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Luzia Otília Bortotti Favero[1], Ervim Lenzi[1(*)], Eduardo Bernardi Luchese[2], e Luciano Márcio de Moraes[1]

[1] - Chemistry Department of State University of Maringá (Departamento de Química da Universidade Estadual de Maringá). Av. Colombo, 5790; Caixa Postal, 331; CEP 87.020-900; Maringá; Paraná - Brasil. [2] - Agronomy Department of State University of Paraná West (Departamento de Agronomia da Universidade Estadual do Oeste do Paraná). Rua Pernambuco, 1777; CEP-85.960-000; Marechal Candido Rondon; Paraná - Brasil. (*) – Author for correspondence.

ABSTRACT: *Samples of six types of soil were tested in a greenhouse. Three doses of boric acid: 0.0 g (control); 0.10 g and 0.20 g were applied to the prepared soils. Seeds of cabbage were sown and only two plants remained after cutting. Mehlich 1, HCl 0.05 mol.L^{-1} and the saturation extract were used to determine the boron in the soil. After determining the dry matter and calcined the plants ashes were dissolved in HCl 0.6 mol.L^{-1}. The boron concentration was determined by the Azomethine-H method. The correlation between boron concentration in the plant (μg.g^{-1}) and total boron in the plant (μg.plant^{-1}) versus boron concentration in the soil showed that among the three extractors the HCl 0.05 mol.L^{-1} and saturation extract presented the best correlation coefficients (r): 0.857 and 0.832, 0.858 and 0.841 respectively. The Mehlich 1 extractor presented the lowest correlation coefficients for all conditions studied.*

Key words: boron extractors, micronutrient, boron, element extraction, boron in soil.

RESUMO: *Amostras de 6 tipos de solo foram testadas em casa de vegetação. Nos solos preparados foram aplicadas três doses de ácido bórico: 0,0 (testemunha); 0,10 g e 0,20 g. Sementes de couve foram semeadas. No desbaste foram deixadas 2 plantas. Os extratores: Mehlich 1; HCl 0,05 mol.L^{-1} e extrator de saturação foram usados para extrair o boro do solo. Após determinar a massa seca, e respectiva calcinação, as cinzas das plantas foram dissolvidas em solução de HCl 0,6 mol.L^{-1}. A concentração do boro foi determinada pelo método da Azometina-H. As correlações entre a concentração de boro na planta (em μg.g^{-1}) e o total de boro na planta (em μg.plant^{-1}) verso a concentração de boro nos solos mostraram que entre os três extratores o HCl 0,05 mol.L^{-1} e o extrator de saturação apresentaram os melhores coeficientes de correlação (r): 0,857 e 0,832; 0,858 e 0,841 respectivamente. O extrator Mehlich 1 apresentou os coeficientes de correlação mais baixos para todas as condições analisadas.*

INTRODUCTION

Extraction by hot[1] water is the most widely used method to detect boron critical content in the plants. Though it is feasible to find in limed soils a better

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correlation between boron extracted by $CaCl_2$ 0.01 $mol.L^{-1}$ and Mehlich 1 extractors and that absorbed by the plant, this is not a normal condition in the soils routinely[2] analyzed in the laboratory.

Boron deficiencies in plants are found when the concentration values interval of this element in soil ranges from 0.1 to 0.7 ppm [3-4-5].

Using coffee shrub as reference and saturation extract as extractor, it was verified that for the DRL (dystrophic red latosol) the boron concentrations in the extract can be considered low when below 0.3 ppm, suitable when close to 0.6-0.8 ppm and high when over 1.0 ppm[6].

Lopes and Carvalho[7] summarized the most frequent methodologies used to determine the boron available for the plants and consider: $Ca(H_2PO_4)_2.H_2O$ solution, hot water, saturation extract, H_2SO_4, HF and HCl diluted solutions as extractors. The soil/extractor relation varies from 1:1 to 1:2 during a five-minutes to sixteen-hours shaking period. Up to now the amplitude of critical levels varies from 0.2 to 2 ppm for the different extractors proposed.

Undoubtedly, hot water is the most appropriate extractor for the determination of boron available, but it does not allow the use of pyrex-type borosilicated glasses. Otherwise, it is a laborious technique which makes it difficult to determine this element in routine laboratory analysis.

With the purpose of dynamizing the routine analysis either as a function of the time spent or the handling of the materials, new extractors such as Mehlich 1, diluted acids, 0.5 $mol.L^{-1}$ mannitol + $CaCl_2$, $CaCl_2$, and 0.005 $mol.L^{-1}$ [8-6-9] are being tested and used. The solution of $BaCl_2$. $2H_2O$ 1.25 $g.L^{-1}$ heated in a microwave stove was also recommended for the extraction of boron in routine laboratory analyses, as well as, for the determination in ICP-AES (Inductively Coupled Plasma – Atomic Emission Spectroscopy)[10]. The characterization of these extractors for several types of soil and plants, and the correlation of the critical levels between them constitutes a vast field of study.

Our purpose is to study three extractors: Mehlich 1, HCl 0.05 $mol.L^{-1}$ and the saturation extract using cabbage as a test plant.

MATERIALS AND METHODS

List of soils

Six classes of soils (DRL-dystrophic red latosol, ERL-eutrophic red latosol, DRAL – dark red allic latosol, AC-allic cambisol, DSRS-dystrophic structured red soil and DDRL-dystrophic dark red latosol), already used in a previous experiment to evaluate cotton plant response to boron[11] application, were used in this study.

Preliminary determination in the soil samples

Some conditions of soil fertility which influence boron concentration in soil was assessed through the routine analysis in the *Laboratório de Agroquímica do Departamento de Química da UEM (Universidade Estadual de Maringá* - Laboratory of Agrochemistry-Department of Chemistry of the *Universidade Estadual de Maringá*), see Table 1.

The pipet[12] method was used for the granulometric analysis in the Laboratory of Pedology, Sedimentology and Palynology of the Department of Geography of the Universidade Estadual de Maringá, (see Table 1).

Boron extraction and determination in the soil

The extractors used to classify boron available for the plants in the soils listed were as follow, with their respective procedures:

HCl 0.05 mol.L^{-1} [6]: to each 10 g of ADFE (air-dried fine earth), add 20 mL of HCl 0.05 mol.L^{-1}, shake for 5 minutes and filter in a Whatman-42 paper filter.

Mehlich 1[8]: transfer 5 mL of ADFE to a erlenmeyer-container, add 50 mL of Mehlich 1 solution, (H_2SO_4 0.0125 mol.L^{-1} + HCl 0.05 mol.L^{-1}), shake for 5 minutes and decant during one night.

Saturation extract[6]: transfer 500 g of soil to plastic recipients, moisten with a determined quantity of distilled water to complete saturation. Then rest it for 12 hours and vacuum-extract the solution.

The Azomethina-H[13] colorimetric method was used to analyze the boron in the respective extracts.

Procedures of the experiment.

The experiment was set up in a greenhouse, using 2.5-liter vases. The triplicate treatments were 0.0 g, 0.10 g and 0.20 g of boric acid-H_3BO_3 respectively. For 15 days the vases were filled with water up to 70% retention capacity. Six cabbage seeds were sown in each vase and after ten days of germination they were pared down, remaining only two plants in each vase.

The vases were kept in a greenhouse according to a totally randomized design and regularly watered to maintain humidity.

Collection of vegetal material and soil samples from the vases.

Eight weeks and half after seeding the aerial portion of the plants was close-cut, washed with distilled and deionized water, dried in a 65 °C hothouse till reaching their constant weight, ground in a Wiley grinder with a 1mm-mesh sieve and packed in plastic bags ready for the analysis[14].

After the plant collecting the vases were emptied and the soils air-dried, ground, in a 2-mm-mesh sieved, homogenized and kept in plastic bags for future analysis[15].

Boron analysis in the plants and soils

For four hours[16] 0.40 g vegetal material, contained in porcelain vases, was incinerated in muffle at 550 °C temperature. After cooling 20 mL of HCl 0.6 mol.L^{-1} were added. Using the complexant Azomethina H[13], the boron concentration was determined in the extract obtained by means of UV-VIS spectrophotometry.

Boron determination in the soils followed the above-mentioned extraction and complexations.

RESULTS AND DISCUSSION

Contents of organic matter and clay and pH which characterize the soils used in the experiment are shown in Table 1. These factors may influence boron micronutrient availability in the soils.

According to Table 2, the extractors behaved differently in the boron-extraction procedure for all types of soil. The Mehlich 1 extractor provided the greatest quantity of the micronutrient while the saturation extract, the smallest. The HCl 0.05 $mol.L^{-1}$ and the Mehlich 1 extractor removed the same quantity of boron content only from the DDRL soil, which is a very sandy soil with low content of organic matter as it can be seen in Table 1.

Table 1 – Characteristics of the soils used in the experiment

Type of soil*	pH (H_2O)	Organic matter $g.dm^{-3}$	Clay %	Silt %	Sand %
DDRL	7.2	5.35	9.65	3.35	87.0
DRL	6.2	6.69	65.47	31.53	3.0
ERL	6.3	12.06	59.45	34.56	6.0
DRAL	6.2	5.35	38.77	47.73	13.5
AC_1	5.7	28.14	39.57	55.43	5.0
AC_2	5.7	30.75	52.62	45.38	2.0
DSRS	6.6	18.75	65.40	33.60	1.0

* **DDRL** - dystrophic dark red latosol; **DRL** – dystrophic red latosol; **ERL** – eutrophic red latosol; **DRAL** – dark red allic latosol; **AC_1** – allic cambisol; **AC_2** - allic cambisol and **DSRS** – dystrophic structured red soil.

The boron extractors HCl 0.05 $mol.L^{-1}$ and the saturation extract showed to be more efficients, as boron extractors, than Mehlich 1 when their respective boron concentrations extracted from the soils were correlated with the boron concentrations absorbed by the plants cultivated. The variance analysis was significant on the level of 1% for these extractors once all data were considered in relation to the boron concentration in the aerial portion in $\mu g.g^{-1}$ as well as in relation to the boron absorbed by the plant in $\mu g.plant^{-1}$ with the correlation coefficient of 0.857 and 0.832 respectively for hydrochloric acid and 0.858 and 0.841 for the saturation extract in Table 3.

Taking into account only the vases that were fertilized with borate (Table 4) the variance analysis was 1% of significance for the HCl 0.05 $mol.L^{-1}$ and saturation extractors. For both extractors, the correlation coefficients remained close to 0.8 in relation to the boron content in the vegetal tissue as well as for the boron absorbed by the plant. Bartz and Magalhães[17] also found better correlation between the boron extracted by acid extractors and the boron absorbed by the plants, as compared to boron fertilized soils. However, Renan and Gupta[18] found a positive correlation between the boron extracted from the soil with HCl 0.05 $mol.L^{-1}$ and that absorbed by the plant in any class of soil.

Table 2 - Boron concentration in soils determined by different extractor solutions

Soil*	Extractors	$\mu g.g^{-1}$ (values intervals)	$\mu g.g^{-1}$ (average)
DRL	Mehlich 1	1.00 - 10.00	5.06
	HCl 0.05 $mol.L^{-1}$	0.03 - 4.57	2.01
	Saturation extract	0.06 - 1.42	0.54
ERL	Mehlich 1	1.16 - 7.35	4.41
	HCl 0.05 $mol.L^{-1}$	0.23 - 2.34	1.37
	Saturation extract	0.03 - 0.52	0.26
DRAL	Mehlich 1	2.67 - 6.11	4.16
	HCl 0.05 $mol.L^{-1}$	1.00 - 3.76	2.69
	Saturation extract	0.05 - 0.70	0.32
AC_1	Mehlich 1	2.15 - 9.14	6.29
	HCl 0.05 $mol.L^{-1}$	0.16 - 4.25	1.98
	Saturation extract	0.19 - 1.80	1.06
AC_2	Mehlich 1	3.32 - 13.34	8.27
	HCl 0.05 $mol.L^{-1}$	0.84 - 3.00	1.76
	Saturation extract	0.27 - 2.73	1.28
DSRS	Mehlich 1	1.45 - 10.94	5.28
	HCl 0.05 $mol.L^{-1}$	0.03 - 3.50	1.61
	Saturation extract	0.03 - 1.01	0.37
DDRL	Mehlich 1	0.47 - 8.70	4.53
	HCl 0.05 $mol.L^{-1}$	0.38 - 8.89	4.21
	Saturation extract	0.13 - 4.49	1.91

* **DDRL** - dystrophic dark red latosol; **DRL** – dystrophic red latosol; **ERL** – eutrophic red latosol; **DRAL** – dark red allic latosol; **AC_1** – allic cambisol; **AC_2** - allic cambisol and **DSRS** – dystrophic structured red soil.

For the soils of the control vases the correlation between boron content extracted by HCl 0.05 $mol.L^{-1}$ and boron concentration in the plant or boron absorbed by the plant was significant at the level of 5% and the correlation coefficients were 0.627 and 0.523 respectively (Table 4).

The Mehlich 1 extractor showed low correlation coefficients (Tables 3 and 4), verifying the literature on the subject. For the hydromorphic and alluvial soils under flooded rice cultivation, Paula *et al.*[19] found correlation coefficients close to Mehlich 1 and hot water extractors, thus indicating the feasibility of replacing hot water by Mehlich 1.

Table 3 – Linear correlation coefficients of boron concentration absorbed by the plant in $\mu g.g^{-1}$ and $\mu g.plant^{-1}$ respectively with the boron content available in the soil in the extractors used for all data obtained in the experiment.

Extractor	Boron concentration in the plant ($\mu g.g^{-1}$)	Boron absorbed by the plant ($\mu g.plant^{-1}$)
Mehlich 1	0.342*	0.411**
HCl 0.05 $mol.L^{-1}$	0.857**	0.832**
Saturation extract	0.858**	0.841**

Levels of significance (* - 5%) (** - 1%)

Table 4 - Linear correlation coefficients of boron concentration absorbed by the plant in $\mu g.g^{-1}$ and $\mu g.plant^{-1}$ respectively with the boron content available in the soil in the extractors used before and after fertilization.

Extrator	Boron concentration in the plant ($g.g^{-1}$)		Absorbed boron by the plant. ($\mu g.plant^{-1}$)	
	Without fertilization (control)	With fertilization	Without fertilization (control)	With fertilization
Melich 1	0.389	0.109	0.326	0.177
HCl 0,05 $mol.L^{-1}$	0.627*	0.874**	0.553*	0.828**
Saturation extract	0.226	0.808**	0.283	0.799**

Levels of significance (* - 5%) (** - 1%)

Table 5 - Linear correlation coefficients between contents of soil boron micronutrients determined by different extractors

Extractor	HCl 0.05 $mol.L^{-1}$	Saturation extract
Mehlich 1	0.527**	0.604**
HCl 0.05 $mol.L^{-1}$	-	0.800**

Level of significance (** - 1%)

The variance analysis of the linear correlation between the extractors was significant at the level of 1% for the Mehlich 1 x HCl 0.05 $mol.L^{-1}$, Mehlich 1 x saturation extract and HCl 0.05 $mol.L^{-1}$ x saturation extract correlations of which the last one showed the best correlation coefficient (0.800) (Table 5).

Figure 1 shows a linear correlation between the boron content absorbed by the plants and that extracted by the three extractors and their respective equations.

CONCLUSIONS

The Mehlich 1 extractor extracted the greatest amount of boron from the soils, while the saturation extract, the least.

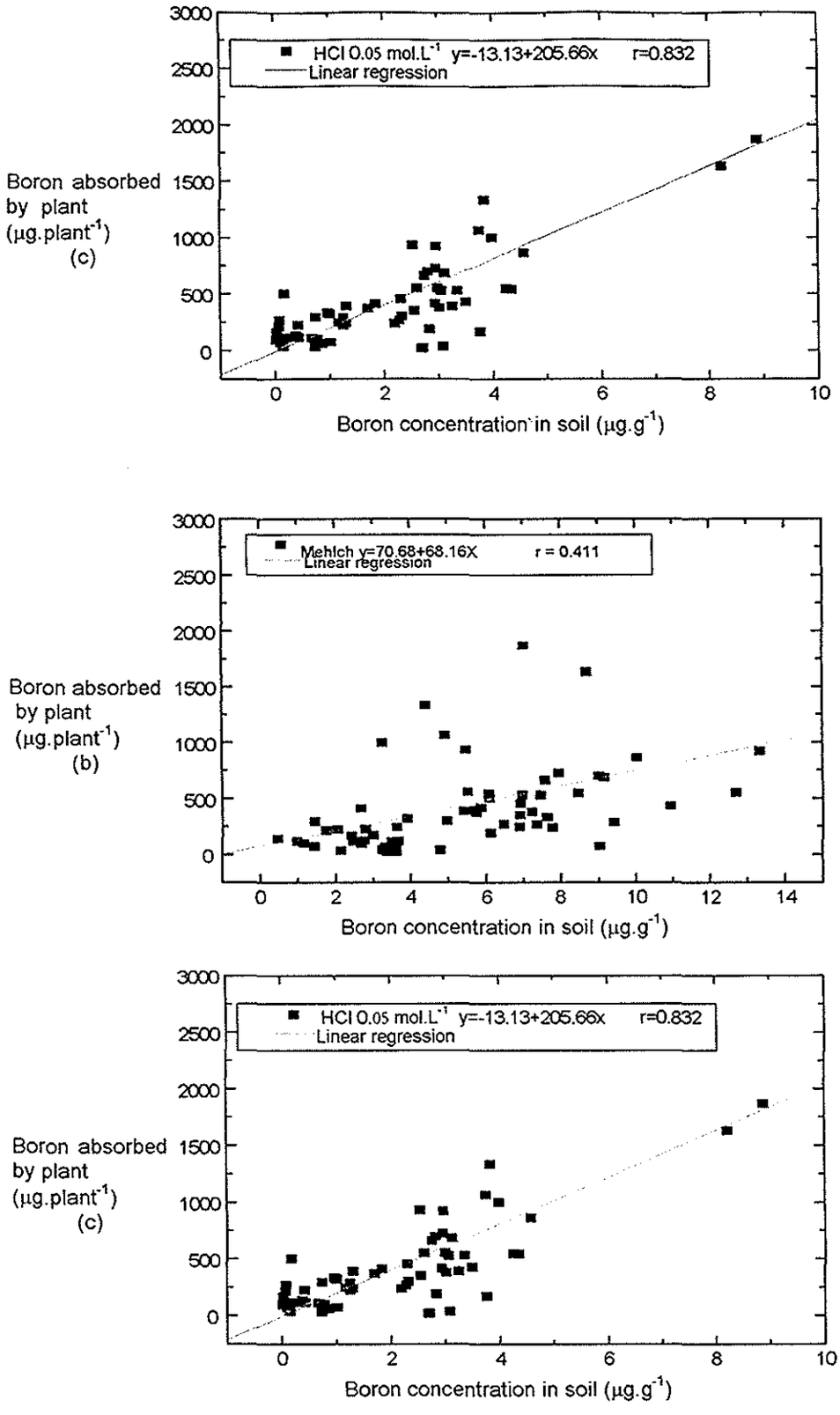

Figure 1 – Linear correlation graphs for boron absorbed by the plant and boron concentration in the soil extracted by (a) saturation extract, (b) Mehlich 1 (c) HCl 0.05 mol.L^{-1}.

Saturation extract and HCl 0.05 mol.L^{-1} extractors showed the best correlation coefficients between boron concentration in the plant in $\mu g.g^{-1}$, and the total boron per plant, in g/plant x the boron extracted from the soil by these extractors.

The correlation coefficient between the HCl 0.05 mol.L^{-1} and the saturation extract extractors was the best (0.800).

Taking into consideration the correlation coefficients and execution facility, the HCl 0.05 mol.L^{-1} is the best among all extractors studied.

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