with chemical extractions. The role of interlayer K in 2:1 soil clay minerals for plant K uptake was emphasized by Barré et al. (2007). Clay content was significantly related to the same extracted K pools as MLP relative content, confirming that MLP minerals constitute the major proportion of soil clay fraction in the studied soils. Soil $K_{non-exch}$ pool is largely determined by soil texture (Škarpa and Hlušek 2005). For other soil parameters (pH, C_{org}, cation exchange capacity), we did not find relation to K pools.

Results suggest that consistency between plant available K and K_{exch} depends also on the type of MLP minerals. Excluding sample 'Vojkovice' from the dataset significantly increased the correlation between $K_{exhaust}$ and K_{exch} contents (Table 5). The Vojkovice soil had rather high K availability ($K_{exhaust}$ = 618 mg/kg), but K_{exch} tests determined too low K contents (K_{M3} = 86 mg/kg) compared to other soils of similar $K_{exhaust}$. This sample was the only one with chlorite-smectite type of MLP, other soils have illite-smectite or illite-vermiculite type (Table 1). Omitting Vojkovice soil, however, did not improve correlation of $K_{exhaust}$ with tests extracting $K_{non-exch}$.

We conclude that plant-available K, as determined by exhaustive perennial ryegrass cropping, can be satisfactorily predicted by all tested methods. Methods based on exchangeable K extraction however highly underestimated available K contents and were also influenced by the character of clay minerals. Based on our results, TPB extraction in the form proposed by Carey et al. (2011) appears to be the most suitable for routine determinations of available K contents in soils low in exchangeable K.

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