Revista Ciência Agronômica, v. 56, e202493055, 2025
Centro de Ciências Agrárias - Universidade Federal do Ceará, Fortaleza, CE
www.ccarevista.ufc.br

Scientific Article
ISSN 1806-6690

Efficiency of available potassium extractants in soils with 2:1 clays in the semi-arid region of Brazil[1]

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ABSTRACT - Evaluating the availability of potassium (K) in soils of the semi-arid region of Brazil is complex, as it varies greatly between soils, especially soils with larger amounts of 2:1 minerals. The aim of this study was to evaluate the efficiency of ammonium acetate, ammonium chloride, Mehlich-1 and mixed ion-exchange resin as extractants in quantifying the K content available to maize plants in soils of the semi-arid region of Brazil. The treatments were distributed in randomised blocks, and included six types of soil (a Red Yellow Argisol, a Fluvic Neosol, two Haplic Cambisols, a Rendzic Chernosol and a Haplic Vertisol) and five levels of K, with three replications. Each plot consisted of a plastic pot containing 3.0 dm^3 soil, where two maize plants were grown for 35 days. There was a good correlation between the extractants under evaluation and the amount of K accumulated by the plants. The ion-exchange resin presented a lower K extraction capacity in the more-clayey soils with a higher CEC and greater quantity of 2:1 minerals, and is not recommended for soils with these characteristics. The extraction capacity of Mehlich-1 may be reduced in alkaline soils with high levels of $CaCO_3$ equivalent, and should not be used in these soils. Ammonium acetate extracted the greatest amounts of K from the soils and showed no limitations in any of the soils under evaluation, making it the most recommended for assessing potassium availability in semi-arid soils.

Key words: Ammonium acetate. Mehlich-1. Mixed ion-exchange resin. Ammonium chloride. Soil analysis.

DOI: 10.5935/1806-6690.20250066
Editor-in-Chief: Profa. Mirian Cristina Gomes Costa - mirian.costa@ufc.br

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Received for publication 28/02/2024; approved on 17/12/2024

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INTRODUCTION

The availability of potassium (K) is controlled by the predominant forms of the element found in the soil (soluble, exchangeable, non-exchangeable and structural) that are in dynamic equilibrium and are influenced by various physical, chemical and mineralogical soil attributes (Zörb; Senbayram; Peiter, 2014).

In Brazil, potassium availability is generally assessed using the Mehlich-1 (Teixeira *et al*., 2017) and mixed ion-exchange resin (Raij *et al*., 2001) extractants. In the states of Rio Grande do Sul and Santa Catarina, Mehlich-3 is also used (Silva *et al*., 2016). These three extractants mainly extract soluble and exchangeable K, which are the predominant fractions in the most-developed soils in the tropical region of the country, and have proved to be effective in estimating available K in these soils.

However, these extractants are unsuitable for semi-arid soils due to the great pedodiversity of the region, where less-developed soils that are rich in 2:1 minerals predominate. These minerals have specific sorption sites for K, which fix the element in their interlayers, forming non-exchangeable K (Bilias; Barbayiannis, 2019) and resulting in a high K buffering capacity (KBC) (Santos *et al*., 2013).

Due to the presence of 2:1 minerals, some soils in the semi-arid region, especially the more clayey soils, can fix up to 80% of the K dose applied via fertilisation (Medeiros *et al*., 2014), making it difficult to extract K using the routine extractants normally used in laboratories.

When evaluating the Mehlich-1, mixed ion-exchange resin and Mehlich-3 extractants in soils from the state of Paraíba with different degrees of pedogenic development, Medeiros *et al*. (2010) found that these extractants were efficient in assessing K availability in highly developed soils. However, in less developed soils collected in the semi-arid region, the mixed ion-exchange resin extracted a lower K content than did Mehlich-1 and Mehlich-3, especially in the more-clayey soils with a higher proportion of 2:1 minerals.

In addition, some soils in the semi-arid region of Brazil may contain high levels of $CaCO_3$ equivalent, which reduces the extraction efficiency of the acid solutions (Mehlich-1 and Mehlich-3) due to the reaction of H^+ ions with carbonate, as verified by Medeiros *et al*. (2021). This reaction reduces the amount of H^+ ions available for exchange with K^+ on the surface of the colloids, resulting in underestimating the availability of the element for plants and a greater likelihood of errors when interpreting and recommending potassium fertilisation.

Among the various methods of K extraction, those whose mechanism of action is the ionic exchange of K^+ for NH_4^+, such as 1 mol L^{-1} ammonium acetate (Breker *et al*., 2019) and 1 mol L^{-1} ammonium chloride (Amorim; Rogeri; Gianello, 2021), may be more efficient in assessing the availability of K in soils rich in 2:1 minerals. This is due to NH_4^+ ions having a hydrated ionic radius similar to that of K^+ ions, allowing soluble and exchangeable K to be extracted from the surfaces of the colloids in addition to part of the K fixed in the interlayers of the 2:1 minerals (Bell; Thompson; Moody, 2021).

The hypothesis of this study is that the 1 mol L^{-1} ammonium acetate and 1 mol L^{-1} ammonium chloride extractants are more efficient in assessing K availability in semi-arid soils with 2:1 minerals than the methods generally used in Brazil.

In view of the above, the aim of this study was to evaluate the efficiency of ammonium acetate, ammonium chloride, Mehlich-1 and mixed ion-exchange resin as extractants in quantifying the K content available to maize plants in soils of the semi-arid region of Brazil.

MATERIAL AND METHODS

Samples were collected from the surface layer (0-30 cm) of six soils from the semi-arid region located between the valleys of the River Jaguaribe in Ceará (CE) and the River Piranhas/Açu in Rio Grande do Norte (RN), under native vegetation and with no history of fertiliser application.

The soils were formed from different parent materials. Four were derived from limestone: a Haplic Cambisol, collected in Baraúna, RN (CX1: 05°05’42.2” S, 37°36’46.7” W); a Haplic Cambisol, collected in Quixeré, CE (CX2: 05°05’07.7” S, 037°51’35.1” W); a Rendzic Chernosol, collected in Mossoró, RN (MD: 05°26’54.7” S, 37°11’19.9’ ‘W); and a Haplic Vertisol, collected in Mossoró, RN (VX: 05°25’26.4” S, 37°12’26” W). One soil was formed from alluvial sediments: a Fluvic Neosol, collected in Carnaubais, RN (RY: 05°22’33.2” S, 36°50’42.2” W); and one soil was derived from sediments of the Barreiras group: a Red Yellow Argisol, collected in Mossoró, RN (PVA: 05°09’58.5’ ‘S, 37°11’57.6” W) (Brazil, 1971, 1973).

The soil samples were air-dried, crushed and passed through a 4-mm mesh sieve. A subsample was removed and passed through a 2-mm mesh sieve for chemical and physical characterisation, as per Teixeira *et al*. (2017). The remaining potassium (K-rem) was quantified by agitating 50 mL of a 10 mmol L^{-1} $CaCl_2$ solution containing 75 mg L^{-1} K and 5 g soil for five minutes, and corresponded to the concentration of K remaining in the solution following equilibrium (Santos, 2022) (Table 1).

The experiment was conducted in a greenhouse at the Federal Rural University of the Semi-Arid (UFERSA), in a randomised block design with three replications, including five doses of K (0, 50, 100, 200 and 300 mg dm^{-3}) applied to six soils (PVA, RY, CX1, CX2, MD and VX). Each experimental unit consisted of a plastic pot with no drainage, containing 3.0 dm^3 of soil where two BRS 2022 hybrid maize plants were grown.

The soil lime requirement was calculated using the base saturation method (Raij *et al*., 1997), and by Al^{3+} neutralisation with Ca^{2+} and Mg^{2+} elevation (Ribeiro; Guimarães; Alvarez, 1999), taking the mean value of both methods. Only the PVA soil needed liming. The corrective comprised a mixture of $CaCO_3$ (p.a.) and $MgCO_3$ (p.a.) at a molar ratio of 4:1, applied to 100% of the volume of soil in the pot. The soil was incubated for 30 days, with moisture corresponding to 50% porosity. Following incubation, the soil was broken up and passed through a 4-mm mesh sieve for later application of the K doses.

The K doses were applied via solution to 3.3 dm^3 of soil in the form of KCl (p.a.). The KCl was diluted in deionised water corresponding to 50% of the total porosity of the soils. The soil samples were then taken to the laboratory, where they were incubated for 30 days in dark plastic bags with a small opening to allow for gas exchange. The samples were weighed weekly, and whenever necessary any evaporated water was replaced with deionised water.

Table 1 - Physical and chemical attributes of six soils from the semi-arid region of Brazil[1]

Attribute	Soil[2]					
	PVA	RY	CX1	CX2	MD	VX
Sand (g kg^{-1})	850	640	660	380	290	290
Silt (g kg^{-1})	20	220	70	220	160	320
Clay (g kg^{-1})	130	140	270	400	550	390
Bulk density (kg dm^{-3})	1.57	1.35	1.42	1.37	1.20	1.28
Total porosity (dm^3 dm^{-3})	0.418	0.492	0.489	0.476	0.522	0.529
K-rem (mg L^{-1})[3]	73.4	58.0	61.3	43.2	21.1	25.7
MKSC (mg g^{-1})[4]	5.45	7.30	9.90	8.55	15.27	15.46
pH	4.1	6.5	6.6	6.7	7.6	8.0
Org. Mat. (g kg^{-1})	7.5	6.2	9.4	11.0	14.4	7.0
P-Mehlich-1 (mg dm^{-3})	4.2	60.7	4.5	4.8	3.1	1.6
K (mg dm^{-3})	35.7	137.3	144.8	149.5	185.8	64.0
Na (mg dm^{-3})	20.1	133.1	54.0	116.3	47.1	76.2
Ca^{2+} (cmol$_c$ dm^{-3})	0.74	6.94	6.04	11.50	40.33	35.73
Mg^{2+} (cmol$_c$ dm^{-3})	0.29	3.10	1.17	1.29	4.77	5.60
Al^{3+} (cmol$_c$ dm^{-3})	0.61	0.00	0.00	0.00	0.00	0.00
(H+Al) (cmol$_c$ dm^{-3})	4.02	1.19	1.58	1.97	0.15	0.00
SB (cmol$_c$ dm^{-3})	1.20	10.97	7.81	13.68	45.78	41.83
t (cmol$_c$ dm^{-3})	1.81	10.97	7.81	13.68	45.78	41.83
CTC (cmol$_c$ dm^{-3})	5.22	12.16	9.39	15.65	45.93	41.83
V (%)	23	90	83	87	100	100
Equiv. $CaCO_3$ (g kg^{-1})[5]	1.59	8.22	4.23	9.49	27.53	254.56
Initial pH with Melichh-1 extraction[6]	1.15	1.32	1.25	1.34	1.55	5.72
Final pH with Melichh-1 extraction[6]	1.24	1.34	1.33	1.46	2.08	6.51

[1]Physical and chemical analyses carried out as per Teixeira *et al*. (2017). [2]PVA = Red-Yellow Argisol (Mossoró, RN); RY = Fluvic Neosol (Carnaubais, RN); CX1 = Haplic Cambisol (Baraúna, RN); CX2 = Haplic Cambisol (Quixeré, CE); MD = Rendzic Chernosol (Mossoró, RN); VX = Haplic Vertisol (Mossoró, RN). [3]MKSC = maximum K sorption capacity estimated by the Langmuir isotherm. [4]K-rem = remaining potassium (Santos, 2022). [5]Data extracted from Medeiros *et al*. (2021). [6]Initial and final pH of the soil-solution suspension with Mehlich-1 extraction. Data extracted from Medeiros *et al*. (2021)

After the incubation period, all of the soil samples were air-dried, crushed, sieved through a 4-mm mesh sieve and then transferred to the pots. Before sowing the maize, a 0.3 dm^3 soil sample was collected from each pot to analyse the K content, which was extracted from the soil using 1 mol L^{-1} ammonium acetate at pH 7.0 (Helmke; Sparks, 1996), 1 mol L^{-1} ammonium chloride (Boeira *et al*., 2004), Mehlich-1 (Teixeira *et al*., 2017), and mixed ion-exchange resin (Raij *et al*., 2001). The extraction procedure for the first three extractants was carried out at a soil to solution ratio (m/v) of 1:10, using a stirring time of five minutes, followed by decantation for 16 hours, in accordance with routine laboratory testing procedures.

One day before planting, all of the soil samples from the pots were fertilised with a nutrient solution. The doses and sources of the nutrients, with the exception of K, were applied as per Medeiros *et al*. (2021): 50 mg dm^{-3} N ($NH_4H_2PO_4$), 200 mg dm^{-3} P ($NH_4H_2PO_4$ and $Ca(H_2PO_4)_2.H_2O$), 4 mg dm^{-3} Mn ($MnCl_2.4H_2O$), 4 mg dm^{-3} Zn ($ZnSO_4.7H_2O$), 1.5 mg dm^{-3} Cu ($CuSO_4.5H_2O$), 0.5 mg dm^{-3} B (H_3BO_3), 0.15 mg dm^{-3} Mo ($(NH_4)_6Mo_7O_{24}.4H_2O$), and 5 mg dm^{-3} Fe ($FeCl_3.6H_2O$ dissolved in EDTA solution).

Five maize seeds were sown in each pot. Five days after emergence, the plants were thinned, leaving two plants per pot. A top dressing of 50 mg dm^{-3} N and 57 mg dm^{-3} S ($(NH_4)_2SO_4$) was applied during the first week, 50 mg dm^{-3} N ($(NH_4)_2SO_4$ and urea) and 23 mg dm^{-3} S ($(NH_4)_2SO_4$) during the second week, and 50 mg dm^{-3} N (urea) during the third week. The pots were weighed twice a day, and deionised water was added to the soils to maintain the soil moisture at 50% of the total porosity of each soil.

After 35 days growth, the maize shoots were cut one centimetre above the level of the soil. The shoots were stored in perforated paper bags to facilitate drying, which was initially carried out on the bench in the laboratory for one day and then in a forced-air circulation oven at 65 °C to constant weight.

The dry weight of the shoots was determined in the dried samples. The samples were then ground and sieved (< 1.7 mm) in a Willey-type mill and stored in plastic containers. From each sample, 0.5 g was transferred to a Teflon tube and then digested using 65% HNO_3 (w.p.) in a wet digestion system in a microwave oven (Silva, 2009). After digestion, the extracts were filtered, and flame photometry was used to quantify the K content of the shoots. Based on the dry matter weight and the K content of the plant, the K content of the shoots in each pot was calculated. The levels of K extracted by the plants was obtained by dividing the K content per pot (extracted by the two plants) by the volume of soil in the pot (3 dm^3).

The data were submitted to analysis of variance and regression analysis. Simple linear regression equations were fitted for the K content determined by the extractants and by the plant as a function of the applied K doses, to obtain the rate of K recovered by the extractants. A simple linear correlation analysis was carried out between the rates recovered by the extractants and the soil characteristics related to K availability, at a significance level of 1% and 5%.

To assess the predictive capacity of the extractants in terms of soil K availability to the plants, simple linear regression equations were fitted for the K content in the shoots as a function of the available K content determined by the extractants (Alvarez V., 1996). The method with an R^2 value > 0.70 was considered suitable.

RESULTS AND DISCUSSION

The amount of extracted K varied between the methods under study. The mean value for available K obtained with ammonium acetate ranged from 146.7 to 523.5 mg dm^{-3}; by ammonium chloride, from 87.3 to 507.5 mg dm^{-3}; by Mehlich-1, from 118.6 to 303.5 mg dm^{-3}; and by the ion-exchange resin, from 63.8 to 184.9 mg dm^{-3}. For the plants the value ranged from 72.6 to 233.4 mg dm^{-3}. Comparing the extractants and the plants in terms of their potassium extraction capacity, the mean amount of K extracted from the soil in decreasing order was ammonium acetate > ammonium chloride > Mehlich-1 > resin > plant (Table 2).

As their mechanism of action is the ionic exchange of K^+ ions for NH_4^+, ammonium acetate and ammonium chloride are able to extract, in addition to the soluble and exchangeable fractions, the K retained in specific adsorption sites found in the ditrigonal pores of the interlayers of 2:1 minerals. This is due to the similarity of their hydrated ionic radii and hydration energies (Bell; Thompson; Moody, 2021), which would explain the higher levels of these elements. On the other hand, Mehlich-1 extracts only soluble K and the exchangeable K retained in the electrical charges on the surface of the organic and inorganic soil colloids (planar sorption sites with low specificity for K) (Firmano *et al*., 2019), while the ion-exchange resin and the plants act by removing K from the solution and diffusing the element to the surface of the resin balls or the plant roots, which is a slow process.

This characteristic of the resin when extracting K explains the lower values found for the extractant in the MD (63.98 mg dm^{-3}) and VX (65.9 mg dm^{-3}) soils, which represent only 12% and 13% of the K extracted by the ammonium acetate. These soils have the highest clay content, the highest values for CEC and MKSC (Table 1) and, probably, the highest proportion of 2:1 minerals, which fix K in specific sorption sites. Since the release of K by the soil solid phase depends on both

Table 2 - Available K content extracted by ammonium acetate (AA), ammonium chloride (AC), Mehlich-1 and mixed ion-exchange resin, or extracted by the plants, for different K doses applied to the soils

Soil[1]	Dose	AA	AC	Mehlich-1	Resin	Plant[2]
	mg dm^{-3}					
PVA	0	45.9	25.5	41.6	48.9	20.8
	50	78.5	47.5	70.6	78.0	52.2
	100	117.0	73.3	119.1	120.6	80.8
	200	215.9	125.8	194.8	192.4	100.5
	300	276.1	164.2	245.6	240.4	108.7
	Mean	146.7	87.3	134.3	136.1	72.6
RY	0	128.4	78.5	138.5	82.7	119.2
	50	145.3	102.6	158.4	90.1	146.8
	100	164.2	122.4	179.2	88.0	142.8
	200	189.8	142.2	207.8	111.3	161.4
	300	237.8	172.0	252.6	129.7	179.7
	Mean	173.1	123.5	187.3	100.4	150.0
CX1	0	169.7	106.9	153.4	108.7	88.3
	50	206.4	128.0	174.9	135.1	106.5
	100	261.8	177.1	226.2	165.1	153.2
	200	340.6	248.2	299.3	227.3	165.7
	300	409.4	311.9	388.8	288.5	204.4
	Mean	277.6	194.4	248.5	184.9	143.6
CX2	0	295.5	200.4	216.1	123.9	99.6
	50	349.2	243.0	237.9	148.9	133.5
	100	371.2	275.8	278.5	153.3	118.6
	200	455.0	367.0	351.2	183.3	154.8
	300	532.8	430.8	433.8	245.6	212.9
	Mean	400.7	303.4	303.5	171.0	143.9
MD	0	455.3	449.8	223.1	54.5	146.0
	50	489.2	480.8	249.6	54.5	161.6
	100	520.5	511.8	266.9	57.3	181.0
	200	538.0	526.3	292.0	71.4	212.7
	300	614.5	568.9	356.5	81.1	233.4
	Mean	523.5	507.5	277.6	63.8	186.9
VX	0	438.0	398.2	84.8	62.1	158.3
	50	466.0	436.9	93.8	58.0	162.0
	100	494.0	491.1	108.6	63.3	199.8
	200	520.6	497.9	138.1	66.2	194.1
	300	597.6	540.5	167.6	80.0	237.4
	Mean	503.2	472.9	118.6	65.9	190.3

[1] PVA = Red Yellow Argisol (Mossoró, RN); RY = Fluvic Neosol (Carnaubais, RN); CX1 = Haplic Cambisol (Baraúna, RN); CX2 = Haplic Cambisol (Quixeré, CE); MD = Rendzic Chernosol (Mossoró, RN); VX = Haplic Vertisol (Mossoró, RN). [2] K contents extracted by two maize plants divided by the volume of soil in the pot

the K concentration of the solution and the diffusion of the element along a concentration gradient between the solution and the soil solid phase, soils with a high CEC and high buffering result in low concentrations of K in the soil solution, which limits the formation of the concentration gradient generated by the removal of K from the solution by the ion-exchange resin, necessary for the release of K from the sorption sites (Bell; Thompson; Moody, 2021).

Medeiros *et al*. (2010) also found a lower potassium extraction capacity for the resin in more-clayey soils with a higher 2:1 mineral content, in the state of Paraíba, showing that the extraction capacity of this extractant for the non-exchangeable fraction is low, and is sensitive to the buffer power of the potassium. Bortolon, Gianello and Schilindwein (2010) found that resin extracted less than 50% of the content extracted by ammonium acetate, and that despite showing a correlation with the content extracted by the plants, the extraction capacity of resin is lower, which would reduce the classes of availability and increase the chance of analytical error. Bortolon, Schilindwein and Gianello (2009) found similar values for potassium extraction capacity between the Mehlich-1 solution and the resin in samples of a Latosol and an Argisol from Rio Grande do Sul.

Although Mehlich-1 generally extracted a greater quantity of K than did resin, its potassium extraction capacity was reduced in soils with a higher $CaCO_3$ equivalent content. In VX, the quantity of K extracted by Mehlich-1 represented only 24% of the K extracted by ammonium acetate (Table 2). This is due to deterioration of the extractant from the neutralisation of its acidity by $CaCO_3$. When evaluating the efficiency of P extractants in the same soil, Medeiros *et al*. (2021) found that the pH of the soil solution suspension using Mehlich-1 was 5.72 at the start of the extraction and 6.51 at the end of the process, while for the other soils in the study, the value was less than 2.0, which would explain its deteriorating and loss of P extraction capacity.

The hypothesis put forward by Medeiros *et al*. (2021) for the poor P extraction in VX can also explain the poor K extraction by Mehlich-1 in VX in this study. It should be noted that the solubilisation of $CaCO_3$ by the reaction with Mehlich-1 releases Ca^{2+} in solution, and this might extract the K. However this appears not to have contributed significantly to the extraction of this element, given the smaller amounts of K extracted by Mehlich-1 compared to Ammonium Acetate, and was probably due to the lower efficiency of Ca^{2+} in removing K^+ at specific adsorption sites, as pointed out by Bell, Thompson and Moody (2021). These authors state that ions such as Ca^{2+} and Ba^{2+} have very stable hydration spheres, so are unable to lose solvate water and penetrate the 2:1 interlayers to take over the sorption sites occupied by K^+.

With ammonium chloride, the amount of K extracted was smaller than with ammonium acetate. However, the results were very similar in the MD and VX soils, which probably present the greatest quantity of 2:1-type minerals, with ammonium chloride extracting 97% and 94% in relation to the amount of K extracted by ammonium acetate, respectively. This confirms the efficiency of NH_4^+ in extracting K^+ from the sorption sites in the interlayers of the 2:1 minerals. Similar results were found by Amorim, Rogeri and Gianello (2021) in soils from Rio Grande do Sul.

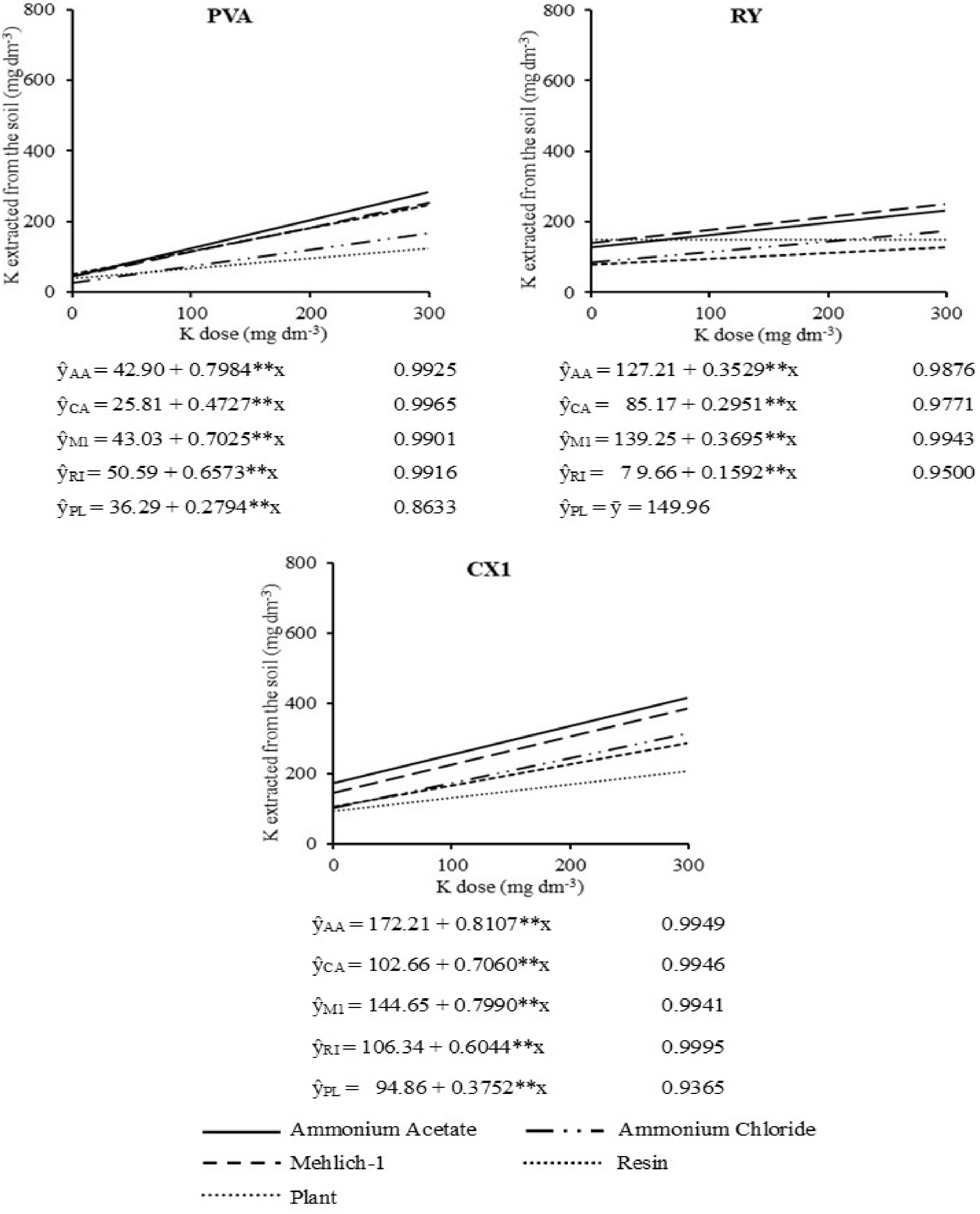
The recovery rate varied both when comparing extractants within the same soil and when comparing the same extractant between all the soils under study (Figures 1 and 2). The PVA, CX1 and CX2 soils showed the smallest variation between extractants, with the exception of ammonium chloride in the first soil and mixed ion-exchange resin in CX1 and CX2. For RY, MD and VX, the recovery rates of the extractants were lower compared to the other soils, this effect being more notable with the resin, which extracted only 15.9%, 9.7% and 6.2% of the K added to these soils, respectively, again demonstrating the low efficiency of the extractant in removing K from more clayey soils with a higher CEC (Table 1) and/or a greater amount of 2:1 minerals. The highest recovery rate, considering all of the soils, was seen with the ammonium acetate extractant; although in RY, this extractant recovered only 35% of the added K.

For all of the soils the extraction rate was less than 100% (Figures 1 and 2), showing that in these soils the fixation process is important, and that sorption occurs at points of high specificity for K, which become inaccessible to the extractants, even those that use NH_4^+ ions as an exchange ion, as is the case with ammonium acetate and ammonium chloride. According to Firmano *et al*. (2019), at specific adsorption sites, the selectivity and retention energy of K can be so high that the K cannot be replaced by other cations with a similar ionic radius, such as NH_4^+, making the buffering of non-exchangeable K to exchangeable K very slow.

This behaviour was not expected in PVA, since this soil is more developed and probably has a predominance of kaolinite and oxides in the mineralogy of the clay fraction. This fixation is possibly linked to traces of micaceous minerals (especially illite). Rêgo *et al*. (2016) found illite in the clay fraction of the same soil, while Mota *et al*. (2007) also found this mineral in the clay fraction and mica in the silt fraction of an Argisol and an Oxisol collected in Mossoró, RN.

The correlation analysis showed that the recovery rate for K of the ammonium acetate extractant correlated with that of the other extractants, and that there was a

Figure 1 - Average K content extracted by ammonium acetate (AA), ammonium chloride (AC), Mehlich-1 (M1), mixed ion-exchange resin (RI) and the plants (PL), for different K doses applied to the soils. PVA = Red Yellow Argisol (Mossoró, RN); RY = Fluvic Neosol (Carnaubais, RN); CX1 = Haplic Cambisol (Baraúna, RN)



PVA

$\hat{y}_{AA} = 42.90 + 0.7984^{**}x$ 0.9925

$\hat{y}_{CA} = 25.81 + 0.4727^{**}x$ 0.9965

$\hat{y}_{M1} = 43.03 + 0.7025^{**}x$ 0.9901

$\hat{y}_{RI} = 50.59 + 0.6573^{**}x$ 0.9916

$\hat{y}_{PL} = 36.29 + 0.2794^{**}x$ 0.8633

RY

$\hat{y}_{AA} = 127.21 + 0.3529^{**}x$ 0.9876

$\hat{y}_{CA} = 85.17 + 0.2951^{**}x$ 0.9771

$\hat{y}_{M1} = 139.25 + 0.3695^{**}x$ 0.9943

$\hat{y}_{RI} = 79.66 + 0.1592^{**}x$ 0.9500

$\hat{y}_{PL} = \bar{y} = 149.96$

CX1

$\hat{y}_{AA} = 172.21 + 0.8107^{**}x$ 0.9949

$\hat{y}_{CA} = 102.66 + 0.7060^{**}x$ 0.9946

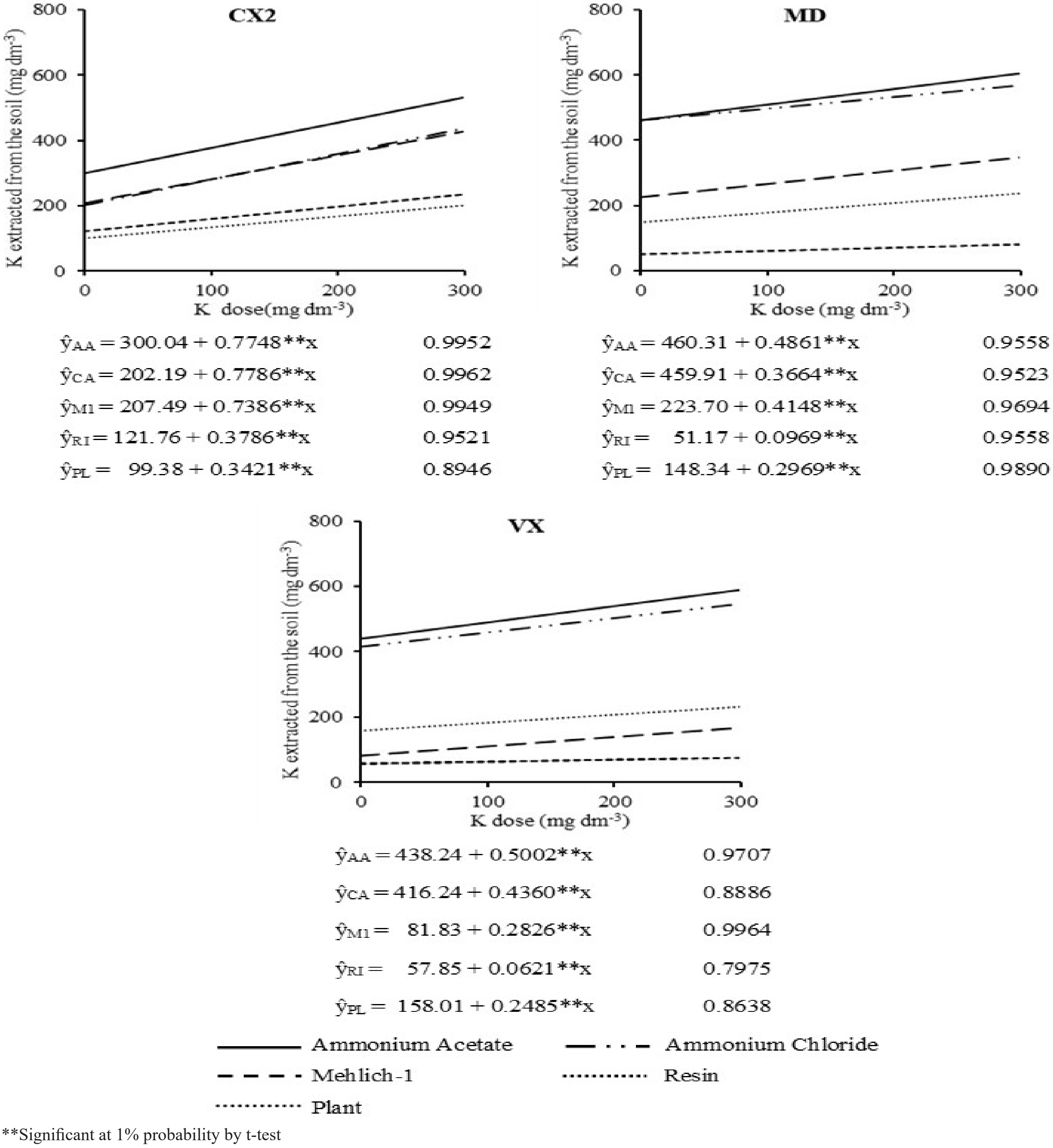
$\hat{y}_{M1} = 144.65 + 0.7990^{**}x$ 0.9941

$\hat{y}_{RI} = 106.34 + 0.6044^{**}x$ 0.9995

$\hat{y}_{PL} = 94.86 + 0.3752^{**}x$ 0.9365

**Significant at 1% probability by t-test significant correlation between the Mehlich-1 extractants and the resin (Table 3). The recovery rates of the extractants did not correlate significantly with the soil attributes that reflect K availability or the K buffering capacity, with the exception of the resin, which showed a significant correlation with the effective CEC (-0.82*) and with K-rem (0.82*) (Table 3), meaning that this extractant recovers a smaller proportion of the K dose applied to soils with a higher effective CEC, probably due to greater K fixation and the greater buffering of these soils.

Figure 2 - Average K content extracted by ammonium acetate (AA), ammonium chloride (AC), Mehlich-1 (M1), mixed ion-exchange resin (RI) and the plants (PL), for different K doses applied to the soils. CX2 = Haplic Cambisol (Quixeré, CE); MD = Rendzic Chernosol (Mossoró, RN); VX = Haplic Vertisol (Mossoró, RN)



**Significant at 1% probability by t-test

It was expected that there would be a significant correlation with attributes that reflect K availability and the KBC, given that these reflect the K sorption capacity and the dynamic equilibrium of the K retained by the solid phase and the K in solution, especially CEC and MKSC; this was possibly due to the high level of K fixation by the soils.

The lack of correlation between attributes that reflect K availability and the recovery rate of the Mehlich-1 extractant was also found by Medeiros *et al*. (2010) in less-developed soils in the State of Paraíba, while for resin, these authors found a significant correlation between the recovery rate and the CEC (-0.81*), effective CEC (-0.82*), silt/clay (-0.69*), Ki (-0.84*) and clay (-0.81*). Kalyani, Sailaja and Rao (2017) found a correlation with the levels of K extracted by resin and the CEC (0.73**), organic carbon (0.65**) and clay (0.96**) in soils from the Rangareddy district of India.

The data on the dry matter production and K content of the plants as a function of the K doses applied to the soils (Table 4) show that maize growth did not increase with increases in applied K, even in the PVA soil, which has the lowest available K content (Table 1), although the K content of the dry matter increased in all of the soils (Tables 4 and 5), indicating possible luxury consumption. This behaviour was also seen in maize plants grown in soils with different degrees of pedogenic development in the state of Paraíba (Medeiros *et al*., 2010).

It should be noted that the maximum accumulation of dry matter and nutrients by the maize plants occurs at around 80 days, with the greatest rate of accumulation between 54 and 63 days, while K absorption is highest during the first 30 days (Borin; Lana; Pereira, 2010). It is therefore probable that excess K was absorbed at the start of the cycle, and the accumulated content in the plant tissues will then be diluted, and the luxury consumption will cease.

Simple linear regression equations were adjusted to estimate the levels of K extracted from the soil by the plants as a function of the levels of available K extracted from the soil by the ammonium acetate, ammonium chloride, Mehlich-1 and resin extractants (Table 6). All of these extractants were shown to be efficient in predicting K availability for the maize plants, with values for the coefficient of variation greater than 70%, as noted by Alvarez V. *et al*. (1996). Similar results were found by Steiner *et al*. (2009), Medeiros *et al*. (2010), Steiner *et al*. (2014) and Amorim, Rogeri and Gianello (2021).

Although all of the methods are efficient in assessing K availability, considering the soils, it should be noted that the lower extraction capacity of the resin, especially in more-clayey soils and those with a higher CEC (Table 1), can cause difficulties in interpreting the K availability in these soils. Furthermore, for soils with a higher $CaCO_3$ content, the Mehlich-1 extractant is subject to deterioration, and should not be used for soils with this characteristic, as this can result in overestimated fertiliser recommendations.

In addition, other factors should be considered when selecting an analytical method, such as the cost, time and labour involved in carrying out the analyses and the degree of environmental impact caused by disposing of the solutions resulting from these analyses (Medeiros *et al*., 2010).

Table 3 - Simple linear correlation coefficients between the slopes of the simple linear regression equations for estimating the available K content using the ammonium acetate (AA), Mehlich-1 (M1), mixed ion-exchange resin and ammonium chloride (AC) extractants and the plants, as a dependent variable of the applied K doses, and the chemical and physical characteristics of the soils

	T	t	Kf	MKSC	K-rem	Argila	Planta	CA	Resin	M1
AA	-0.54	-0.55	-0.55	-0.41	0.48	-0.14	0.79	0.83	0.88	0.91
M1	-0.73	-0.74	-0.71	-0.61	0.64	-0.27	0.62	0.80	0.91	-
RTIM	-0.81	-0.82	-0.80	-0.70	0.82	-0.53	0.46	0.58	-	-
CA	-0.38	-0.38	-0.41	-0.21	0.19	0.12	0.74	-	-	-
Planta	0.08	0.06	0.07	0.22	-0.12	0.47	-	-	-	-
Argila	0.83	0.83	0.84	0.83	-0.91	-	-	-	-	-
K-rem	-0.96	-0.96	-0.95	-0.92	-	-	-	-	-	-
MKSC	0.95	0.96	0.93	-	-	-	-	-	-	-
Kf	0.99	0.99	-	-	-	-	-	-	-	-
T	1.00	-	-	-	-	-	-	-	-	-

T = CEC at pH 7.0; t = effective CEC; K-rem = remaining potassium; MKSC = maximum K sorption capacity; Kf = Freundlich K constant. Values in bold show a significant correlation at 5% probability

Table 4 - DM dry matter production and K content in maize plants for different doses of K applied to the soils

Soil[1]	Dose	DM	K Content	Soil[1]	Dose	DM	K Content
	mg dm^{-3}	g pot^{-1}	g kg^{-1}		mg dm^{-3}	g pot^{-1}	g kg^{-1}
PVA	0	6.5	9.6	CX2	0	14.2	21.4
	50	10.6	14.8		50	13.8	28.9
	100	11.6	21.2		100	13.1	28.7
	200	9.1	33.7		200	13.5	40.2
	300	8.3	39.5		300	15.0	42.8
	Mean	9.2	23.7		Mean	13.9	32.4
RY	0	16.9	21.8	MD	0	15.6	28.0
	50	16.2	27.2		50	16.4	29.8
	100	16.2	26.4		100	16.2	33.8
	200	17.5	27.5		200	16.9	37.8
	300	19.4	35.3		300	16.8	41.2
	Mean	17.2	27.6		Mean	16.4	34.1
CX1	0	15.8	16.6	VX	0	15.6	31.9
	50	14.8	21.7		50	15.0	32.5
	100	16.6	27.7		100	17.9	34.1
	200	15.2	32.6		200	16.8	35.3
	300	15.2	40.3		300	16.7	42.8
	Mean	15.5	27.8		Mean	16.4	35.3

[1] PVA = Red-Yellow Argisol (Mossoró, RN); RY = Fluvic Neosol (Carnaubais, RN); CX1 = Haplic Cambisol (Baraúna, RN); CX2 = Haplic Cambisol (Quixeré, CE); MD = Rendzic Chernosol (Mossoró, RN); VX = Haplic Vertisol (Mossoró, RN)

Table 5 - Regression equations to estimate the dry matter production (y, in g pot^{-1}) and K content (y, in g kg^{-1}) of maize plants, as a function of the applied K dose (x, in mg dm^{-3}) in semi-arid soils

Soil[1]	Caracteristic	Equation	R^2
PVA	Dry Matter	$\hat{y} = \bar{y} = 9.23$	0.9815
	K Content	$\hat{y} = 10.31 + 0.103425^{**}x$	
RY	Dry Matter	$\hat{y} = \bar{y} = 17.24$	0.8266
	K Content	$\hat{y} = 22.88 + 0.036552^{**}x$	
CX1	Dry Matter	$\hat{y} = \bar{y} = 15.55$	0.9796
	K Content	$\hat{y} = 17.88 + 0.076213^{**}x$	
CX2	Dry Matter	$\hat{y} = \bar{y} = 13.92$	0.9323
	K Content	$\hat{y} = 23.15 + 0.071080^{**}x$	
MD	Dry Matter	$\hat{y} = \bar{y} = 16.35$	0.9807
	K Content	$\hat{y} = 28.27 + 0.045086^{**}x$	
VX	Dry Matter	$\hat{y} = \bar{y} = 16.42$	0.8717
	K Content	$\hat{y} = 30.89 + 0.034017^{**}x$	

[1] PVA = Red Yellow Argisol (Mossoró, RN); RY = Fluvic Neosol (Carnaubais, RN); CX1 = Haplic Cambisol (Baraúna, RN); CX2 = Haplic Cambisol (Quixeré, CE); MD = Rendzic Chernosol (Mossoró, RN); VX = Haplic Vertisol (Mossoró, RN). **Significant at 1% probability by t-test

Table 6 - Simple linear regression equations to estimate the K content extracted from the soil by the plants (y, in mg dm^{-3}) as a function of the available K content (x, in mg dm^{-3}) extracted from the soil by the ammonium acetate, ammonium chloride, Mehlich-1 and mixed ion-exchange resin extractants, in soils from the semi-arid region of Brazil

Soil[1]	Extratant	Equation	R[2]
PVA	Ammonium acetate	$\hat{y} = 21.52 + 0.3483^{*}x$	0.8612
	Ammonium chloride	$\hat{y} = 20.61 + 0.5959^{*}x$	0.8804
	Mehlich-1	$\hat{y} = 18.35 + 0.4039^{*}x$	0.8989
	Resin	$\hat{y} = 13.88 + 0.4316^{*}x$	0.8978
RY	Ammonium acetate	$\hat{y} = 63.70 + 0.4984^{*}x$	0.8965
	Ammonium chloride	$\hat{y} = 75.44 + 0.6033^{**}x$	0.9283
	Mehlich-1	$\hat{y} = 59.91 + 0.4809^{*}x$	0.9089
	Resin	$\hat{y} = 42.23 + 1.0734^{*}x$	0.8810
CX1	Ammonium acetate	$\hat{y} = 14.13 + 0.4665^{**}x$	0.9564
	Ammonium chloride	$\hat{y} = 40.10 + 0.5325^{**}x$	0.9452
	Mehlich-1	$\hat{y} = 27.26 + 0.4682^{**}x$	0.9368
	Resin	$\hat{y} = 29.27 + 0.6184^{**}x$	0.9300
CX2	Ammonium acetate	$\hat{y} = -35.32 + 0.4471^{**}x$	0.9221
	Ammonium chloride	$\hat{y} = 11.74 + 0.4355^{*}x$	0.8824
	Mehlich-1	$\hat{y} = 3.69 + 0.4610^{*}x$	0.8944
	Resin	$\hat{y} = -13.59 + 0.9208^{**}x$	0.9762
MD	Ammonium acetate	$\hat{y} = -115.43 + 0.5780^{**}x$	0.9248
	Ammonium chloride	$\hat{y} = -206.70 + 0.7760^{**}x$	0.9521
	Mehlich-1	$\hat{y} = -2.86 + 0.6837^{**}x$	0.9303
	Resin	$\hat{y} = 2.61 + 2.8909^{**}x$	0.9242
VX	Ammonium acetate	$\hat{y} = -64.52 + 0.5064^{**}x$	0.9237
	Ammonium chloride	$\hat{y} = -71.23 + 0.5531^{*}$	0.9152
	Mehlich-1	$\hat{y} = 86.35 + 0.8769^{*}x$	0.8611
	Resin	$\hat{y} = -39.97 + 3.4938^{*}x$	0.8281

[1] K content extracted by two maize plants divided by the volume of soil in the pot. [2] PVA = Red Yellow Argisol (Mossoró, RN); RY = Fluvic Neosol (Carnaubais, RN); CX1 = Haplic Cambisol (Baraúna, RN); CX2 = Haplic Cambisol (Quixeré, CE); MD = Rendzic Chernosol (Mossoró, RN); VX = Haplic Vertisol (Mossoró, RN)

CONCLUSIONS

1. Each of the extractants under evaluation showed a good correlation with the K accumulated by the plant;
2. The ion-exchange resin showed a lower capacity for extracting K in more-clayey soils, with a higher CEC and greater quantity of 2:1 minerals, and is not recommended for soils with these characteristics;
3. The Mehlich-1 extractant may show a reduced capacity for extracting K in alkaline soils with high levels of $CaCO_3$ equivalent, and should not be used for soils with these characteristics;
4. Ammonium acetate extracted the greatest amounts of K from the soils and showed no limitations in any of the soils under evaluation, making it the most recommended for evaluating potassium availability in semi-arid soils.

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