POTASSIUM EXTRACTABILITY FROM SOILS OF BRAZILIAN COFFEE REGIONS

EXTRACTABILIDADE DE POTÁSSIO DE SOLOS BRASILEIROS CULTIVADOS COM CAFEEIRO

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ABSTRACT: Coffee, which is an important commodity for Brazil, is a highly K-demanding crop. Methods for recommending K fertilization to coffee crops in Brazil are based on the amount of exchangeable K. Mineralogical studies estimating K supply capacity from different soil fractions, from medium to long term, were performed in Brazilian soils, but very few studies have been carried out focusing on the use of successive chemical extractions. This study evaluated K release from whole soil, as well as clay, silt, and sand fractions of B-horizon samples of a basalt-derived Oxisol and a sienite-derived Ultisol, both representative soils from coffee regions of Minas Gerais State. Successive extractions (0 to 665 h) of K were performed with 10^{-3} mol L⁻¹ of either citrate or oxalate at 1:10 solid:solution ratio. The cumulative results were compared with forms of K (exchangeable, non-exchangeable, and total) as measured by different extraction procedures. The results showed that exchangeable K was higher, and non-exchangeable as well as total K were lower in the whole soil fraction of the Oxisol, than the K forms extracted from the Ultisol. Citrate and oxalate extracted similar amounts of K from the Oxisol, which is probably related to the fact that most of its K supply came from the exchangeable form. Citrate caused a greater release of K (part of which being from the non-exchangeable form) from the Ultisol than did oxalate. This work showed that soil texture alone is not a good indicator of K supply capacity, since for the same particle size, the studied soils revealed different K extractability.

KEYWORDS: Citric acid. Oxalic acid. Soil texture.

INTRODUCTION

Most of the tropical soils – including the Brazilian ones – have very low natural reserves of K (WOOD et al., 2001). In contrast, coffee, which is grown in 2.34 million ha in Brazil (CONAB, 2012), demands high amounts (~200 kg ha⁻¹) of such a nutrient (SILVA et al., 2001). As a result, studies on K release from these soils are important as most of the K used in Brazilian agriculture is imported (FAO, 2004).

The prediction of coffee yield in response to land-applied K fertilizers based on soil tests for exchangeable K is sometimes of limited accuracy (PAL et al., 2001). Potassium uptake by plants from non-exchangeable forms has been demonstrated (FITZPATRICK; GUILLARD, 2004) and several mineralogical studies (CASTILHOS; MEURER, 2001; VILLA et al., 2004) show differences among soils concerning their K-supply potential. For this reason, K fertilizer recommendations based on soil exchangeable-K (RIBEIRO et al., 1999) might underestimate the soil's capacity of supplying K to plants, especially for perennial crops, such as coffee. A lack of response to K fertilization observed in some studies concerning coffee production suggests that this plant species may use K from non-exchangeable pools in the soil (SILVA et al., 1995).

Coffee plants exude organic substances including citric and oxalic acids (SILVA et al., 2002), which play an important role on K release from non-exchangeable forms (FITZPATRICK; GUILLARD, 2004), as well as on the weathering of K-bearing minerals (BORTOLUZZI et al., 2005), and on physico-chemical characteristics of variablecharge soils (MARCHI et al., 2006a,b). Therefore, studies of K release from soils by organic acids can contribute to a better understanding of chemical methods for evaluating K availability to plants (SILVA et al., 2008).

This investigation was carried out to: i) characterize K pools – total, exchangeable, and non-exchangeable – in soil fractions (whole soil, sand, silt, and clay) of an Oxisol and an Ultisol from Minas Gerais State; and ii) evaluate citrate and oxalate effects on the amount of K released from the studied soils.

MATERIAL AND METHODS

Samples from the B horizon of a basaltderived Oxisol from São Sebastião do Paraíso, EPAMIG experimental station (80-120 cm depth; 20°53'15"S, 47°00'06"O), and of a sienite-derived Ultisol from Poços de Caldas (65-85 cm depth; located at 21°51'29"S, 46°38'30"O), both representative soils from coffee regions of Minas Gerais State were collected (Table 1).

MARCHI, G. et al.

Table 1. Chemical and physical attributes and particle size distribution¹ of B-horizon samples from a basaltderived Oxisol and a sienite-derived Ultisol from coffee regions of Minas Gerais State.¹

Properties	Oxisol	Ultisol
pH _{H2O}	5.9	5.8
$CEC pH 7 (mmol_c kg^{-1})$	27.58	60.17
CEC effective $(mmol_c kg^{-1})$	8.86	26.39
$K (mg kg^{-1})^{(a)}$	29.82	18.87
$P(mg kg^{-1})$	1	1
$Ca (mmol_c kg^{-1})$	5.91	14.78
Mg (mmol _c kg ⁻¹)	1.97	11.61
Al $(\text{mmol}_c \text{kg}^{-1})$	0	0
$H + Al (mmol_c kg^{-1})$	18.71	33.78
Carbon $(g kg^{-1})$	30	70
Organic matter $(g kg^{-1})$	50	120
Sand (g kg ⁻¹)	260	300
Silt $(g kg^{-1})$	240	280
$Clay (g kg^{-1})$	500	420

¹Silva et al. (2008); (a) Embrapa (1997); ^(a) Extracted with Mehlich I (0.05 mol L⁻¹ HCl + 0.0125 mol L⁻¹ H₂SO₄).

Soil samples were air-dried, passed through a 2-mm sieve and then dispersed with 0.1 mol L⁻¹ NaOH. This suspension was submitted to ultrasonic dispersion for 10 minutes (Misonix, XL2020; 20 kHz) and then fractionated into sand (>50 μ m) - by sieving - silt (2-50 μ m), and clay (<2 μ m) - by centrifugation (JACKSON, 1975).

Total- (JACKSON, 1958), HNO₃- (PRATT, 1973) and exchangeable-K (EMBRAPA, 1997) forms were determined in all soil fractions and extracts were analyzed by flame photometry. Non-exchangeable K was calculated from the difference between HNO₃- and exchangeable-K.

Cumulative amounts of K extracted with citrate or oxalate were obtained as follows: 40 mL of the extractant solution $(10^{-3} \text{ mol } \text{L}^{-1} \text{ citrate or} \text{ oxalate in } 0.03 \text{ mol } \text{L}^{-1} \text{ NaCl at } \text{pH} = 5.6)$ were added to 100-mL centrifuge tubes containing 4 g of soil sample (whole soil, sand, silt or clay). These tubes were then shacked head to cap successively during 1.0, 2.72, 4.48, 12.18, 33.11, 90.02, 244.69, and 665.14 hours, and after each of these periods, centrifuged at 2,800 g for 15 minutes. Potassium was measured in supernatant solutions by flame photometry and a new extractant solution was added into the tubes for the next extraction. All extractions were performed in triplicate.

The experiment was conducted in a completely randomized design, arranged in a factorial scheme $2 \times 4 \times 3$ [(2 soils, 4 particle sizes

(clay, silt, sand and whole soil), and 3 extractant solutions (citrate and oxalate 10^{-3} mol L⁻¹, and control)], with three replications. Statistical analyses ANOVA, Tukey tests, and correlation were performed with use of the softwares SISVAR and SAEG.

RESULTS AND DISCUSSION

The total K for the Oxisol was nearly the same in all fractions, while for the Ultisol, the silt fraction was the richer in total K and the clay fraction was the poorer, suggesting that long-term reserves of K for the Ultisol are allocated in the silt fraction (Table 2). The greater amount of total K found in silt than in other soil fractions was probably due to the presence of K-feldspars or micaceous materials of difficult weathering in such fraction (LEPSH et al., 1978). Feldspars and mica are usually the main minerals potentially able to supply K to crops (BORTOLUZZI et al., 2005).

The total-K values from the whole fraction of the Oxisol were higher than those obtained by Silva et al. (1995) for a B horizon of two Oxisols from Paraná State (641 and 565 mg kg⁻¹). The sand fraction presented smaller amounts, while the silt and clay fractions from the Oxisol showed total-K values very close to those verified by Meurer et al. (1996) in an Oxisol from Rio Grande do Sul State (1,500; 2,857 and 3,440 mg kg⁻¹ for the sand, silt, and clay fractions, respectively). The total-K values from the sand fraction of the Oxisol were higher than those verified by Goedert (1973) in the sand 915

fraction of an Oxisol $(1,800 \text{ mg kg}^{-1})$ and lower than in the sand fraction of a Mollisol $(4,400 \text{ mg kg}^{-1})$.

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Fraction		E	xtracted K (mg kg ⁻¹)		
	Total K [§]	HNO ₃ -K	Exchangeable K ^{II}	Non-exchangeable K [#]	
			Oxisol	C C	
Whole soil	2,423.20 a*	44.08 a	29.82 a	14.26 ab	
Sand	2,553.63 a	10.00 b	2.19 d	7.81 bc	
Silt	2,503.47 a	12.13 b	11.31 c	0.82 c	
Clay	3,595.17 a	38.30 a	16.52 b	21.78 a	
-			Ultisol		
Whole soil	11,087.00 b	164.66 ab	18.87 b	145.79 a	
Sand	10,608.00 b	106.93 b	7.92 c	99.01 a	
Silt	17,026.60 a	230.08 a	30.60 a	199.48 a	
Clay	5,722.20 c	112.07 b	19.91 b	92.16 a	
* V 1 (C	4 1 4 1 6 11	11 4 1 4 4			

Table 2. Forms of K in the soils.

*Values (mean of three replicates) followed by the same letter in the columns, inside soil classes, do not differ statistically by the Tukey test (5%); [§]Extracted by HF-HClO₄; ^{II}Extracted by Mehlich I; [#]Non-exchangeable K = HNO₃-K – exchangeable K.

The amount of total K extracted from the different soil fractions of both soils was found to be one to two orders of magnitude higher than the other analyzed forms of K (Table 2), however just a small portion of this form will be available to plants even in the long term, as for perennial crops (e.g., coffee). Thus, only non-exchangeable and exchangeable K are useful to predict K release to soil solution, and therefore, to plants. Exchangeable K is related to the portion readily available and, for annual crops, is the form of K that contributes most to the plant nutrition (CASTILHOS; MEURER, 2001; VILLA et al., 2004).

The Ultisol presented a much greater amount of non-exchangeable K than did the Oxisol (Table 2). This means, for instance, that the Ultisol possess a better K supply in the medium term. Nonexchangeable-K values from the clay fraction of the Oxisol were higher than those from other fractions, but similar to those of the whole soil. Values for HNO₃-K in the whole soil fraction of the Oxisol were similar to those found by Mielniczuk and Selbah (1978) for a whole soil and silt fraction of a basalt-derived Oxisol from Rio Grande do Sul State (46.92 mg kg⁻¹).

The sand and clay fractions of the Ultisol presented similar contents of HNO₃-K and non-exchangeable K. In contrast, the clay fraction of the Oxisol presented higher values of HNO₃-K and non-exchangeable K, which were similar to those found for the whole soil, indicating that the clay fraction holds up the main reserves of K on the Oxisol.

Results of HNO₃-K for the Ultisol were greater than for the Oxisol, which agrees with data

reported by Mielniczuk (1977) who pointed out that highly weathered soils normally present low HNO₃-K values. The HNO₃-K extracted from sand- and clay-fractions of the Oxisol were lower than those determined by Meurer et al. (1996) for an Oxisol, in which was found 288, 200, and 314 mg kg⁻¹ for sand, silt, and clay fractions, respectively. Thus, it is noteworthy that even among soils from the same taxonomic class, there is a great variability in terms of HNO₃-K extractions.

Mielniczuk (1977) suggests that soils presenting HNO_3 -K results smaller than 136 mg kg⁻¹ normally have a small amount of K-supply on the medium term. This limit could then be applied to the studied soils, suggesting that the Ultisol presents higher medium-term supply capacity for K than the Oxisol.

When submitted to successive extractions with citrate or oxalate, both soils released greater amounts of K than the existing amount of exchangeable K, thus indicating that nonexchangeable K was released by organic acids (Table 3). Cumulative results for the successive extractions with citrate or oxalate showed that the Oxisol released similar amounts of K from all soil fractions, while the Ultisol released different amounts of K upon variation of both the soil fraction and the extractant solution. The cumulative amount released by successive extractions with citrate or oxalate in all soil fractions of the Ultisol followed the same decreasing order reported by Castilhos and Meurer (2001) for oxalate-extracted K in a Planosol from Rio Grande do Sul State: silt>clay>sand.

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Fraction	Extracted K (mg kg ⁻)				
	Control [§]	Citrate [#]	Oxalate [#]		
		Oxisol			
Whole soil	30.68 aA*	32.64 aA	33.13 aA		
Sand	2.03 aD	2.92 aD	2.76 aD		
Silt	10.67 bC	11.60 abC	14.01 aC		
Clay	25.33 aB	24.19 abC	22.23 bB		
		Ultisol			
Whole soil	23.48 bB	39.95 aA	20.70 bB		
Sand	14.03 cC	25.45 aD	18.05 bC		
Silt	36.35 aA	36.24 aB	33.48 aA		
Clay	21.38 bB	28.94 aC	22.28 bB		

Table 3. Cumulative amount of K released by citrate or oxalate in 8 successive extractions from the whole soil, sand, silt, and clay fractions of an Oxisol and an Ultisol.¹

¹Silva et al. (2008); *All values are mean of three replicates; no significant difference to same lower and capital case letters in lines and columns, respectively, Tukey test (p < 0.05); [§]0.03 mol L⁻¹ NaCl; [#]0.03 mol L⁻¹ NaCl , 10⁻³ mol L⁻¹ organic acid.

The sand fraction from both soils released the least amount of K. The cumulative amount of organic acids-extractable K from the sand fraction of both soils was greater than the exchangeable form extracted by Mehlich I. The amount of K extracted by the acids followed the order: citrate>oxalate>control.

While for the silt fraction of the Oxisol the cumulative amount extracted by successive extractions with organic acids only released K from the exchangeable pool, for the Ultisol the silt fraction released a greater amount of K when compared to the other fractions.

In the Oxisol, successive extractions with organic acids released much more K from clay than from sand and silt. The cumulative K extracted from the clay by organic acids was also bigger than the exchangeable K. For the clay fraction of the Ultisol, the cumulative amount of K released by successive extractions with organic acids was greater than that extracted from the sand fraction, but smaller than that extracted from the silt fraction.

Cumulative amounts of K released by successive extractions with oxalate and control correlated well with values reported for exchangeable K (Table 4). Results from citrate extraction correlated better with the total- and HNO₃-K than did those for control or oxalate. These results are relevant since the total-, and HNO₃-K analyses are dangerous, even using microwave digestions, and burdensome when compared to successive extractions with citrate.

Despite the fact that the correlation coefficients for exchangeable K and K extracted by citrate were lower than those extracted by oxalate (Table 4), correlation coefficients higher than 0.72 were considered satisfactory by Meurer and Anghinoni (1993) for K absorption by sorghum and amount of exchangeable-K for eleven soils from Rio Grande do Sul State.

Table 4.	Correlation	coefficients	found among	the K amour	nts extracted	l by different	extraction 1	methods for	or the
	whole soil.	sand, silt, an	d clav fraction	ns of an Ultis	ol and an O	xisol.			

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Extractant	Control [§]	Citrate [#]	Oxalate [#]	Mehlich I	HNO ₃
Citrate	0.852***				
Oxalate	0.955***	0.822***			
Mehlich I	0.947***	0.789***	0.944***		
HNO ₃	0.596***	0.729***	0.526**	0.524**	
HF-HClO ₄	0.490***	0.621***	0.433*	0.375*	0.894***
		1 7 2 1 1 2	See 11-121 CT		1.7.1 1 1.1

*** 0.1% of significance; ** 1% of significance; * 5% of significance; $^{\$}0.03$ mol L⁻¹NaCl; $^{\#}0.03$ mol L⁻¹NaCl, 10⁻³ mol L⁻¹organic acid.

CONCLUSIONS

Soil texture alone is not a reliable indicator of K-supply capacity from soils. From successive extractions the non-exchangeable K compartment from the soil was assessed whereas, by using citrate, it is possible to assess the K availability from medium to long term. Thus, the Ultisol showed to have a higher K-supply capacity than the Oxisol. However, the whole soil fraction of the Ultisol released less exchangeable K than the Oxisol Potassium extractability ...

although it has higher K reserves as non-exchangeable- and total-K forms.

While the main source of K was the silt fraction for the Ultisol and the clay fraction for the Oxisol, non-exchangeable K represents a greater reserve for the former than for the latter soil.

No effect of either citrate or oxalate was observed in the Oxisol, which is related to the fact that most of its K supply came from the exchangeable form. Citrate caused a greater release of K from the Ultisol than did oxalate and the control, as part of the K was released from the non-exchangeable forms of K).

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RESUMO: O café, importante mercadoria para o Brasil, é uma cultura que demanda grande quantidade de potássio (K). Os métodos de recomendação de fertilizantes potássicos para lavouras cafeeiras no Brasil são baseados na quantidade de K trocável no solo. Estudos mineralógicos que estimam a capacidade de suprimento de K em diferentes frações do solo, em médio e longo prazo, foram realizados em solos brasileiros, mas poucos foram realizados com foco em extrações sucessivas. No presente estudo, avaliou-se a liberação de K do solo e das frações argila, silte e areia de amostras do horizonte B de um Latossolo Vermelho distrófico e de um Nitossolo Háplico, ambos representativos de regiões cafeeiras de Minas Gerais. Extrações sucessivas de K, com tempos de agitação variando de 0 a 665 h, foram realizadas $com 10^{-3}$ mol L⁻¹ de citrato ou oxalato, na relação solo:solução de 1:10. Os resultados cumulativos foram comparados às formas de K (trocável, não-trocável e total) medidas por diferentes procedimentos de extração. Verificou-se que a concentração de K trocável foi maior e as concentrações de K não-trocável e total foram menores no Latossolo que as mesmas formas de K extraídas do Nitossolo. A fração argila foi a principal fonte de K do Latossolo, ao passo que a fração silte foi a que mais contribuiu para a liberação de K do Nitossolo. Citrato e oxalato extraíram quantidades similares de K do Latossolo, resultado este relacionado ao fato de que a maioria do suprimento de K foi extraída da forma trocável. O citrato extraiu mais K (parte na forma não-trocável) do Nitossolo que o oxalato. Observou-se, neste trabalho, que a textura do solo, isoladamente, não é uma boa indicação da capacidade do solo em suprir K para as plantas, visto que, para o mesmo tamanho de partículas, os solos estudados revelaram extratabilidade de K diferenciada.

PALAVRAS-CHAVE: Cítrico. Oxálico. Textura do solo.

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