

## NOTA CIENTÍFICA

### EXTRACTION METHODS AND POTASSIUM AVAILABILITY FOR SOYBEAN IN THE PARANA STATE'S SOILS, BRAZIL

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**ABSTRACT:** Evaluating the potassium (K) availability to the plants can be performed by different methods. In general, multielement methods are employed and they are used for K due to increase operational of soil testing laboratory. The efficacy of ammonium acetate, Mehlich-1, Mehlich-3 and ion exchange resin soil K extractants in determining the K availability for soybean [*Glycine max* L. (Merrill)], in different Parana's soils, were investigated in this study. Twelve soil samples, collected from the upper layer 0–0.20 m, were cultivated with soybean for 42 days in greenhouse conditions. The average amount of available soil K extracted in test soils was found, displayed in decreasing order: ammonium acetate > Mehlich-3 > Mehlich-1 = ion exchange resin. The correlation coefficients between the K extracted content by ammonium acetate, Mehlich-1, Mehlich-3 and ion exchange resin and the K accumulated amount in the plants were, respectively, 0.90, 0.77, 0.88 and 0.89. The efficacy of ammonium acetate, Mehlich-3 and ion exchange resin extractants in assessing plant-available K in soils from Parana State were similar. The Mehlich-1 was the least efficient in estimating plant-available K in Parana's soils.

**KEY WORDS:** *Glycine max*, exchangeable K, extractants, soil tests.

### MÉTODOS DE EXTRAÇÃO E DISPONIBILIDADE DE POTÁSSIO PARA A SOJA EM SOLOS DO PARANÁ

**RESUMO:** A avaliação da disponibilidade de potássio (K) para às plantas pode ser realizada por diferentes métodos. Em geral, são métodos multielementares e utilizados para o K em razão da maior operacionalidade nos laboratórios de análises de solo. Neste estudo avaliou-se a eficiência dos extratores acetato de amônio, Mehlich-1, Mehlich-3 e resina de troca iônica na avaliação da disponibilidade de K para às plantas de soja [*Glycine max* L. (Merrill)] em diferentes solos do Estado do Paraná. Amostras de doze solos coletadas na camada de 0–0,20 m de profundidade foram cultivadas com plantas de soja por um período de 42 dias, em casa de vegetação. A capacidade de extração de K dos solos seguiu a seguinte ordem decrescente da quantidade média de K extraído: acetato de amônio > Mehlich-3 > Mehlich-1 = resina. Os coeficientes de correlação entre o teor de K extraído pelo acetato de amônio, Mehlich-1, Mehlich-3 e resina e a quantidade de K acumulada nas plantas foram, respectivamente, de 0,90, 0,77, 0,88 e 0,89. Os extratores acetato de amônio, Mehlich-3 e resina de troca iônica apresentaram eficiência semelhante na avaliação da disponibilidade de K às plantas, e podem ser utilizados para quantificar os teores de K em solos do Estado do Paraná. O extrator

Mehlich-1 foi menos eficiente em estimar a disponibilidade de K para as plantas em solos do Paraná.

**PALAVRAS-CHAVE:** *Glycine max*, K trocável, extratores, análise de solo.

## INTRODUCTION

The evaluation of potassium (K) availability in soil is accomplished by the estimating their amount in readily available pools to plants – i.e., soil solution K and exchangeable K. However, in many situations the predicting plant available soil K has proven to be a difficult task because the plants typically remove more K from the soil than is present in these two pools (ROSOLEM et al., 2012; GARCIA et al., 2008; KAMINSKI et al., 2007; MOODY; BELL, 2006). When solution and exchangeable K are reduced to low levels by plant uptake, non-exchangeable K (fixed pools) can be released from the soils to replenish the plant available pool of K ions (ROSOLEM et al., 2012).

Various extractants and methods have been used for assessing the portion of soil K that is available to plants. The first purpose of each extractant is the determination of available nutrients for plants. Two parameters are very important, when an extractant is utilized: (i) evaluating the nutrient labile form and (ii) extracting methods that are fast and economic. The neutral 1 mol L<sup>-1</sup> ammonium acetate (NH<sub>4</sub>OAc) solution, which extracts both solution and exchangeable K is the most common extractant in determining available K (NACHTIGALL; RAIJ, 2005). The method is simple, fast, low cost and well accurate. However, for increasing the efficiency of soil testing laboratory, extractants that extract several elements simultaneously are more attractive. In Brazil, the Mehlich-1 (TEDESCO et al., 1995) and the ion exchange resin (RAIJ et al., 2001) soil K extractants are the methods used to assess K availability to plants.

The selection of an extractant to determine the availability of a nutrient in the soil is based on the standard extractant – i.e., the plant. Martins et al. (2004) used 1 mol L<sup>-1</sup> NH<sub>4</sub>OAc, Mehlich-1, and boiling nitric acid (HNO<sub>3</sub>) at different concentrations (0.25, 0.5, 1.0, 2.0 and 4.0 mol L<sup>-1</sup>) in extracting available K for wheat in soils of Campos Gerais, Paraná, Brazil. They found that 1 mol L<sup>-1</sup> NH<sub>4</sub>OAc and boiling 4 mol L<sup>-1</sup> HNO<sub>3</sub> extractants were good indices for K availability. Slaton et al. (2010) compared Mehlich-3 and 1 mol L<sup>-1</sup> HNO<sub>3</sub> and found that Mehlich-3-extractable K is an excellent predictor of soil K availability for soybean grown on silt loams in eastern Arkansas. Bortolon et al. (2010) found that Mehlich-1, Mehlich-3, and ion exchange resin extractants were good indices for available K estimation for soybean and wheat in soils of southern Brazil. On the other hand, Hosseinpour and Zarenia (2012) showed that 1 mol L<sup>-1</sup> NH<sub>4</sub>OAc, 0.1 mol L<sup>-1</sup> BaCl<sub>2</sub>, 0.1 mol L<sup>-1</sup> HCl, and boiling 1 mol L<sup>-1</sup> HNO<sub>3</sub> can not be used as available K extractants, whereas 0.1 mol L<sup>-1</sup> HNO<sub>3</sub>, Mehlich-1, and 0.01 mol L<sup>-1</sup> CaCl<sub>2</sub> extractants would be suitable as soil testing methods for determining available K for common bean in Iran soils.

Many researchers suggested various extractants for available K determination according to research sites (MEDEIROS et al., 2010; HOSSEINPUR; SAMAVATI, 2008; ZBÍRAL; NEMEC, 2005; WANG et al., 2004; MARTINS et al., 2004). Therefore, it is necessary to assess the ability of K extractants to predict plant-available K in a wide range of soils and plants.

In Paraná State, there have been few studies on methods for assessing K availability, thus making it difficult to define the most appropriate method for soil conditions in Paraná. However, it is essential to conduct research relating the amount extracted by the different

extractants and the amount of K accumulated by plants using different extractants in soil testing laboratories.

The objectives of this study were to assess and compare the efficacy of ammonium acetate, Mehlich-1, Mehlich-3, and ion exchange resin extractants in determining potassium availability for soybean in different Paraná soils.

## MATERIAL AND METHODS

The experiment was carried out in an experimental greenhouse, localized in the Universidade Estadual do Oeste do Paraná, in Marechal Cândido Rondon, Paraná, Brazil (24°31' S, 54°01' W and altitude of 420 m), where the environmental conditions were: minimum and maximum mean air temperature of 18 and 36°C, respectively; mean air relative humidity of 65%.

Soil samples were collected from the upper layer 0–0.20 m at twelve sites in Paraná State, covering different regions and characteristics (Table 1). The physic and chemical properties of soils (Table 2) were determined by adopting standard procedures (EMBRAPA, 1999).

Table 1. Soil classification, parent material and sampling site of the twelve soils used in the experiment

Soil	Brazilian soil classification <sup>†</sup>	US taxonomy <sup>††</sup>	Parent material	Sampling Municipality
Ox1	Red Latosol	Rhodic Acrudox	Basalt	Marechal Cândido Rondon
Ox2	Red Latosol	Rhodic Hapludox	Shale	Ponta Grossa
Ox3	Red-Yellow Latosol	Typic Hapludox	Caiuá sandstone	Umuarama
Ox4	Red-Yellow Latosol	Typic Hapludox	Furnas sandstone	Ponta Grossa
Alf1	Red Nitisol	Typic Hapludalf	Basalt	Marechal Cândido Rondon
Alf2	Red Nitisol	Typic Hapludalf	Shale	Ponta Grossa
Alf3	Haplic Plinthosol	Typic Plinthaqualf	Shale	Ponta Grossa
Ult1	Red-Yellow Argisol	Arenic Hapludult	Caiuá sandstone	Umuarama
Ult2	Red-Yellow Argisol	Arenic Hapludult	Basalt	Mercedes
Ent	Regolithic Neosol	Typic Usthorthent	Basalt	Marechal Cândido Rondon
Ert	Haplic Gleysol	Typic Endoaquert	Alluvial sediments	Marechal Cândido Rondon
Ept	Haplic Cambisol	Typic Fragiudept	Furnas sandstone	Ponta Grossa

<sup>†</sup> Brazilian soil classification (EMBRAPA, 2006). <sup>††</sup> Approximate equivalence to USDA soil taxonomy (Soil Survey Staff, 2010).

Lime (CaO 25%, MgO 12% and EEC 96%) was applied before the experiment to raise base saturation up to 70% for clay soils, 50% for sandy soils and 60% for medium texture soils (Raij et al., 1997). The soils were then moistened to reach 80% water retention capacity and incubated for 25 days. The range of base saturation was 55–74%, of pH in CaCl<sub>2</sub> 5.6–6.4 and of organic matter 7–32 g dm<sup>-3</sup>. Afterwards, 7.5-L subsamples of each soil were transferred to 8-L plastic pots.

Soybean [*Glycine max* (L.) Merrill, cv. CD 215] was sown, and seven days after seedling emergence, they were thinned to two plants per pot. Fertilization was performed with macro and micronutrients in solution four days after plant emergence, as recommended for soybean crop grown under greenhouse as described by Novais et al. (1991), with modification. The only exception was the K, which was omitted the application this nutrient.

The total amounts of fertilizer were: 40 mg dm<sup>-3</sup> of N (ammonium sulfate), 160 mg dm<sup>-3</sup> P (triple superphosphate), 15 mg dm<sup>-3</sup> of S (calcium sulfate), 5 mg dm<sup>-3</sup> of Cu (copper sulfate), 5 mg dm<sup>-3</sup> of Zn (zinc sulfate) and 2 mg dm<sup>-3</sup> of B (boric acid). At 15 and 30 days after emergence, the application of 40 mg dm<sup>-3</sup> N as urea solution was also performed. Soil water content was monitored daily and kept around 80% of the soil water retention capacity.

Table 2. Soil chemical properties and clay content of the different soils from Paraná State used in the experiment

Soil	pH	Organic matter	Clay	S	CEC	V	K <sub>non-ex</sub>	PBC <sup>K</sup>
		----- g kg <sup>-1</sup> -----		---- mmol <sub>c</sub> dm <sup>-3</sup> ----		%	mg kg <sup>-1</sup>	
Ox1	4.6	22.7	900	53	149	35	231	7.8
Ox2	3.8	21.3	785	34	141	25	450	3.5
Ox3	4.9	20.3	250	63	129	64	192	4.1
Ox4	4.2	29.4	315	42	131	31	207	4.7
Alf1	5.2	32.7	550	78	157	49	539	9.2
Alf2	3.9	31.8	780	31	161	19	374	5.8
Alf3	5.2	9.1	85	26	64	43	134	1.3
Ult1	3.7	30.0	700	81	166	49	208	12.8
Ult2	5.1	15.7	635	86	153	56	567	10.9
Ent	3.8	31.2	615	18	142	12	263	2.1
Ert	3.6	20.7	450	59	175	34	384	6.7
Ept	5.2	16.2	235	42	99	42	211	2.6

pH in 0.01M CaCl<sub>2</sub>, soil:solution ratio (1:2,5). Organic matter, Walkley-Black method. Clay content determined by the pipette method (EMBRAPA, 1997). S: sum of exchangeable basic cations (Ca + Mg + K). CEC: cation exchange capacity. V: soil base saturation. K<sub>non-ex</sub>: non-exchangeable K, obtained as the difference in the amount of K extracted with boiling 1N HNO<sub>3</sub> and K extracted with 1N NH<sub>4</sub>OAc solution buffered to pH 7.0 (KNUDSEN et al., 1982). PBC<sup>K</sup>: potential buffering capacity of K [in (mmol<sub>c</sub> kg<sup>-1</sup>)/(mmol L<sup>-1</sup>)<sup>1/2</sup>] determined as previously described by Mielniczuk (1978).

Forty days after plant emergence, the shoots were harvested, washed and oven-dried at 65 °C for 72 h, and then weighed. The plant material was ground, digested in nitric-perchloric acid, and the K concentration analyzed by a flame photometer (MALAVOLTA et al., 1997). The total K amount in the above-ground plant parts was calculated from the K concentration and dry matter data.

Before starting the experiment, the soil from each pot was sampled, oven-dried at 50 °C for 48 h and ground to pass through a 2.0 mm mesh screen.

The amount of soil-available K was extracted by the following methods: (i) ammonium acetate (1 mol L<sup>-1</sup> CH<sub>3</sub>COONH<sub>4</sub>; pH 7.0) in a soil:solution ratio of 1:10, and shaken for 15 min on a reciprocating shaker at 120 oscillations min<sup>-1</sup> and standing overnight (16 h) as previously described by Sanzonowicz and Mielniczuk (1985); (ii) Mehlich-1 (0.05 mol L<sup>-1</sup> HCl + 0.0125 mol L<sup>-1</sup> H<sub>2</sub>SO<sub>4</sub>; pH 1.2) in a soil:solution ratio of 1:10, shaken for 5 min on a reciprocating shaker at 120 oscillations min<sup>-1</sup> and standing overnight (16 h) as previously described (TEDESCO et al., 1995); (iii) Mehlich-3 (0.2 mol L<sup>-1</sup> CH<sub>3</sub>COOH + 0.25 mol L<sup>-1</sup> NH<sub>4</sub>NO<sub>3</sub> + 0.015 mol L<sup>-1</sup> NH<sub>4</sub>F + 0.013 mol L<sup>-1</sup> HNO<sub>3</sub> + 0.001 mol L<sup>-1</sup> EDTA; pH 2.5) in a soil:solution ratio of 1:10, shaken for 5 min on a reciprocating shaker at 220 oscillations min<sup>-1</sup> and standing overnight (16 h) (TEDESCO et al., 1995); and, (iv) ion exchange resin in a soil:solution ratio of 1:20, and shaken for 16 h on a reciprocating shaker at 120 oscillations min<sup>-1</sup> (RAIJ et al., 2001). In all extracts, K concentration was determined by a flame photometer.

The experiment was arranged in a randomized block design with four replicates (an individual pot containing two plant represented one replicate). The K content extracted by  $\text{NH}_4\text{OAc}$ , Mehlich-1, Mehlich-3 and resin and the accumulated amounts in the shoots of soybean plants were subjected to analysis of variance (ANOVA); the effect of contents was partitioned for each extractant by linear regression analysis. Correlation analyses between K extracted by four extractants were performed. In addition to the selection of better extractants, simple correlation coefficients were calculated between K extracted by chemical methods and amount accumulated by plants. All analyses were performed using SigmaPlot version 11.0 software for Windows (Systat Software, Inc., San Jose, CA, USA).

## RESULTS AND DISCUSSION

The amounts of K extracted using the different soil tests are shown in Table 3. Soil K amounts extracted with  $1 \text{ mol L}^{-1} \text{NH}_4\text{OAc}$  were, on average, higher than the K amounts extracted with Mehlich-1 and resin (Table 3). Potassium amounts extracted by  $\text{NH}_4\text{OAc}$  solution were 34% higher than the amounts extracted with Mehlich-1 (Table 3). These results confirm those reported by other authors (BORTOLON et al., 2010; HOSSEINPUR; SAMAVATI, 2008; ZBÍRAL; NEMEC, 2005; WANG et al., 2004). The agitation time of soils (15 min for  $\text{NH}_4\text{OAc}$  and 5 min for Mehlich-1) may be one of the factors that resulted in greater extraction of the K by  $\text{NH}_4\text{OAc}$  solution, by enabling higher contact time and ion exchange between K ion of soil exchange sites and the  $\text{NH}_4^+$  ion of extraction solution (HABBY et al., 1990).

The lowest K amounts extracted by the ion exchange resin (Table 3) may be associated with the extraction mechanism of the method. The acid extractors (Mehlich-1 and Mehlich-3) and neutral ( $\text{NH}_4\text{OAc}$ ) displace most of the soil exchangeable K considered available for correlation studies while in the extraction with resin occurs the removal by K exchange in the solution and that can move to the solution (TEDESCO et al., 1995). This difference in the extraction mechanism provides different levels of K extracted among the methods. The lower K extraction of the resin was also reported by other studies (MEDEIROS et al., 2010; BORTOLON et al., 2010; SCHLINDWEIN; GIANELLO, 2005).

Soil K amount extracted with  $\text{NH}_4\text{OAc}$  was similar to the amount extracted with Mehlich-3 (Table 3), confirming the results obtained by Bortolon et al. (2010), for soils of Rio Grande do Sul, Brazil. The similarity between these two extractors is due to the fact of extract similar K pools, mainly due to the formation of  $\text{NH}_4\text{OAc}$  during compounding of Mehlich-3 solution (MEHLICH, 1984).

Table 3. Mean values, minimum and maximum for soil potassium concentrations extracted by different extractant solutions, potassium content and accumulation in the soybean shoot and dry matter production (n = 48)

Parameter	Soil potassium				Plant potassium		Dry matter
	$\text{NH}_4\text{OAc}$	Mehlich-1	Mehlich-3	Resin	Content	Accumulated	
	----- $\text{mg dm}^{-3}$ -----				$\text{g kg}^{-1}$	$\text{mg pot}^{-1}$	$\text{g pot}^{-1}$
Mean	160	124	140	118	20.0	266	14.7
Minimum	45	38	25	22	16.2	112	7.7
Maximum	314	238	296	218	22.8	454	22.4
Median	152 A	113 B	127 AB	108 B	19.6	255	14.5

Mean values represented by the different letters, for each extractant show significant differences (Tukey test,  $P < 0.05$ ).

The highest amount of K extracted with Mehlich-3 compared to the Mehlich-1 (Table 3) may be attributed to the composition and higher pH (pH 2.5) of Mehlich-3 solution. This solution contains ammonium acetate, formed from the addition of all reagents that comprise the method; thereby increasing the K levels extracted (MEHLICH, 1984). These results are similar to those reported by other authors (BORTOLON et al., 2010; HOSSEINPUR; SAMAVATI, 2008; ZBÍRAL; NEMEC, 2005; WANG et al., 2004).

Correlation coefficients for the amounts of K extracted by the different methods are shown in Figure 1.

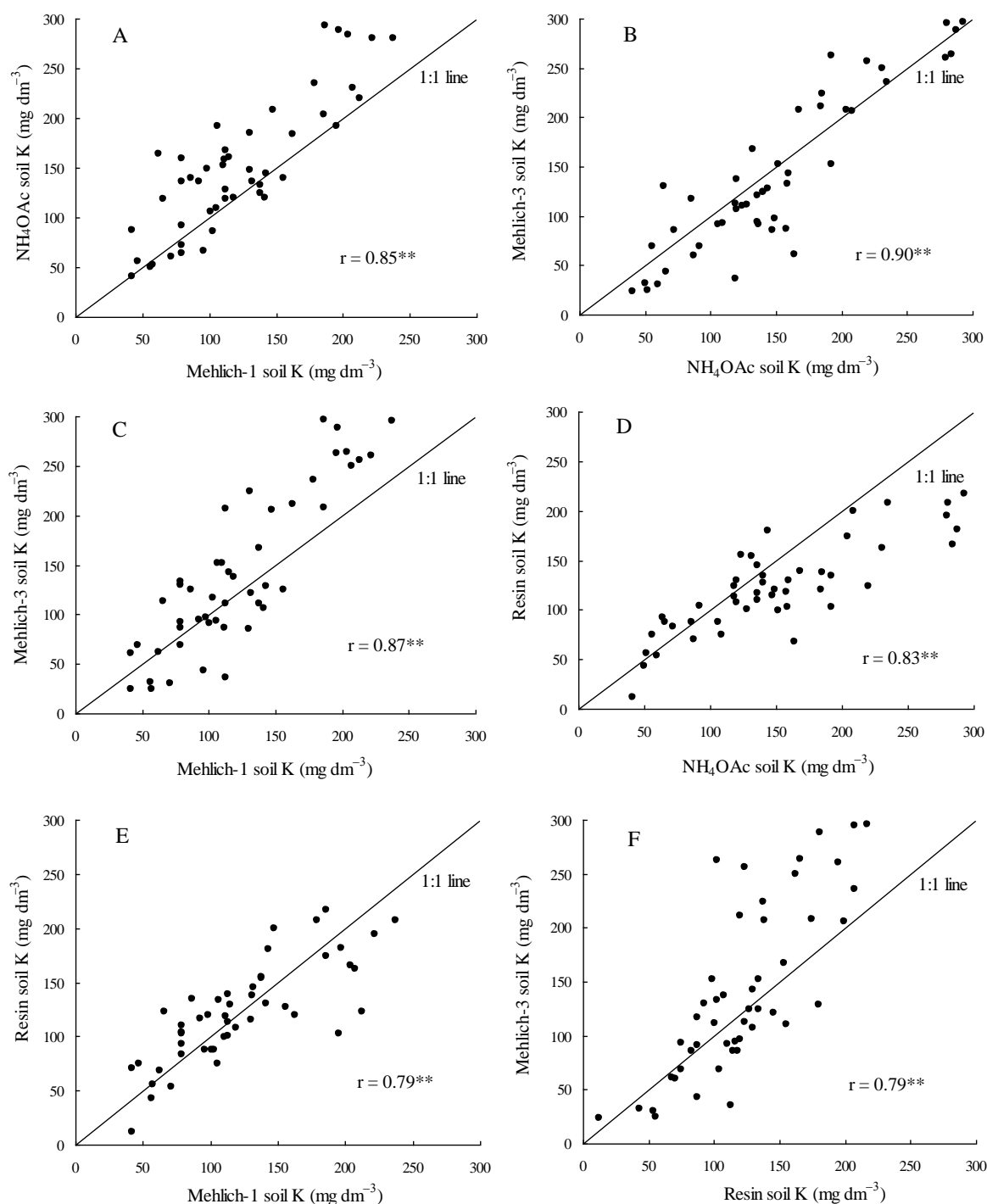


Figure 1. Correlation between extracted potassium from the soil by the different extractants tested. \*\*: statistical significance at 1% by F test. (n = 48).

Potassium extracted by Mehlich-1 (common method in Paraná State) was significantly correlated to the K extracted by 1 mol L<sup>-1</sup> NH<sub>4</sub>OAc ( $r = 0.85$ ), Mehlich-3 ( $r = 0.87$ ) and ion exchange resin ( $r = 0.79$ ) (Figure 1a, 1c and 1e). On the other hand, the correlation coefficients between the K amounts extracted with NH<sub>4</sub>OAc and Mehlich-3 and ion exchange resin were, respectively, 0.90 and 0.83 (Figures 1b and 1d), while the correlation between Mehlich-3 and ion exchange resin was 0.79 (Figure 1f). This high degree of association and significance among extractants indicates that all the studied methods used to evaluate the K availability to plants, although each extractant has its efficiency and accuracy.

Bortolon et al. (2009) also showed the efficiency of Mehlich-1, Mehlich-3 and resin extractants for determination of K availability in soils of Rio Grande do Sul, Brazil under no-tillage system. However, due to the greater convenience of operation in routine laboratories, mainly due to the ease of obtaining clear extracts by decantation, thus eliminating the filtration of soils suspensions, and the low cost of analysis (ROSSI; FAGUNDES, 1998), Mehlich-1 is more suitable for the evaluation of available K in soil.

The relationships between extracted K by four extractants and amount of K taken up by soybean plants are shown in Figure 2. Correlation coefficients between the K concentrations extracted from the soil and the K amount accumulated in the soybean plants were 0.90 ( $p < 0.01$ ) for 1 mol L<sup>-1</sup> NH<sub>4</sub>OAc (Figure 2a), 0.78 ( $p < 0.01$ ) for Mehlich-1 (Figure 2b), 0.87 ( $p < 0.01$ ) for Mehlich-3 (Figure 2c), and 0.89 ( $p < 0.01$ ) for resin (Figure 2d). The NH<sub>4</sub>OAc, Mehlich-3 and ion exchange resin extractants were the most efficient in estimating K availability to for soybean in different soils of Paraná State, whit wide variation in physical and chemical properties. Adequate to assess the availability of nutrients to plants method should be efficient for a broad range of soil types and crops. According to Alvarez Venegas (1996), an appropriate method to assess nutrient availability to plants should be effective for a broad range of soil types and different types of crops.

Mehlich-1 had the lowest correlation coefficients between the K concentration in the soil and that accumulated by the soybean plants, indicating that this method was the least effective in assessing K availability in soils of Paraná, Brazil. Martins et al. (2004) also observed the low efficiency of Mehlich-1 in assessing K availability for wheat in soils of Campos Gerais, Paraná, Brazil. These authors verified that 1 mol L<sup>-1</sup> NH<sub>4</sub>OAc and boiling 4 mol L<sup>-1</sup> HNO<sub>3</sub> extractants were good indices for K availability. Slaton et al. (2010) compared Mehlich-3 and 1 mol L<sup>-1</sup> HNO<sub>3</sub> and found that Mehlich-3-extractable K is an excellent predictor of soil K availability for soybean. However, in study performed with soil samples from the Rio Grande do Sul, Bortolon et al. (2010) found similar correlation coefficients between the K extracted with NH<sub>4</sub>OAc, Mehlich-1, Mehlich-3 and resin and the amount accumulated by maize plants (0.83 to 0.86) and soybean (0.84 to 0.94).

Hosseinpour and Zarenia (2012) showed that 0.1 mol L<sup>-1</sup> HNO<sub>3</sub>, Mehlich-1, and 0.01 mol L<sup>-1</sup> CaCl<sub>2</sub> extractants would be suitable as soil testing methods for determining available K for common bean in Iran soils. In general, the high efficiency of 1 mol L<sup>-1</sup> NH<sub>4</sub>OAc, Mehlich-1, Mehlich-3 and ion exchange resin extractants in determining the K availability for the majority of crops cultivated in Brazil is usually reported in the literature (Medeiros et al., 2010; Bortolon et al., 2009; Schlindwein and Gianello, 2005).

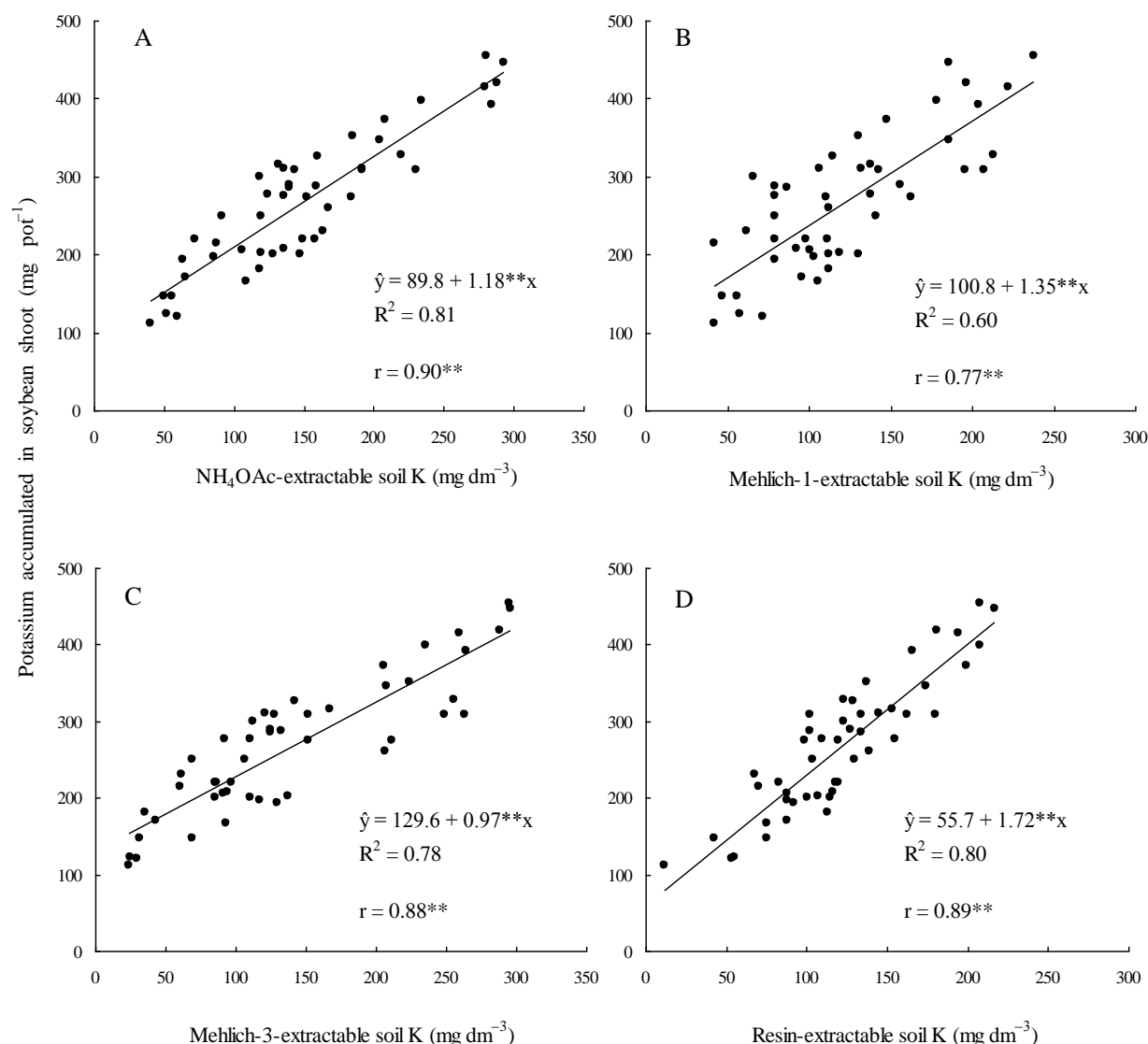


Figure 2. Relationship between potassium contents extracted from soil by ammonium acetate (A), Mehlich-1 (B) Mehlich-3 (C) and ion exchange resin (C) and the amount of potassium uptake by soybean plants. \*\*: statistical significance at 1% by F test. (n = 48).

## CONCLUSIONS

The efficacy of ammonium acetate, Mehlich-3 and ion exchange resin extractants in assessing potassium availability for soybean in soils from Paraná State was similar.

The Mehlich-1 extractant was less efficient in estimating the soybean-available K in the soils studied.

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