# Ion Exchange Resin Membrane Sensitivity Analysis of Selected Parameters for Soil Nutrient Extraction

Plinio L. Kroth<sup>1</sup>, Clesio Gianello<sup>1</sup>, Leandro Bortolon<sup>2</sup>, Jairo A. Schlindwein<sup>3</sup> & Elisandra S. O. Bortolon<sup>2</sup>

<sup>1</sup> Soil Science Department, Federal University of Rio Grande do Sul, Porto Alegre, Brazil

<sup>2</sup> Embrapa Pesca e Aquicultura, Palmas, Brazil

<sup>3</sup> Soil Science Department, Federal University of Rondônia, Porto Velho, Brazil

Correspondence: Leandro Bortolon, Embrapa Pesca e Aquicultura, Palmas, TO, Brazil. Tel: 55-3229-7825. E-mail: Leandro.bortolon@embrapa.br

Received: July 19, 2016	Accepted: August 25, 2016	Online Published: September 15, 2016
doi:10.5539/jas.v8n10p189	URL: http://dx.doi.org/10.	5539/jas.v8n10p189

The research is financed by CAPES, UFRGS Soil Testing Laboratory.

# Abstract

The ion exchange resin method has received considerable attention as an alternative soil test method to evaluate plant available nutrients. This study sought to investigate the effect of changes to the resin standard method in the capacity of the resin to extract soil P, K, Ca, and Mg in soils with different texture. We tested the following modifications: soil grinding levels (< 2 mm; < 0.3 mm), shaking time (8 h; 16 h; 24 h), reciprocation level (12.5 rpm; 25 rpm; 50 rpm), solution and elution saturation concentration (0.25/0.25 mol L<sup>-1</sup>; 0.5/0.5 mol L<sup>-1</sup>; 1.0/1.0 mol L<sup>-1</sup>), soil:solution ratio (1:5 v:v; 1:10 v:v; 1:16 v:v; 1:25 v:v), room temperature (10 °C, 15 °C, 25 °C, 40 °C), and resin amount (1 n; 2 n). When one factor was changed all the others were kept the same as the standard procedure. We selected the five most representative soil orders used for crop production in Southern Brazil which have a wide range of clay, organic matter, Mehlich-1 extractable P and K, and KCl exchangeable Ca, and Mg contents. Results showed that modifications on the standard extraction procedure affected the amounts of soil P, K, Ca, and Mg extracted. Temperature was the main factor affecting the amount of P extracted from the soil with ion exchange membrane resin. Our results can be useful to other regions that might be interested in adopting the resin soil test method, allowing others to identify the impacts of similar method modifications on soil nutrient availability according to soil type, soil management, and temperature conditions.

Keywords: phosphorus, soil testing, potassium, nutrient management, resin

# 1. Introduction

Ion exchange resin membranes (resin) have received considerable attention as an alternative method to extract soil nutrients (Amer et al., 1955; Takahashi, 2013). The majority of resin-based research evaluated soil phosphorus (P) availability to plants (Kovar et al., 2009). The high correlation between extracted resin soil P and plant P uptake (or crop yield), and due to its multi-element simultaneous extraction capacity, led some researchers to conclude that the resin methodology is adequate for soil multi-element extraction in a single extraction procedure (Raij et al., 1986; Schoenau & Huang, 1991; Qian et al., 1992; Bortolon & Gianello, 2008).

There are two broad methodologies used to evaluate soil nutrient availability with exchange resins. The first method involves two steps: (i) nutrient extraction with resin by shaking a soil-resin-solution suspension for a pre-determined period of time and; (ii) the recovery of the adsorbed ions eluted with a solution. The second method incubates resin membranes in situ with soils for a pre-determined amount of time, and then the nutrients are recovered from the resins (Skogley et al., 1990; Yang et al., 1991b; Cooperband & Logan, 1994; Dobermann et al., 1994; Kovar, 2003; Kovar et al., 2009; Schlindwein & Gianello, 2009). Independent of the procedure used; there is a wide range of variables that affect the efficacy of exchange resins in extracting nutrients from the soil. These variables, include but are not limited to, the type and amount of resin (Sibbesen, 1978; Vaidyanathan & Talibudeen, 1970), shaking time (Bache & Ireland, 1980; Qian et al., 1992), soil-solution ratio (Cooke & Hislop, 1963), concentration and counter-ions (Sibbesen, 1978), temperature (Yang et al., 1991a), and resin extraction capacity (Somarisi & Edwards, 1992).

The time consumed to perform a soil test is an important factor that should be taken in consideration when evaluating the suitability of a new method for use in a commercial laboratory. The resin method (Tedesco et al., 1995) used in many Brazilian laboratories requires a 16 h resin-soil-solution suspension shaking time. Schoenau and Huang (1991) compared different shaking times (1 h, 6 h, and 16 h) and found that P concentration increased with shaking time, but shaking time did not influence the interpretation of the soil P levels. Qian et al. (1992) showed that P extracted with 15 min of shaking was nearly 40% of the P amount extracted with a shaking time of 60 min. The authors concluded that the difference did not impact the interpretation of the P level relative to fertilizer recommendation. However, statistical differences do exist in the quantities of nutrients that are extracted from soils and are directly affected by shaking time, resin type, and soil type (Sibbesen, 1978; Bache & Ireland, 1980).

The amount of soil-P that can be extracted is dependent on the type and amount of resin used for extraction (Sibbesen, 1978). However, the major factor that affects the resin suitability in evaluating soil-P availability to plants is the resin's ionic saturation during the extraction period (Sibbesen, 1978). Bicarbonate is the most common counter-ion used due to its buffering effect during the extraction process, and it does increase soil P extractability.

Temperature affects soil nutrient availability to plants through ion activity, soil buffer capacity, and ion exchange and diffusion (Sparks, 2003). However, relatively few studies have looked at the effects of temperature on soil nutrient content extracted by resin (Yang et al., 1991b; Haagsma & Miller, 1963). Laboratory temperature effects during extraction are specific to each nutrient, and temperature must be kept constant during the extraction period and analysis (Yang et al., 1991b; Simonis, 1996).

Factors such as resin ion saturation, eluent volume, and shaking time are important and their effects on the content of nutrients extracted vary among nutrients (Somasiri & Edwards, 1992). Variations on soil grinding level also affects extracted nutrient amounts, and therefore, must be quantified for both resin and diluted-acid extractants (Takahashi, 1996). The influence of these factors on the resin extraction procedure must be investigated to improve soil analysis quality and to avoid potential mistakes on crop fertilizer recommendations, which could impact both farmer's profitability and the environment.

Since 1955 (Amer et al., 1955), the use of the resin method to evaluate soil-P has increased due to advances in technology, such as impregnating a resin onto a plastic membrane (Mallarino & Atia, 2005; Vandecar et al., 2010). There is a wide range of synthetic resins with different physical-chemical properties and reticulation degrees to keep the resin structure stable during the extraction process. Stability is important for resins made in membrane form, because some of their properties (physical-chemical) are modified during the polymerization process to keep the resin stable, which can affect the resin's capacity to extract nutrients from the soil.

The objective of this study was to determine the sensitivity of resin membrane extraction capacity during simultaneous extraction of P, K, Ca, and Mg, to the following modifications to the standard procedure used by soil testing laboratories in Southern Brazil: (i) soil particle size; (ii) shaking time; (iii) oscillations of soil:resin suspension; (iv) concentrations of both saturation and elution solution; (v) soil:solution ratio; (vi) room temperature during the extraction process and; (vii) quantity of resin. We also investigated the effect of laboratory temperature on phosphorus extracted with ion exchange resin membrane and the impact on nutrient recommendation to corn in Southern Brazil.

# 2. Method

The standard resin procedure adopted by soil testing laboratories in Southern Brazil at the time of this study, uses a 7.5 cm<sup>2</sup> ion resin exchange membrane strips (CRG1CZR42—strong cationic and AR103DQP 434—strong anionic; Ionics, Inc., Watertown, MA), separated by a nylon screen. Before using the strips, the resin strips are saturated with 0.5 mol L<sup>-1</sup> NaHCO<sub>3</sub> for a 24 h period and then washed with distilled water 2-fold with the same volume of sodium bicarbonate. For P, K, Ca, and Mg extraction, 2.5 cm<sup>3</sup> of soil, ground to pass a 2-mm sieve, 40 mL distilled water, and two exchange membrane strips are placed into a 50 mL glass snap-cap flask and shaken at 27 rpm for 16 h on an end-over-end shaker (Tecnal, Piracicaba). The resin strips are then removed, washed with a minimum amount of distilled water (about 50 mL) to remove excess of sodium bicarbonate from resin, and eluted with 40 mL of 0.5 mol L<sup>-1</sup> HCl. The flasks are allowed to stand for 30 min, and then shaken for 90 min on a reciprocating shaker at 110 oscillations per minute (rpm). Phosphorus in the resin strip extracts is determined colorimetrically (1-amino-2-naftol-4-sulfonic and ammonium molybdate method, Tedesco et al., 1995) using a FEMTO 600 spectrophotometer (FEMTO S.A., São Paulo, Brazil), K determined by flame photometry, and Ca and Mg determined by atomic spectrometry using a AAnalyst 200 Atomic Absorption Spectrometer (Perkin Elmer spectrometer, Waltham, MA).

We investigated the following modifications of the standard procedure: (i) soil grinding-particle size; (ii) shaking time; (iii) oscillations of soil:resin suspension; (iv) concentration of both saturation and elution solutions; (v) soil:solution (water) ratio; (vi) environmental (room) temperature during the extraction period and; (vii) quantity of resin used (Table 1). When one factor was changed all the others were kept the same as the standard procedure. Five most representative soil orders used for crop production in southern Brazil were selected, with a wide range of chemical, physical, and mineralogical properties (Table 2). Within each soil type, we had three soil-P levels (Very Low, Adequate, and Very High) and four replicates. The change in interpretation of available soil P, K, Ca, and Mg was determined for each factor tested. In addition, the extraction of P, K, Ca, and Mg using resin beads with temperature as the single variable were conducted according to the procedure described in Raij et al. (1987). The use of resin spheres by several laboratories across Brazil prompted this testing.

Table 1. Alterations applied to the standard ion exchange resin membrane procedure used for soil analysis in Southern Brazil

Factor	Standard <sup>1</sup>	Modifications
Soil particle size - grinding (mm)	< 2	< 0.3
Shaking time (h)	16	8; 24
Oscillations (rpm)	25	12.5; 50
Concentrations of saturation/elution solutions (mol L <sup>-1</sup> )	0.5/0.5	0.25/0.25; 1.0/1.0
Soil:solution ratio (v:v)	1:16	1:5; 1:10; 1:25
Laboratory temperature (°C)	25	10; 15; 40
Resin amounts (n)	1	2

*Note.*<sup>1</sup> Tedesco et al. (1995).

Soil Order	Pa	rticle s	ize	Organic Matter	pH in water	SMP Index		Soil a	vailable <sup>1</sup>	
Soli Oldel	Sand	Silt	Clay	Organic Matter	pii ili watei	Sivil muck	Р	K	Ca	Mg
			g kg <sup>-</sup>	·			mg	dm <sup>-3</sup>	cmol	$_{\rm c}  {\rm dm}^{-3}$
Oxisol	50	330	620	61	6.1	6.0	2.6	123	10.7	3.6
				60	6.3	6.1	14.7	98	12.1	3.7
				52	6.1	6.1	11.5	94	11.7	3.8
Inceptisol	110	410	480	81	6.5	6.2	1.6	101	17.1	4.5
				83	5.9	5.6	8.3	114	18.2	4.8
				78	6.6	6.2	18.3	134	17.9	4.5
Spodosol	470	210	320	21	6.8	6.6	9.1	46	3.5	1.8
				18	6.4	6.5	33.2	45	4.3	1.9
				22	6.4	6.6	58.2	46	4.4	1.7
Alfisol	650	90	260	21	6.1	6.4	5.5	42	4.0	1.2
				22	6.0	6.3	14.2	46	4.1	1.2
				18	6.0	6.3	63.0	44	3.6	1.2
Acrisol	930	30	40	6	6.3	6.9	3.1	38	1.3	0.4
				6	5.9	6.9	41.8	28	1.6	0.4
				5	6.1	6.8	72.8	29	1.4	0.4

Table 2. Orders and characterization of soils used to grow crops in Southern Brazil

*Note.*<sup>1</sup> Soil P and K extracted with Mehlich-1 solution and exchangeable Ca and Mg extracted with 1.0 mol L<sup>-1</sup> KCl.

The influence of laboratory temperature on P recommendation to crop was also investigated. We considered 25  $^{\circ}$ C as the standard laboratory temperature and compared its influence on soil-P, changes in soil-P status, P applied, and P balance. Soil P status was classified based on soil-P extracted with membrane resin according to Southern Brazil Manual for Crop Nutrient and Lime Recommendations (SBCS/NRS, 2004). Soil P classes according to soil-P (mg dm<sup>-3</sup>) extracted with membrane resin are: Very Low (VL = soil-P  $\leq$  5.0); Low (L = soil-P

range of 5.1-10.0); Medium (M = soil-P range of 10.1-20.0); High (H = soil-P range of 20.1-40.0); Very High (VH = soil-P  $\ge$  40.0). Soil-P critical level which is the soil-P level in soil that is adequate to crop production in Southern Brazil is 20.0 mg dm<sup>-3</sup>. We tested the P recommendation for corn (*Zea mays* L.) considering an expected yield of 10 Mg ha<sup>-1</sup>, and we verified the P amounts recommended according to soil-P extracted at each different laboratory temperature. We calculated P applied balance (%), that is the P amount applied based on soil-P extracted at 25 °C laboratory temperature (100%), compared to P amount applied base on soil-P extracted at 10 °C, 15 °C and 40 °C laboratory temperature.

The statistical analyses were performed comparing standard versus procedure modifications by ANOVA, and the mean differences in each factor were evaluated with Tukey test (0.05) using SAS (SAS, 2002).

## 3. Results

#### 3.1 Effect of Soil Grinding Size

Soil clay content affected the efficiency of resin P extraction and higher P amounts were found in high sand content soils compared to soils with high clay content (Table 3). The difference in higher soil P amounts extracted in sandy soils is probably due to the soil grinding processes and also because the sandy soils had more available P (Table 2). The finer screen (< 0.3 mm) passed the soil fractions with both high soil clay and organic matter to which P can be adsorbed, increasing the soil P amounts extracted (Bortolon et al., 2011). The finer constituents also increased surface sorption area. High clay content soils probably had greater amounts of Ca bonded with clays that are highly resistant to detachment, which increased the tortuosity of the path of P from the soil solution through resin exchange surface, reducing P in the solution (Bortolon et al., 2011). In clayey soils, the finer sieve decreased the sized particle content passing through the sieve, which may also have preferentially selected for Fe and Al oxides, increasing P adsorption, and consequently decreased the amount of P extracted. On the other hand, the high concentrations of bicarbonate ion, released from the resin during the extraction period, could increase the pH of the suspension and promote the precipitation of calcium phosphate (Raij et al., 1986), leading to a decrease in the P available to react with the resin. Contrasting results were found by Rolim et al. (2008), where the authors concluded that soil particle size did not affect amounts of soil-P extracted by resin in both fertilized and non-fertilized soils. However, the differences observed in our study can be explained by a 2 h shaking time at 150 rpm (Rolim et al., 2008) as compared to 16 h at 27 rpm on our procedure.

Soil particle size did affect the amount of Ca extracted with resin (Table 3). The greatest amounts were observed in soils with high clay content. Higher Ca amount extracted in clayey soils can be explained by the higher soil exchange capacity, which could compete with the resin on Ca adsorption. Calcium could also be precipitated as calcium phosphate. This process can also explain the reduction of extracted K due to particle size (Table 3). On average, extracted K amounts were different between grinding sizes and they did not affect the interpretation of K levels in the soil or the fertilizer recommendations to crops. The results showed that extraction of P with resin was affected by the soil grinding level, clay content, and amounts of P and Ca in soils. The obstruction of the resin surface by attached fine soil particles making some resin sites unavailable to adsorption may explain the effects on P and K; however, detailed studies must be carried out to validate this hypothesis.

				Soil grin	ding size (mm)				
Soil Order	< 2.0	< 0.3	< 2.0	< 0.3	< 2.0	< 0.3	< 2.0	< 0.3	
		Р		K		Ca	Mg		
		m	g dm <sup>-3</sup>			cmol	<sub>c</sub> dm <sup>-3</sup>		
Oxisol	4.9 a	3.9 a	58.7 a	59.5 a	5.22 a	3.74 a	2.11 a	1.83 a	
	31.5 a	27.9 a	41.5 a	37.7 a	6.47 a	5.16 a	2.37 a	2.13 a	
	60.3 a	55.9 a	50.8 a	48.0 a	4.67 a	4.11 a	2.27 a	2.26 a	
Inceptisol	6.3 a	6.5 a	40.9 a	38.9 a	10.09 a	8.88 a	3.03 a	3.14 a	
	18.1 a	20.6 a	39.8 a	37.1 a	4.49 a	4.91 a	2.68 a	3.10 a	
	58.8 a	57.5 a	39.0 a	37.1 a	9.99 a	10.08 a	2.82 a	3.17 a	
Spodosol	19.0 a	19.2 a	38.6 a	37.6 a	2.38 a	2.43 a	1.26 a	1.36 a	
	50.8 a	49.7 a	34.3 a	31.0 a	2.66 a	2.67 a	1.36 a	1.43 a	
	81.9 a	82.4 a	35.2 a	33.4 a	3.03 a	3.04 a	1.25 a	1.38 a	
Alfisol	8.2 a	9.8 a	32.8 a	32.9 a	2.09 a	2.56 a	0.84 a	1.11 a	
	20.0 b	24.5 a	29.1 a	31.0 a	1.86 a	2.42 a	0.75 a	0.99 a	
	73.0 b	87.0 a	29.1 a	29.2 a	2.27 a	2.61 a	0.80 a	1.05 a	
Acrisol	2.7 a	3.0 a	12.7 a	12.2 a	0.37 a	0.55 a	0.14 a	0.21 a	
	28.5 b	36.3 a	15.0 a	14.0 a	0.53 a	0.54 a	0.12 a	0.19 a	
	62.3 b	81.1 a	16.2 a	16.4 a	0.51 a	0.73 a	0.14 a	0.23 a	
Average	35.1 b	37.7 a	34.2 a	33.1 b	3.63 a	3.77 a	1.48 a	1.57 a	

Table 3. Phosphorus, potassium, calcium and magnesium extracted with ion exchange resin membrane affected by soil grinding size

*Note.* Means followed with the same lower case letter within a row did not differ statistically by Tukey test (5%); C.V. (%) P = 4.1; C.V. (%) K = 8.4; C.V. (%) Ca = 15.3; C.V. (%) Mg = 4.9.

#### 3.2 Shaking Time

Shaking time is an important issue during the development of a soil test method. The goal during the shaking process is to increase the contact of soil particles and the soil extractant. As shaking time increased, there was an increase in extracted soil P (Table 4), which was greater in clayey than in sandy soils, probably due to the high P adsorption capacity associated with most clay soils. Ascertaining soil-P using the resin method is a lengthier process compared to Mehlich-1 or Mehlich-3, due to the long shaking time involved, which is independent of resin type (Sibbesen, 1978). Qian et al. (1992) showed that although longer shaking times extracted more soil P, shaking time did not affect the interpretation of soil P levels and P fertilizer recommendations. Longer shaking time can extract insoluble soil P forms that have no bearing on plant P availability (Schoenau & Huang, 1991) and may lead to a lower recommendation for crops which could reflect in lower yields. The increase of shaking time affected Ca extraction, and more Ca was extracted from soil with longer shaking time (Table 4), especially on those soils with higher Ca content (Table 2). For both P and K, the greatest extracted amounts were found with the 24 h shaking time. As the shaking time increased, the amount of Ca and Mg extracted increased and K decreased (Table 4). This finding can be explained by different charges among the two cations in the solution even though in similar concentration; resin affinity favors high valence cations (Sparks, 2003). The results showed that 16 h of shaking time is not sufficient to extract soil exchangeable Ca and Mg, and 24 h shaking extracted more Ca and Mg than 16 h shaking. In addition, soil-P was greater with 24 h shaking in some cases 2-fold compared to 8 h shaking.

	Shaking time (h)												
Soil Order	8	16	24	8	16	24	8	16	24	8	16	24	
		Р			Κ			Ca		. <u> </u>	Mg		
			mg o	1m <sup>-3</sup>					cmol <sub>c</sub> d	lm <sup>-3</sup>			
Oxisol	2.9 b	4.9 ab	6.5 a	67.0 a	58.7 b	51.4c	2.00 b	5.22 a	5.38 a	1.23 b	2.11 a	2.42 a	
	23.0 c	31.5 b	50.4 a	48.1 a	41.5 b	35.1 c	2.97 b	6.47 a	7.04 a	1.56 b	2.88 a	3.36 a	
	43.1 c	60.3 b	88.0 a	56.0 a	50.8 b	44.8 c	2.39 c	4.67 b	6.37 a	1.63 c	2.27 b	3.05 a	
Inceptisol	4.4 b	6.3 b	9.7 a	45.1 a	40.9 ab	36.9 b	5.33 c	10.09 b	12.31 a	2.28 c	3.03 b	3.78 a	
	11.9 c	18.1 b	27.9 a	39.0 a	39.8 a	32.7 b	2.22 c	4.49 b	6.46 a	1.94 c	2.68 b	3.64 a	
	38.9 c	58.8 b	85.0 a	42.0 a	39.0 ab	35.7 b	5.78 c	9.99 b	13.01 a	2.24 b	2.82 b	3.61 a	
Spodosol	15.9 b	19.0 b	25.2 a	33.5 a	38.6 a	35.7 a	1.24 b	2.38 a	2.86 a	0.99 b	1.26 ab	1.69 a	
	41.2 c	50.8 b	64.4 a	29.2 a	34.3 a	32.1 a	1.28 b	2.66 a	3.34 a	1.04 b	1.36 ab	1.85 a	
	68.1 c	81.9 b	103.6 a	28.0 b	35.2 a	33.9 a	1.54 b	3.03 a	3.45 a	0.97 b	1.25 ab	1.63 a	
Alfisol	7.7 a	8.2 a	10.0 a	23.8 b	32.8 a	30.8 a	0.98 b	2.09 a	2.66 a	0.63 a	0.84 a	1.16 a	
	18.3 b	20.0 b	26.3 a	22.5 b	29.1 a	29.0 a	1.13 b	1.86 b	2.69 a	0.62 a	0.75 a	1.07 a	
	61.4 c	73.0 b	97.2 a	22.5 b	29.1 a	29.6 a	1.41 c	2.27 b	3.38 a	0.66 a	0.80 a	1.13 a	
Acrisol	2.9 a	2.7 a	3.2 a	7.9 a	12.7 a	12.7 a	0.15 a	0.37 a	0.47 a	0.13 a	0.54a	0.21a	
	24.9 c	28.5 b	35.8 a	7.3 b	15.0 a	13.9 a	0.10 a	0.53 a	0.56 a	0.08 a	0.12 a	0.13 a	
	56.3 c	62.3 b	90.3 a	9.1 b	16.2 a	17.5 a	0.13 a	0.51 a	0.55 a	0.13 a	0.14 a	0.20 a	
Average	28.1 c	35.1 b	48.2 a	32.1 b	34.2 a	31.4 b	1.91 c	3.77b	4.68 a	1.08 c	1.55 b	1.90 a	

Table 4. Phosphorus, potassium, calcium, and magnesium extracted with ion exchange resin membrane affected by shaking time

Means followed with the same lower case letter within a row did not differ statistically by Tukey test (5%); C.V. (%) P = 4.6; C.V. (%) K = 8.2; C.V. (%) Ca = 12.0; C.V. (%) Mg = 21.5.

#### 3.3 Oscillations of Soil: Resin Suspension

The shaking process accelerates nutrient extraction that promotes a fast and constant contact between soil solid phase and resin, in addition to the initial nutrient in the soil solution. The application of different oscillation rates based on the end-over-end shaker were not different to soil-P compared to 25 and 50 rpm, and in almost all soils orders soil-P concentrations extracted with 12.5 rpm were similar to those extracted at 50 rpm (Table 5). However, the differences due to this factor showed little effect among soil orders and were related to the increased contact time between soil P and resin. We verified from the data shown in Table 5 that it was not necessary to have vigorous oscillations for extracting soil Ca and Mg with resin. However, the lowest oscillation rate (12.5 rpm) did reduce the extraction of Ca and Mg and it would affect the calculation of soil CEC that is used to classify the K status in soils from Southern Brazil. Increasing the number of oscillations per minute reduced extractable K concentrations (Table 5) in four soils following polyvalence cations selectivity, which increased Ca and Mg amounts extracted and reduces K amounts extracted (Sparks, 2003). The results show that oscillation rate affects extractable nutrient concentrations. Also, the differences found for P and K had little or no effect on the nutrient recommendations to crops (SBCS/NRS, 2004).

		Oscillation (rpm)												
Soil Order	12.5	25	50	12.5	25	50	12.5	25	50	12.5	25	50		
		Р			Κ			Ca			Mg			
			mg	dm <sup>-3</sup>					cmol <sub>c</sub>	dm <sup>-3</sup>				
Oxisol	3.7 a	4.9 a	3.5 a	60.5 a	59.3 a	60.0 a	3.64 b		4.51 ab	1.42 b	2.11 a	2.12 a		
	31.3 a	31.5 a	32.7 a	42.3 a	44.2 a	37.9 b	4.91 b	6.47 a	5.93 a	1.68 b	2.37 a	2.52 a		
	56.9 b	60.3 ab	63.0 a	53.3 a	53.4 a	48.6 b	4.52 a	4.67 a	4.96 a	1.81 b	2.27 a	2.58 a		
Inceptisol	6.1 a	6.3 a	5.6 a	41.6 b	47.2 a	42.3 ab	8.77 b	10.09 a	9.82 a	2.33 b	3.03 a	3.26 a		
	18.1 a	18.1 a	16.3 a	40.7 ab	43.1 a	37.3 b	4.46 a	4.49 a	4.47 a	2.19b	2.68 a	2.86 a		
	54.7 b	58.8 a	56.7 ab	42.3 ab	49.2 a	36.6 b	9.55 b	9.99 ab	10.49 a	2.24 b	2.82 a	3.13 a		
Spodosol	18.1 a	19.0 a	19.1 a	39.7 a	31.5 b	34.7 b	1.81 a	2.38 a	2.62 a	0.91 b	1.26 ab	1.45 a		
	45.5 b	50.8 a	47.5 ab	34.8 a	30.6 b	31.6 b	2.14 a	2.66 a	2.95 a	0.98 b	1.36 a	1.54 a		
	80.2 b	81.9 ab	84.6 a	34.5 a	34.8 a	33.5 a	2.85 a	3.03 a	3.13 a	1.00 b	1.25 ab	1.40 a		
Alfisol	8.6 a	8.2 a	8.7 a	33.8 a	33.8 a	29.7 a	1.72 a	2.09 a	2.34 a	0.67 a	0.84 a	1.01 a		
	20.3 a	20.0 a	22.4 a	31.9 a	30.6 a	29.0 a	1.98 a	1.86 a	2.28 a	0.60 a	0.75 a	0.90 a		
	74.0 b	73.0 b	79.3 a	29.9 a	28.7 a	27.8 a	2.26 a	2.27 a	2.73 a	0.65 a	0.80 a	0.99 a		
Acrisol	3.1 a	2.9 a	2.7 a	15.7 a	13.5 ab	10.8 b	0.21 a	0.37 a	0.57 a	0.13 b	0.14 a	0.23 ab		
	26.6 a	28.5 a	29.4 a	17.6 a	12.5 b	15.1 ab	0.26 a	0.53 a	0.50 a	0.10 a	0.12 a	0.17 a		
	66.2 b	62.3 c	69.9 a	17.6 a	12.9 b	15.8 ab	0.29 a	0.51 a	0.66 a	0.12 a	0.14 a	0.19 a		
Average	34.2 b	35.1 ab	36.2 a	35.8 a	34.3 ab	32.7 b	3.29 b	3.77 a	3.86 a	1.12 c	1.46 b	1.62 a		

Table 5. Phosphorus, potassium, calcium, and magnesium extracted with ion exchange resin membrane affected by oscillation of soil:solution:resin

*Note.* Means followed with the same lower case letter within a row did not differ statistically by Tukey test (5%); C.V. (%) P = 5.0; C.V. (%) Ca = 13.2; C.V. (%) Mg = 13.2; C.V. (%) K = 9.3.

#### 3.4 Concentration of Saturation and Elution Solutions

Several solutions have been studied for the saturation of the resin and eluting the adsorbed ions after soil extraction. For saturation of anion exchange resin, the bicarbonate anion has been preferred due the buffering effect, which increases soil extraction of P (Sibbesen, 1978; Raij et al., 1986), while Na is preferable for saturation of cation exchange resins. There was significant interaction between concentration of extraction solution and soil-P concentration on sandy soils with high soil-P, where the most concentrated solution extracted greater amounts of soil-P (Table 6). There were differences in only for Alfisol and Acrisol soils (both with very high soil-P content) where the standard method (0.5 mol L<sup>-1</sup> solution) extracted the lowest amount of P. Extraction of these soils does not follow the same behavior as the solution concentration. This behavior is probably due to similar mineralogical properties on both Alfisol and Acrisol. Since this finding was only observed in soils with high P concentrations, the use of a more concentrated solution would not impact fertilizer recommendations to crops, since soil-P level is considered adequate to crops and the fertilizer recommendation on that case is based only on the crop P removal (SBCS/NRS, 2004).

The results on Table 6, for soil Ca, Mg, and K extracted by ion-exchange resin membrane saturated with different concentrations of sodium bicarbonate, show that in soils with greater Ca content, the amount of Ca extracted decreased as extraction solution concentration increased. On average, the extracted concentrations of soil K and Mg were higher with increasing concentration of both saturation and elution solutions, regardless of the element content in soil, and were statistically different for both elements in the three concentrations. Although the results for the four elements (P, K, Ca, and Mg) in soil by using different solution concentrations showed statistical differences, the use of the most concentrated solution (1.0 mol  $L^{-1}$ ) would double the amount of chemicals required for the analyzes compared to the standard method. In addition, the 1.0 mol  $L^{-1}$  concentration would also have a negative effect on laboratory equipment and waste management.

## 3.5 Soil:Solution Ratio

There was significant interaction between soil:solution ratio and soil-P (Table 7). On average, the extracted soil-P concentrations were greatest for 1:25 soil:solution ratio. Similar results were obtained by McLaughlin et al. (1993). At a soil:solution ratio of 1:16, there was a decrease in soil-P concentrations. Whereas in other

relationships studied such as soil grinding and shaking time, there was little difference in the results. For the 1:10 and 1:25 soil solutions ratio the effect, soil-P extraction although statistically significant different for some soils, is not pronounced enough in terms of soil-P value to affect the results if the soil:solution remains close to that established for the method.

Table 6. Phosphorus, potassium,	, calcium, and magnesium	extracted with ion exchange	ge resin membrane affected
by solution concentration			

					Solut	ion Conce	entration (m	nol L <sup>-1</sup> )				
Soil	0.25	0.5	1.0	0.25	0.5	1.0	0.25	0.5	1.0	0.25	0.5	1.0
		Р			Κ			Ca			Mg	
			mg o	dm <sup>-3</sup>					cmol	<sub>c</sub> dm <sup>-3</sup>		
Oxisol	4.1 a	4.9 a	3.7 a	55.8 a	59.3 a	49.4 b	5.20 a	5.22 a	4.24 a	1.68 c	1.82 b	1.92 a
	33.6 ab	31.5 b	35.5 a	39.4 a	44.2 a	40.2 a	6.08 a	6.47 a	5.60 a	2.05 c	2.26 b	2.41 a
	61.3 a	60.3 a	63.5 a	48.7 b	53.4 a	53.0 a	5.85 a	4.67 b	5.09 ab	2.19 b	2.05 b	2.60 a
Inceptisol	5.6 a	6.3 a	6.0 a	40.3 b	47.2 a	47.4 a	10.64 a	10.09 a	8.82 b	2.83 b	2.96 b	3.12 a
	17.8 a	18.1 a	18.1 a	38.7 b	43.1 a	42.8 a	4.73 a	4.49 a	4.88 a	2.52 b	2.69 b	3.07 a
	56.8 a	58.8 a	57.2 a	37.6 c	49.2 a	40.8 b	12.02 a	9.99 b	9.46 b	2.87 b	2.89 b	3.02 a
Spodosol	18.5 a	19.0 a	19.4 a	35.2 b	31.5 b	42.8 a	2.81 a	2.38 a	2.49 a	1.11 b	1.40 a	1.45 a
	50.4 a	50.8 a	49.8 a	31.1 b	30.6 b	36.4 a	2.57 a	2.66 a	2.68 a	1.19b	1.51 a	1.53 a
	83.2 a	81.9 a	84.3 a	32.3 b	34.8 a	36.4 a	3.58 a	3.03 a	3.16 a	1.14 b	1.48 a	1.44 a
Alfisol	7.5 a	8.2 a	8.1 a	28.8 b	33.8 a	31.9 a	2.03 a	2.09 a	2.23 a	0.74 b	0.97 a	0.98 a
	20.7 a	20.0 a	21.9 a	27.0 b	30.6 a	32.5 a	2.26 a	1.86 a	2.23 a	0.67 b	0.91 a	0.93 a
	76.1 ab	73.0 b	78.4 a	27.6 a	28.7 a	31.3 a	1.89 a	2.27 a	2.49 a	0.69 b	0.98 a	0.96 a
Acrisol	2.9 a	2.7 a	3.1 a	14.1 b	13.5 b	17.2 a	0.41 a	0.37 a	0.49 a	0.14 a	0.18 a	0.18 a
	29.1 a	28.5 a	30.6 a	14.1 b	12.5 b	20.1 a	0.44 a	0.53 a	0.41 a	0.12 a	0.14 a	0.14 a
	70.0 a	62.3 b	71.0 a	17.0 b	12.9 c	19.2 a	0.47 a	0.51 a	0.57 a	0.15 a	0.16 a	0.14 a
Average	35.8 ab	35.1 b	36.7 a	32.5 c	34.3 b	36.4 a	4.06 a	3.77 ab	3.65 b	1.34 c	1.49 b	1.59 a

*Note.* Means followed with the same lower case letter within a row did not differ statistically by Tukey test (5%); C.V. (%) P=4.8; C.V. (%) Ca = 15.5; C.V. (%) Mg = 12.5; C.V. (%) K = 8.2.

								Soil:solut	ion ratio							
Soil	1:5	1:10	1:16	1:25	1:5	1:10	1:16	1:25	1:5	1:10	1:16	1:25	1:5	1:10	1:16	1:25
		F	)			ŀ	ζ			C	Ca			М	[g	
				mg	dm <sup>-3</sup>							cm	$ol_c dm^{-3}$			
Oxisol	5.9a	5.4a	4.9a	7.5a	35.8d	48.6c	58.8b	89.4a	9.13a	9.76a	3.81b	3.58b	3.71a	2.65b	1.82b	2.13b
	42.7a	42.8a	31.5b	44.5a	29.0c	32.8be	41.5b	65.0a	10.7a	8.22a	4.85b	5.18b	3.82a	3.18b	2.26b	2.62b
	72.8b	73.8b	60.3c	77.9a	29.6d	41.3c	50.8b	77.2a	9.76a	9.13a	4.47b	4.26b	4.18a	3.35b	2.05b	2.74b
Inceptisol	8.2a	7.7a	6.3a	8.5a	29.6c	34.6be	40.9b	66.la	13.8a	12.3a	8.97b	9.92b	3.89a	3.77a	2.96b	3.74a
	22.9a	23.2a	18.1b	26.7a	29.6c	32.2be	39.8b	61.0a	9.19a	9.97a	4.47b	4.16b	4.76a	4.04ab	2.69b	3.32b
	67.0b	70.5b	58.8c	76.4a	24.1c	32.8be	39.0b	64.la	14.0a	13.5a	9.75b	10.4b	3.56a	3.73a	2.89b	3.61a
Spodosol	21.5a	21.1a	19.0a	22.3a	34.0b	35.2b	38.6b	57.9a	3.86a	3.41a	2.16b	1.79b	2.25a	1.86a	1.40b	1.50b
	55.0a	56.3a	50.8b	55.6a	25.9b	30.9b	34.3b	51.8a	4.36a	3.80a	2.57b	2.41b	2.37a	2.06a	1.51b	1.61b
	91.3a	92.5a	81.9b	90.6a	27.2b	29.8b	35.2b	50.8a	4.82a	4.28a	2.99b	2.51b	2.13a	1.87ab	1.48b	1.45b
Alfisol	9.la	8.9a	8.2a	10.0a	25.3b	28.6b	32.8b	44.7a	3.06a	3.20a	1.63b	1.95b	1.47a	1.28ab	0.97b	0.99b
	23.3a	22.5a	20.0a	23.9a	25.9b	26.7b	29.1b	42.7a	3.58a	3.09a	2.07b	1.94b	1.42a	1.20ab	0.91b	0.94b
	86.5a	84.5a	73.0b	82.9a	24.8b	27.3b	29.1b	44.7a	3.69a	3.56a	2.27b	2.22b	1.41a	1.24a	0.98a	0.96b
Acrisol	3.8a	3.6a	2.7a	4.7a	12.4b	9.8b	12.7b	22.4a	0.24a	0.39a	0.38a	0.33b	0.23a	0.21a	0.18a	0.22a
	32.4ab	32.8a	28.5b	34.la	12.4b	12.8b	15.0ab	22.4a	0.04b	0.54a	0.36b	0.44a	0.13a	0.16a	0.14a	0.18a
	74.8a	75.9a	62.3b	77.la	15.4a	15.8a	16.3a	19.8a	0.42b	0.75a	0.38b	0.55a	0.18a	0.20a	0.16a	0.21a
Average	41.1b	41.4b	35.1c	42.8a	25.4d	29.3c	34.3b	52.0a	6.04a	6.04a	3.77b	3.46b	2.78a	2.08ab	1.49b	l.75b

Table 7. Phosphorus,	potassium, o	calcium, a	ind magnesium	extracted	with ion of	exchange re	esin memb	rane affe	cted
by soil:solution ratio									

*Note.* Means followed with the same lower case letter within a row did not differ statistically by Tukey test (5%); C.V. (%) P = 4.8; C.V. (%) K = 12.2; C.V. (%) Ca = 5.6; C.V. (%) Mg = 6.

We observed an interaction between soil dilution and the K content in the soil (Table 7), where increasing soil:solution ratio increased the amount of soil K extracted compared to the standard method. The soil dilution effect was less pronounced in soils with low K amounts; however in soils with higher K content, there was a significant increase on the amount of soil-K extracted due to soil dilution increase. For Ca and Mg, the soil:solution ratio affected the amounts extracted in all soils, regardless the element content in the soil (Table 7). The amounts of soil Ca and Mg extracted decreased as soil dilution increased. The results (Table 7) showed that K had an opposite behavior to Ca and Mg; thereby, when the extraction of Ca and Mg decreased, the extraction of K increased, as previously observed in other studies. These results differ from Sparks (2003), who reported that higher solution dilution favors the exchange of cations of higher valence, namely Ca and Mg in this study compared to K.

#### 3.6 Laboratory Temperature

As the temperature and soil-P increased, the amounts of P extracted increased significantly (Table 8). Extractable soil-K was not significantly affected by laboratory or room temperature, likely due to the low levels of K in most of the soils used in this study. Kinetic energy of the substances increases with temperature, and therefore the ion activity in the solution, as well as the buffering capacity and CEC. These factors are more important for the extraction of K, Ca, and Mg than for P. The greater extraction of P with increasing temperature is probably due to increased solubility of low solubility compounds. More importantly, the temperature increase also expands the resin matrix structure. The resin expansion exposes additional sites for sorption of ions present in the solution. In addition, increasing temperature increases ion activity with adsorbed ions of the solution, thereby, increases the concentration of elements in soil solution. Yang et al. (1991b) observed that the resin extraction of elements from the soil can affect nutrient extraction positively or negatively due to temperature range and is specific for each nutrient and soil type.

							Envi	ronmental	tempera	ature (°C	5)					
Soil	10	15	25	40	10	15	25	40	10	15	25	40	10	15	25	40
			Р			K	<u>c</u>			(	Ca			Ν	Лg	
				mg c	lm <sup>-3</sup>							cmol <sub>c</sub>	dm <sup>-3</sup>			
Oxisol	3.0 a	4.5 a	4.9 a	7.2 a	59.1 a	63.0 a	59.3 a	58.5 a	3.93 b	4.42 b	5.22 a	5.70 a	1.85 b	2.19 ab	2.11 b	2.47 a
	15.5 d	24.3 c	31.5 b	59.4 a	42.6 a	43.5 a	44.2 a	42.2 a	4.68 c	6.02 b	6.47 b	7.44 a	2.06 c	2.45 b	2.37 be	2.95 a
	32.2 d	46.6 c	60.3 b	103.8 a	51.5 b	52.2 b	53.4 b	57.9 a	3.29 c	3.45 c	4.67 b	6.36 a	1.90 c	2.21 be	2.27 b	3.18 a
Inceptisol	3.0b	6.2 ab	6.3 ab	9.3 a	44.5 a	43.5 a	47.2 a	39.8 b	5.77 c	9.80b	10.09 b	12.30 a	2.35 c	3.57 a	3.03 ab	3.88 a
	8.7 c	14.1 b	18.1b	32.1 a	38.1 b	43.5 a	43.1 a	38.0 b	2.89 c	3.43 c	4.49 b	6.94 a	2.12 c	2.63 b	2.68 b	3.93 a
	29.7 d	45.0 c	58.8 b	95.5 a	40.7 b	40.9 b	49.2 a	38.6 b	6.43 d	9.99 c	11.01 b	13.28 a	2.39 c	3.52 a	2.82 b	3.65 a
Spodosol	12.1 c	17.7 b	19.0 b	27.8 a	38.8 b	40.2 a	31.5 c	42.8 a	1.68 b	2.11 b	2.38 ab	2.92 a	1.10b	1.36 ab	1.26 b	1.68 a
	33.5 d	42.9 c	50.8 b	70.7 a	31.8 b	35.4 a	30.6 b	37.4 a	1.99 b	2.20b	2.66 ab	3.43 a	1.18b	1.31 b	1.36 b	1.82a
	52.8 d	71.0 c	81.9b	118.9 a	31.8 b	39.5 a	34.8 b	38.0 a	2.07 c	2.45 bc	3.03 ab	3.75 a	1.06 b	1.26 b	1.25 b	1.69 a
Alfisol	6.1b	7.5 ab	8.2 ab	11.8 a	29.9 b	36.8 a	33.8 a	35.6 a	1.64 b	1.60 b	2.09 ab	2.73 a	0.72 b	0.84 ab	0.84 ab	1.13 a
	11.3 c	20.0 b	20.9 b	28.6 a	26.7 b	33.4 a	30.6 a	32.6 a	1.52 b	1.60 b	1.86 ab	2.57 a	0.58 b	0.76 ab	0.75 ab	1.08 a
	43.2 d	61.4 c	73.0b	100.1 a	27.3 b	32.8 b	28.7 b	43.4 a	1.92 b	1.69 b	2.27 ab	2.88 a	0.68 b	0.74 ab	0.80 ab	1.04 a
Acrisol	1.9 a	2.6 a	2.7 a	4.1 a	13.3 b	15.6 ab	13.5 b	18.1 a	0.59 a	0.35 a	0.37 a	0.49 a	0.19b	0.16b	0.54 a	0.20 ab
	17.5 c	26.6 b	28.5 b	40.9 a	13.3 b	16.1 a	12.5 b	18.1 a	0.65 a	0.21 a	0.53 a	0.41 a	0.14 a	0.09 a	0.12 a	0.16 a
	39.0 d	54.5 c	62.3 b	92.1a	13.3 b	18.8 a	12.9 b	20.6 a	0.48 a	0.21 a	0.51 a	0.66 a	0.17 a	0.13 a	0.14 a	0.17 a
Average	20.6 d	29.7 c	35.1 b	53.5 a	34.4 b	37.0 a	34.3 b	37.3 a	2.64 c	3.37 c	3.77 b	4.79 a	1.23 c	1.55 b	1.49b	1.93 a

Table 8. Phosphorus, potassium, calcium, and magnesium extracted with ion exchange resin membrane affected by laboratory temperature

*Note.* Means followed with the same lower case letter within a row did not differ statistically by Tukey test (5%); C.V. (%) P = 7.1; C.V. (%) K = 10.6; C.V. (%) Ca = 10.3; C.V. (%) Mg = 10.5.

Due to the numerous changes occurring in soil as a result of temperature, the process of extraction with resin is always affected, regardless of the type of resin used in the extraction procedure. Data (Table 9) were obtained by the spherical resin method proposed by Raij et al. (1986). Just as in the extraction with resin membrane, the amounts extracted increased with increasing temperature. The same was observed for the extraction of K, particularly with higher levels in the soil. The extracted Ca and Mg concentrations were unaffected by higher temperatures (Table 9). These results are similar to those obtained with the resin membrane (Table 8). The results show that the extraction technique with resin is influenced by temperature and must be controlled throughout the extraction process. This finding is therefore a negative aspect of the use of resin in the determination of nutrient availability as a standard laboratory practice, especially in regions with a wide range temperature throughout the year, requiring a more rigorous control of this effect to achieve and maintain analytical reproducibility.

	Environmental temperature (°C)												
Soil	15	25	15	25	15	25	15	25					
		Р		К		Ca	]	Mg					
		mg	dm <sup>-3</sup>			cmol <sub>c</sub>	dm <sup>-3</sup>						
Oxisol	12.8 b	17.0 a	42.9 b	74.1 a	2.43 a	2.38 a	0.85 a	0.84 a					
	53.0 b	68.7 a	35.1 b	54.6 a	2.32 a	2.31 a	0.91 a	0.98 a					
	66.1 b	91.1 a	109.2 b	187.2 a	2.42 a	2.39 a	0.98 a	0.93 a					
Inceptisol	16.0 a	18.9 a	35.1 b	58.5 a	2.33 a	2.29 a	0.99 a	0.99 a					
	33.7 b	43.1 a	136.5 b	198.9 a	2.45 a	2.48 a	0.75 a	0.77 a					
	94.2 b	105.6 a	42.9 b	50.7 a	2.32 a	2.39 a	0.98 a	0.99 a					
Spodosol	22.7 b	28.7 a	31.2 a	35.1 a	2.31 a	2.18 a	0.91 a	0.85 a					
	54.0 b	69.2 a	31.2 b	39.0 a	2.45 a	2.38 a	0.99 a	0.96 a					
	84.7 b	133.4 a	31.2 a	31.2 a	2.35 a	2.29 a	0.85 a	0.88 a					
Alfisol	7.4 b	12.0 a	35.1 a	27.3 b	2.45 a	2.38 a	0.99 a	0.97 a					
	21.0 b	28.5 a	15.6 b	23.4 a	2.32 a	2.33 a	0.89 a	0.85 a					
	68.1 b	97.7 a	15.6 b	23.4 a	2.15 a	2.25 a	0.93 a	0.93 a					
Acrisol	2.4 a	4.6 a	3.9 a	3.9 a	2.43 a	2.33 a	0.94 a	0.91 a					
	27.8 b	37.9 a	3.9 a	3.9 a	2.41 a	2.29 a	0.98 a	0.95 a					
	58.3 b	77.1 a	3.9 b	11.7 a	2.18 a	2.23 a	0.85 a	0.88 a					
Average	41.6 b	55.6 a	38.3 b	54.6 a	2.36 a	2.33 a	0.92 a	0.91 a					

Table 9. Phosphorus, potassium, calcium, and magnesium extracted with ion exchange resin in sphere affected by environmental temperature

*Note.* Means followed with the same lower case letter within a row did not differ statistically by Tukey test (5%); C.V. (%) P = 4.1; C.V. (%) K = 8.4; C.V. (%) Ca = 15.3; C.V. (%) Mg = 4.9.

#### 3.7 Resin Amounts

There was interaction between the resin amounts and soil P concentration (Table 10). The use of two resin membranes extracted greater amounts of P from all soils. However, the effect was more pronounced in soils with high contents of both clay and P. This effect was also found by Sagger et al. (1990) who obtained 25-40% more P extracted by using two resin membranes. Increasing the amount of resin, besides increasing the exchange capacity, increases the contact surface between the resin and the ions in solution and therefore the activity of bicarbonate ions in the solution. According to Sibbesen (1978), the increase of bicarbonate ions creates more competition with phosphate ions for exchange sites, releasing the P.

The content of extracted Ca and Mg were affected by the amount of resin (Table 10). There was a significant difference in the amounts extracted in the soils with higher contents of these elements. The amount of soil-K extracted increased in all soil when two resin membranes were used, regardless the soil-K content. The increase of resin amount can be more important for extraction of all cations, whereas in the study of some factors we observed competition between K, Ca, and Mg for the resin.

# 3.8 Impact on Crop Nutrient Recommendations

Soil testing quality plays an important role on results that directly impact crop nutrient recommendations. Temperature in the laboratory is one important factor to be controlled during the soil testing routine. We observed that at 40 °C laboratory temperature the P recommendations to corn were underestimated in soils with very low and very high P (Table 11). In some cases, the P applied was reduced by 33% and the impact on farmer's profit could be great especially due the high costs involved in corn production, mainly in Brazil where P sources are costly. We observed a shift in soil P classes according to increased laboratory temperature (Table 11). Lower temperature, on average reduced the P recommended for corn by 33%. This large rate change surely could adversely affect corn yield. Temperatures from 15 to 25 °C did not affect the P recommendation, except for the Alfisol where the P level was originally classified as high (25 °C) and shifted to medium (15 °C). Based on the results, laboratory temperature must be taken into account and controlled in the soil testing laboratory to produce reliable P fertilizer recommendations for crops based upon a predictive soil-test result.

Soil	Amount of resin (n)													
	1	2	1	2	1	2	1	2						
	Р		K			Ca	Mg							
		m	g dm <sup>-3</sup>			cmo	$vl_{c} dm^{-3}$							
Oxisol	4.9 a	6.4 a	59.3 b	69.3 a	5.22 a	5.83 a	2.11 b	2.66 a						
	31.5 b	44.2 a	44.2 a	48.6 a	6.47 a	7.38 a	2.37 b	3.08 a						
	60.3 b	76.8 a	53.4 a	57.0 a	4.67 a	5.44 a	2.27 b	3.45 a						
Inceptisol	6.3 b	9.8 a	47.2 a	48.5 a	10.09 b	13.38 a	3.03 b	3.94 a						
	18.1 b	26.8 a	43.1 a	41.4 a	4.49 b	6.66 a	2.68 b	4.07 a						
	58.8 b	76.1 a	49.2 a	44.7 a	9.99 b	14.39 a	2.82 b	4.22 a						
Spodosol	19.0 a	19.2 a	31.5 b	39.5 a	2.38 a	3.23 a	1.26 b	1.72 a						
	50.8 b	54.0 a	30.6 b	36.3 a	2.66 a	3.49 a	1.36 b	1.91 a						
	81.9 b	85.6 a	34.8 a	36.2 a	3.03 a	3.90 a	1.25 b	1.76 a						
Alfisol	8.2 a	8.8 a	33.8 a	30.4 a	2.09 a	2.94 a	0.84a	1.19 a						
	20.0 a	21.4 a	30.6 a	31.1 a	1.86 a	2.72 a	0.75 a	1.10 a						
	73.0 b	82.3 a	28.7 b	34.3 a	2.27 a	3.03 a	0.80 a	1.09 a						
Acrisol	2.7 a	3.4 a	13.5 a	14.2 a	0.37 a	0.68 a	0.54 a	0.19 a						
	28.5 a	29.3 a	12.5 b	16.2 a	0.53 a	0.63 a	0.12 a	0.12 a						
	62.3 b	72.1 a	12.9 b	16.8 a	0.51 a	0.86 a	0.14 a	0.19 a						
Average	35.1 b	41.1 a	34.3 b	37.6 a	3.77 b	4.97 a	1.49 b	2.05 a						

Table 10. Phosphorus, potassium, calcium, and magnesium extracted with ion exchange resin membrane affected by amount of resin

Means followed with the same lower case letter within a row did not differ statistically by Tukey test (5%); C.V. (%) P = 4.6; C.V. (%) K = 8.0; C.V. (%) Ca = 13.0; C.V. (%) Mg = 14.

## 4. Conclusion

Modifications in the extraction procedure with ion-exchange resin membrane resulted in significant differences in the extracted amounts of soil P, K, Ca, and Mg. Among the variables studied, temperature had the greatest effect on the amounts of P extracted by resin ion exchange membrane and similarly with ion-exchange resin spheres. Temperature effects on soil P extraction can result in inadequate P recommendations to crops, which would have a larger impact on those soils with already low P availability to crops. Temperature must be controlled to avoid inaccurate results that can affect fertilizer recommendations to crops. Studies with soils from other regions and also with large variability in soil nutrient contents are necessary to better understand the factors that affect resin membrane efficiency in the soil testing analyzes.

		Laboratory temperature (°C)														
Soils	10				15				25				40			
	р															
	Soil-P mg·dm <sup>-3</sup>	Status <sup>†</sup>	Applied <sup>‡</sup> kg·ha <sup>-1</sup>	Balance <sup>¥</sup> %	Soil-P mg·dm <sup>-3</sup>	Status	Applied kg·ha <sup>-1</sup>	Balance %	Soil-P mg·dm <sup>-3</sup>	Status	Applied kg·ha <sup>-1</sup>	Balance %	Soil-P mg·dm <sup>-3</sup>	Status	Applied kg·ha <sup>-1</sup>	Balance %
Oxisol	3.0a	VL	215	100	4.5a	VL	215	100	4.9a	VL	215	100	7.2a	L	175	81
	15.5d	М	165	122	24.3c	Н	135	100	31.5b	Н	135	100	59.4a	VH	90	67
	32.2d	Н	135	150	46.6c	VH	90	100	60.3b	VH	90	100	103.8a	VH	90	100
Inceptisol	3.0b	VL	215	123	6.2ab	L	175	100	6.3ab	L	175	100	9.3a	L	175	100
	8.7c	L	175	106	14.1b	М	165	100	18.lb	М	165	100	32.1a	Н	135	82
	29.7d	Н	135	150	45.0c	VH	90	100	58.8b	VH	90	100	95.5a	VH	90	100
Spodosol	12.1c	М	165	100	17.7b	М	165	100	19.0b	М	165	100	27.8a	Н	135	82
	33.5d	Н	135	150	42.9c	VH	90	100	50.8b	VH	90	100	70.7a	VH	90	100
	52.8d	VH	90	100	71.0c	VH	90	100	81.9b	VH	90	100	1189a	VH	90	100
Alfisol	6.lb	L	175	100	7.5ab	L	175	100	8.2ab	L	175	100	11.8a	М	165	94
	11.3c	М	165	122	20.0b	М	165	122	20.9b	Н	135	100	28.6a	Н	135	100
	43.2d	VH	90	100	61.4c	VH	90	100	73.0b	VH	90	100	100.la	VH	90	100
Acrisol	1.9a	VL	215	100	2.6a	VL	215	100	2.7a	VL	215	100	4.1a	VL	215	100
	17.5c	М	165	122	26.6b	Н	135	100	28.5b	Н	135	100	40.9a	VH	90	67
	39.0d	Н	135	100	54.5c	VH	90	100	62.3b	VH	90	100	92.la	VH	90	100
Average	20.6d	-	90	67	29.7c	-	135	100	35.1b	-	135	100	53.5a	-	90	67

Table 11. Phosphorus extracted with ion exchange resin membrane affected by laboratory temperature and the impact on nutrient recommendation to corn in Southern Brazil<sup>§</sup>

*Note*. §: Means in soil-P followed with the same lower case letter within a row in soil-P did not differ statistically by Tukey test (5%); C.V. (%) = 7.1;

†: P status in soil based on soil-P extracted (mg dm<sup>-3</sup>) with membrane resin according to Southern Brazil Manual for Crop Nutrient and Lime Recommendations. Very Low (VL = soil-P  $\leq$  5.0); Low (L = soil-P range of 5.1-10.0); Medium (M = soil-P range of 10.1-20.0); High (H = soil-P range of 20.1-40.0); Very High (VH = soil-P  $\geq$  40.0);

‡: P applied to corn to an expected yield of 10 Mg ha<sup>-1</sup> according to Southern Brazil Manual for Crop Nutrient and Lime Recommendations.

<sup>¥</sup>: P balance is amount of P (in percent) above or below recommendation P applied considering 25 °C as laboratory temperature standard (100%).

#### References

- Amer, F., Bouldin, D. R., Black, C. A., & Duke, F. R. (1955). Characterization of soil phosphorus by anion exchange resin adsorption and P32 equilibration. *Plant Soil, 6*, 391-408. http://dx.doi.org/10.1007/ BF01343648
- Bache, B. W., & Ireland, C. (1980). Desorption of phosphate from soils using anion exchange resins. *Journal of Soil Science*, *31*, 297-306. http://dx.doi.org/10.1111/j.1365-2389.1980.tb02082.x
- Bortolon, L., & Gianello, C. (2008). Interpretação de resultados analíticos de fósforo pelos extratores Mehlich-1 e Mehlich-3 em solos do Rio Grande do Sul. *Revista Brasileira de Ciência do Solo, 32*, 2751-2756. http://dx.doi.org/10.1590/S0100-06832008000700019
- Bortolon, L., Gianello, C., & Kovar, J. L. (2011). Phosphorus availability to corn and soybean evaluated by three soil test methods for southern Brazilian soils. *Communications in Soil Science and Plant Analysis*, 42, 39-49. http://dx.doi.org/10.1080/00103624.2011.528488
- Cooke, I. J., & Hislop, J. (1963). Use of anion-exchange resin for the assessment of available soil phosphate. *Soil Science*, *96*, 308-312. http://dx.doi.org/10.1097/00010694-196311000-00004

- Cooperband, L. R., & Logan, T. J. (1994). Measuring in situ changes in labile soil phosphorus with anion-exchange membranes. *Soil Science Society of America Journal*, 58, 105-114. http://dx.doi.org/ 10.2136/sssaj1994.03615995005800010015x
- Dobermann, A., Langer, H., & Mutcher, H. (1994). Nutrient adsorption kinetics of ion exchange resin capsules: A study with soils of international origin. *Communications in Soil Science and Plant Analysis*, 25, 1329-1353. http://dx.doi.org/10.1080/00103629409369119
- Haagsma, T., & Miller, M. H. (1963). The release of non-exchangeable soil potassium to cation exchange resins as influenced by temperature, moisture and exchanging ion. *Soil Science Society of America Proceedings*, 27, 153-156. http://dx.doi.org/10.2136/sssaj1963.03615995002700020020x
- Kovar, J. L. (2003). Searching for more effective corn starters in conservation-till. Fluid Journal, 11, 8-11.
- Kovar, J. L., Bortolon, L., & Karlen, D. L. (2009). Distribution of phosphorus and potassium following surface banding of fertilizer in conservation tillage systems. *Plant Nutrition Colloquium Proceedings*. Retrieved from http://escholarship.org/uc/item/8j0224dq
- Mallarino, A. P., & Atia, A. M. (2005). Correlation of a resin membrane soil phosphorus test with corn yield and routine soil tests. Soil Science Society of America Journal, 69, 266-272. http://dx.doi.org/10.2136/ sssaj2005.0266
- McLaughlin, M. J., Lancaster, P. A., & Sale, P. W. G. (1993). Use of cation/anion exchange membranes for multi-element testing of acid soils. *Plant Soil*, 22, 249-252.
- Qian, P., Schoenau, J. J., & Huang, W. Z. (1992). Use of ion exchange membrane in routine soil testing. Communications in Soil Science and Plant Analysis, 23, 1791-1804. http://dx.doi.org/10.1080/00103629 209368704
- Raij, B., Quaggio, J. A., & Silva, M. N. (1986). Extraction of phosphorus, potassium, calcium, and magnesium from soils by an ion-exchange resin procedure. *Communications in Soil Science and Plant Analysis*, 17, 547-566. http://dx.doi.org/10.1080/00103628609367733
- Rolim, M. V., Novais, R. F., Nunes, F. N., & Alvarez, V. H. V. (2008). Efeito da moagem do solo no teor de fósforo disponivel por Mehlich-1, resina em esferas e em lâmina. *Revista Brasileira de Ciência do Solo, 32*, 1181-1190. http://dx.doi.org/10.1590/S0100-06832008000300026
- Saggar, S., Hedley, M. J., & White, R. E. (1990). A simplified resin membrane technique for extracting phosphorus from soils. *Nutrient Cycling in Agroecossystem*, 24, 173-180. http://dx.doi.org/10.1007/bf01 073586
- SBCS/NRS (Sociedade Brasileira de Ciência do Solo/Núcleo Regional Sul). (2004). *Manual de adubação de de calagem para os estados do Rio Grande do Sul e Santa Catarina*. Porto Alegre, UFRGS. Retrieved from http://www.sbcs-nrs.org.br/docs/manual\_de\_adubacao\_2004\_versao\_internet.pdf
- Schlindwein, J. A., & Gianello, C. (2009). Fósforo disponível determinado por lâmina de resina enterrada. *Revista Brasileira de Ciência do Solo, 33*, 77-84. http://dx.doi.org/10.1590/S0100-06832009000100008
- Schoenau, J. J., & Huang, W. Z. (1991). Anion-exchange membrane, water, and sodium bicarbonate extractions as soil tests for phosphorus. *Communications in Soil Science and Plant Analysis*, 22, 465-492. http://dx.doi.org/10.1080/00103629109368432
- Sibbesen, E. A. (1978). Simple ion-exchange resin procedure for extracting plant-available elements from soil. *Plant Soil, 46*, 665-669. http://dx.doi.org/10.1007/BF00015928
- Simonis, A. D. (1996). Effect of temperature on extraction of phosphorus and potassium from soils by various extracting solutions. *Communications in Soil Science and Plant Analysis*, 27, 665-484. http://dx.doi.org/10.1080/00103629609369586
- Skogley, E. O., Geortis, S. J., & Yang, J. E. (1990). The phytoavailability soil test PST. Communications in Soil Science and Plant Analysis, 21, 1229-1243. http://dx.doi.org/10.1080/00103629009368301
- Somarisi, L. L. W., & Edwards, A. C. (1992). An ion exchange resin method for nutrient extraction of agricultural advisory soil samples. *Communications in Soil Science and Plant Analysis*, 23, 645-657. http://dx.doi.org/10.1080/00103629209368616
- Sparks, D. L. (2003). Environmental soil chemistry. San Diego: Academic Press.

- Takahashi, S. (1996). Influence of soil particle size and grinding on Bray-2 extractable phosphorus. Communications in Soil Science and Plant Analysis, 27, 2829-2835. http://dx.doi.org/10.1080/ 00103629609369744
- Takahashi, S. (2013). Soil phosphorus extracted by three anion exchange membranes compared to water-soluble and truog-extractable phosphorus. *Communications in Soil Science and Plant Analysis, 44*, 3231-3234. http://dx.doi.org/10.1080/00103624.2013.840835
- Tedesco, M. J., Gianello, C., Bissani, C. A., Bohnen, H., & Volkweiss, S. J. (1995). Análise de solo, plantas e outros materiais. Porto Alegre, UFRGS.
- Vaidyanathan, L. V., & Talibuden, O. (1970). Rate process in the desorption of phosphate from soils by ion-exchange resins. *Journal of Soil Science*, 21, 173-183. http://dx.doi.org/10.1111/j.1365-2389.1970.tb 01165.x
- Vandecar, K. L., Lawrence, D., & Clarck, D. (2010). Phosphorus Sorption Dynamics of Anion Exchange Resin Membranes in Tropical Rain Forest Soils. Soil Science Society of America Journal, 75, 1520-1529. http://dx.doi.org/ 10.2136/sssaj2010.0390
- Yang, J. E., Skogley, E. O., & Geortis, S. J. (1991a). Phytoavailability soil test: development and verification of theory. Soil Science Society of America Journal, 55, 1358-1365. http://dx.doi.org/10.2136/sssaj1991.036159 95005500050027x
- Yang, J. E., Skogley, E. O., & Schaff, B. E. (1991b). Nutrient flux to mixed-bed ion exchange resin: temperature effects. Soil Science Society of America Journal, 55, 762-767. http://dx.doi.org/10.2136/sssaj1991.036159 95005500030021x

## Copyrights

Copyright for this article is retained by the author(s), with first publication rights granted to the journal.

This is an open-access article distributed under the terms and conditions of the Creative Commons Attribution license (http://creativecommons.org/licenses/by/4.0/).