

Role of Organic Matter on Boron Adsorption-Desorption Hysteresis of Soils

Sabine Goldberg, PhD and Donald L. Suarez, PhD

Abstract: In this study, we evaluated the boron (B) adsorption-desorption reaction of six soils and examined the extent to which organic matter content, as well as incubation time, affected B release. Six soils varying in initial pH, clay content, and organic matter content were selected for the study. Adsorption experiments were carried out to determine B adsorption isotherms using 0.01 M NaCl solutions containing 0 to 100 mg B kg⁻¹. The soils were equilibrated for 23 h. Boron desorption was investigated after replacing aliquots with B-free 0.01 M NaCl solution three times. Three of the soils were also spiked with 0 to 100 mg B kg⁻¹ and equilibrated for up to 23 months at field capacity water content in a temperature-controlled room at 25°C. At various intervals, sub-samples were extracted with DTPA-sorbitol, boiling water, and 0.1 M NaCl. The B adsorption-desorption behavior of all six soils was found to be nonhysteretic in the short-term experiment, regardless of organic matter content. Apparent hysteresis of B adsorption can result from difficulties in the experimental procedure itself. Some B adsorption hysteresis was found for two soils with all three extractants after long-term equilibrations of 1 to 23 months. Our results do not support the hypothesis that B desorption hysteresis increases in soils with increasing organic matter content.

Key words: Extractable B, B release, Langmuir adsorption isotherm.

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Boron is an important plant essential micronutrient whose soil availability is influenced by many soil factors. Organic matter is an important constituent that adsorbs boron (B) and influences the solution B concentration and B availability. A highly significant correlation was found between native soil B content and soil organic carbon content (Elrashidi and O'Connor, 1982). Soluble B extracted by hot water was highly significantly correlated with organic matter content for acid Wisconsin soils (Berger and Truog, 1945), as well as for Canadian soils (Miljkovic et al., 1966; Gupta, 1968). Adsorbed B was also highly significantly correlated with carbon content for 23 Japanese soils (Harada and Tamai, 1968) and for 10 soils from New Mexico (Elrashidi and O'Connor, 1982). The magnitude of B adsorption maxima obtained from the Langmuir adsorption isotherm equation was significantly correlated with organic carbon content for 20 Canadian soils (Evans, 1987). Organic matter content was also an important constituent needed for prediction of B adsorption parameters for the constant capacitance surface complexation model (Goldberg et al., 2000).

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A different type of behavior was found by Marzadori et al. (1991), who observed increased B adsorption by soils after organic matter removal and suggested that the organic matter had been occluding some B-adsorbing sites on iron and aluminum oxides. Similarly, Mandal et al. (1993) observed that organic matter application increased recovery of added B by four different extractants. The effect of small amounts of native organic matter on B adsorption by soils was found to be negligible (Mezuman and Keren, 1981). Data on the effect on B adsorption from external applications of organic matter appear dependent on the type of organic matter added, with solid materials enhancing adsorption and dissolved organic matter suppressing adsorption. Application of farmyard manure was found to increase B adsorption by soils (Sharma et al., 2006). These authors suggested that such applications to soils receiving high concentrations of B could mitigate B toxicity by reducing the release of adsorbed B. Increasing the amount of composted organic matter in soil increased the amount of B adsorbed at any given solution pH and B concentration (Yermiyahu et al., 1995).

Humus extracted from soil was considered to play an important role in B adsorption, despite its small total content in mineral soils, because it retained significant amounts of B (Parks and White, 1952). The B adsorption capacity of bark compost was found to be 10-fold greater than that of a greenhouse soil (Van et al., 2005). Boron adsorption capacity of humic acid and composted organic matter on a weight basis was greater than the adsorption capacity of clay minerals (Yermiyahu et al., 1988; Gu and Lowe, 1990; Lemarchand et al., 2005).

In contrast, B adsorption was decreased as the application of dissolved organic matter from sewage effluent was increased (Communar and Keren, 2008). Adsorption of B on polysaccharide coated clays was lower than on pure plays (Gu and Lowe, 1992). Boron adsorption on amorphous aluminum hydroxide was also lower when it was coated with 5% humic acid (Xu and Peak, 2007). Conversely, Prodromou (2004) found that amorphous aluminum hydroxide adsorbed 76% more B in the presence of low-molecular-weight organic acids.

The assumption is often made that B adsorption-desorption reactions in soils occur virtually instantaneously and completely reversibly, justifying the use of B adsorption data to describe B desorption behavior. However, information on the reversibility of B adsorption is contradictory. For some soils, the B desorption isotherm closely mirrored the adsorption isotherm (Hatcher and Bower, 1958; Elrashidi and O'Connor, 1982). For other soils, B desorption exhibited hysteresis, meaning that the desorption data did not show complete reversibility of the adsorption data (Elrashidi and O'Connor, 1982). Information on the effect of soil organic matter on B desorption hysteresis is also contradictory. Elrashidi and O'Connor (1982) found no significant correlation between soil organic matter and hysteretic B desorption. However, Datta and Bhadoria (1999) found significantly increasing irreversibility of B desorption with increasing organic matter content for 25 Indian soils. In contrast, B desorption hysteresis of three Italian soils was increased after organic matter was removed (Marzadori et al., 1991). The B

adsorption behavior of composted organic matter was found to be profoundly hysteretic (Yermiyahu et al., 1988). Boron adsorption by the inorganic soil constituent clay minerals, kaolinite, illite, and montmorillonite, did not exhibit hysteresis (Hingston, 1964). Desorption hysteresis was found to be present at low solution pH and low B concentration and absent at higher solution pH and increasing B concentrations (Meyer and Bloom, 1997; Chen et al., 2009; Majidi et al., 2010). Clearly, the contribution of soil organic matter to the hysteresis of B adsorption-desorption requires further investigation.

Hysteresis of the B desorption reaction from organic matter was considered indicative of a chemical association (Yermiyahu et al., 1995). Parks and White (1952) suggested that B reacts with diols released and formed during the microbiological breakdown of organic matter in soils. Spectroscopic ^{11}B NMR experiments suggested that B was tetrahedrally adsorbed by reference humic acid (Lemarchand et al., 2005). One of the observed chemical shifts corresponded to a 1,3 diol six-membered ring complex, whereas the other was attributed to either a 1,2 diol five-membered ring complex or to a dicarboxylic complex.

The objectives of this study were (i) to evaluate B adsorption as a function of solution B concentration on a set of soils containing various amounts of organic matter and (ii) to evaluate the relationship between any observed hysteresis in B desorption and organic matter content of the soils.

MATERIALS AND METHODS

Boron adsorption was investigated using one soil sample from California, one soil sample from Arizona, two soils from the International Humic Substances Society Source Materials (one from Illinois and one from Florida), and two soils from the Catholic University Leuven soil archive (one from the Netherlands and one from Britain). Soil chemical and physical characteristics, clay mineralogy, and soil types are provided in Table 1. Soil surface areas were measured using ethylene glycol monoethyl ether adsorption (Cihacek and Bremner, 1979). To extract free iron and aluminum oxides, samples were treated with a citrate/citric acid buffer and hydrosulfite. Dissolved Fe and Al concentrations were analyzed using inductively coupled plasma-optical emission spectrometry (ICP-OES). Carbon content was quantified colorimetrically; inorganic carbon (IOC) using an

acidification module and heating; and organic carbon (OC) as the difference between total carbon determined by combustion at 950°C and IOC. Clay content was obtained using the basic hydrometer method (Gee and Bauder, 1986). Clay minerals present in the fraction less than 2 μm were determined by X-ray diffraction analyses of Mg- and K-saturated samples.

Boron adsorption experiments were carried out on the soils in batch systems to determine adsorption isotherms (amount of B adsorbed as a function of equilibrium solution B concentration). Samples of soil (20 g for Hanford, Glendale, Elliott; 10 g for Pahokee, Woburn, Zegveld) were added to 50-mL polypropylene centrifuge tubes and equilibrated with 20 mL of a 0.01 M NaCl solution for 23 h on a reciprocating shaker. The electrolyte solutions consisted of 0, 1, 2, 5, 10, 25, 50, or 100 mg B L $^{-1}$. The reaction temperature was 23.5°C \pm 0.2°C. After the reaction, the samples were centrifuged; aliquots (10 mL for Hanford, Glendale, Elliott, Woburn and 5 mL for Pahokee, Zegveld) of the supernatant were removed, analyzed for pH, passed through 0.45- μm membrane filters, and analyzed for B concentrations using ICP-OES. To investigate B desorption, the sampled aliquots were replaced with equivalent aliquots of B-free 0.01 M NaCl solution, shaken vigorously to resuspend, and equilibrated for another 23 h on a reciprocating shaker. This procedure was repeated two more times for a total of three desorption steps. We did not attempt to alter the pH of the soils but rather report the pH values in 0.01 M NaCl solution.

Incubation experiments were carried out at the approximate field capacity water content, considered to correspond to -1 kPa pressure, for 23 months for the Hanford soil and for 17 months for the Woburn and Zegveld soils. The experimental procedure for the incubation was described in a prior publication (Goldberg and Suarez, 2011), where the data for the first 5 months of incubation for the Hanford soil were presented. In brief, samples of air-dry soil were equilibrated with appropriate amounts of boric acid solution to achieve -1 kPa pressure water content and added soil B contents of 0, 3, 8, 20, or 50 mg B kg $^{-1}$ soil. The soil-solution mixtures were homogenized and incubated in a constant temperature room at 25°C \pm 0.5°C. The treatments were replicated three times. At monthly intervals for 5 months, the soils were thoroughly mixed and sub-sampled. The Hanford soil was also sampled after 11 and 23 months, whereas the Woburn and Zegveld soils were sampled after 17 months. The

TABLE 1. Chemical and Physical Characteristics and Classifications of Soils

Classification and Clay Mineralogy	Depth (cm)	Clay (%)	SA (m 2 g $^{-1}$)	SA	IOC	OC	Fe	Al
				m 2 g $^{-1}$	-----%			
Hanford—Typic Xerorthent montmorillonite, illite, kaolinite	0–10	9.6	7.4	17.5	0.00525	0.72	0.489	0.0314
Glendale—Typic Torrifluent montmorillonite, illite, kaolinite	Surface	20.0	8.0	98.4	0.33	1.06	0.786	0.0489
Elliott—Aquic Argiudoll illite, kaolinite, montmorillonite	0–15	23.0	6.9	51.6	<0.001	2.93	1.10	0.127
Woburn—Dystric Cambisol* montmorillonite, chlorite, kaolinite, illite	0–15	27.0	6.7	108	0.022	3.21	2.77	0.0818
Pahokee—Lithic Haplosaprist kaolinite, vermiculite	0–15	9.3	5.0	117	<0.001	47.0	1.08	0.178
Zegveld—Histosol* illite, kaolinite, vermiculite	0–15	20.0	4.7	12.2	0.013	29.1	0.881	0.251

*European Soil Classification System.

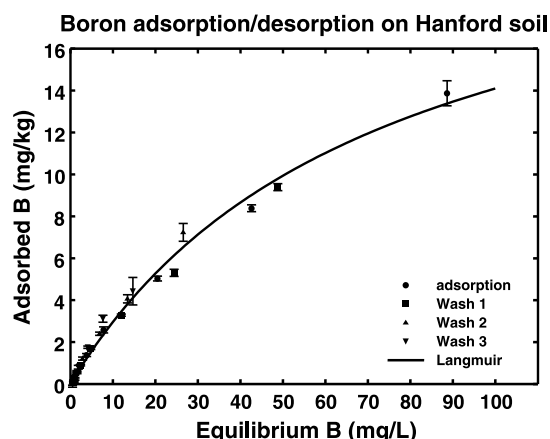


FIG. 1. Boron adsorption-desorption as a function of solution B concentration for Hanford soil. Adsorption data are represented by circles, Wash 1 data by squares, Wash 2 data by up triangles, and Wash 3 data by down triangles. Error bars represent 1 S.D. from the mean of three replicates. If error bars are not visible, they are smaller than the symbol size. The solid line represents the fit of the Langmuir adsorption isotherm.

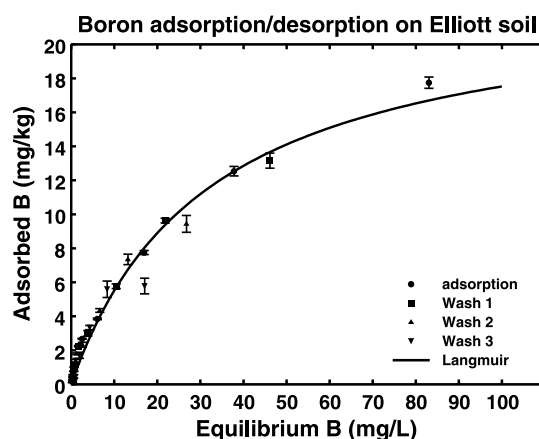


FIG. 3. Boron adsorption-desorption as a function of solution B concentration for Elliott soil. Adsorption data are represented by circles, Wash 1 data by squares, Wash 2 data by up triangles, and Wash 3 data by down triangles. Error bars represent 1 S.D. from the mean of two replicates. If error bars are not visible, they are smaller than the symbol size. The solid line represents the fit of the Langmuir adsorption isotherm.

sub-samples were analyzed for hot water-soluble B, DTPA-sorbitol extractable B, and 0.1 M NaCl extractable B as described in Goldberg and Suarez (2011). The extractions were carried out without drying the soils from their field capacity water content to avoid changes in adsorbed B that can occur from wetting and drying regimes. Boron concentrations in the filtrates were determined using ICP-OES spectrometry.

RESULTS AND DISCUSSION

Boron adsorption as a function of equilibrium B concentration is depicted in Figs. 1–4, 5B, and 6 by circles. Boron adsorption was greatest in the Pahokee and Glendale soils and least in the Hanford soil. Soil factors that may contribute to the

extent of B adsorption are clay content, pH, IOC, OC, and Al and Fe oxide content. The data conform to the Langmuir adsorption isotherm equation over the entire concentration range (0–100 mg B L⁻¹) investigated. The Langmuir equation parameters are provided in Table 2. Correlation coefficients relating the soil properties listed in Table 1 to the Langmuir adsorption maxima in Table 2 were not statistically significant, most likely due to the small size of the sample population.

Data points for the three desorption steps corresponding to the B adsorption data are indicated on Figs. 1–4, 5B, and 6. The desorption data are depicted by squares for Wash 1, up triangles for Wash 2, and down triangles for Wash 3. For all six soils, the B desorption data transverse along the B adsorption isotherms indicating complete reversibility of the B adsorption reaction. The observation of complete reversibility was surprising for the Glendale soil because Elrashidi and O’Connor (1982) had

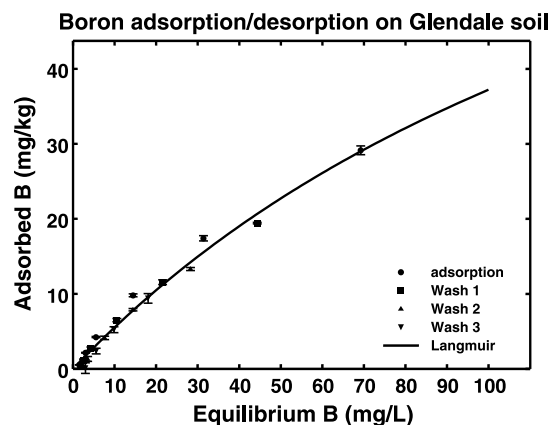


FIG. 2. Boron adsorption-desorption as a function of solution B concentration for Glendale soil. Adsorption data are represented by circles, Wash 1 data by squares, Wash 2 data by up triangles, and Wash 3 data by down triangles. Error bars represent 1 S.D. from the mean of three replicates. If error bars are not visible, they are smaller than the symbol size. The solid line represents the fit of the Langmuir adsorption isotherm.

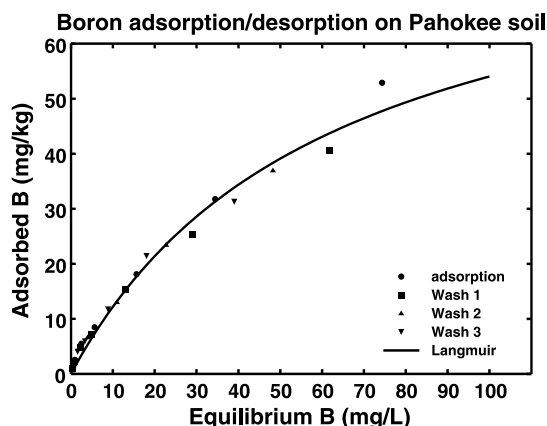


FIG. 4. Boron adsorption-desorption as a function of solution B concentration for Pahokee soil. Adsorption data are represented by circles, Wash 1 data by squares, Wash 2 data by up triangles, and Wash 3 data by down triangles. The solid line represents the fit of the Langmuir adsorption isotherm.

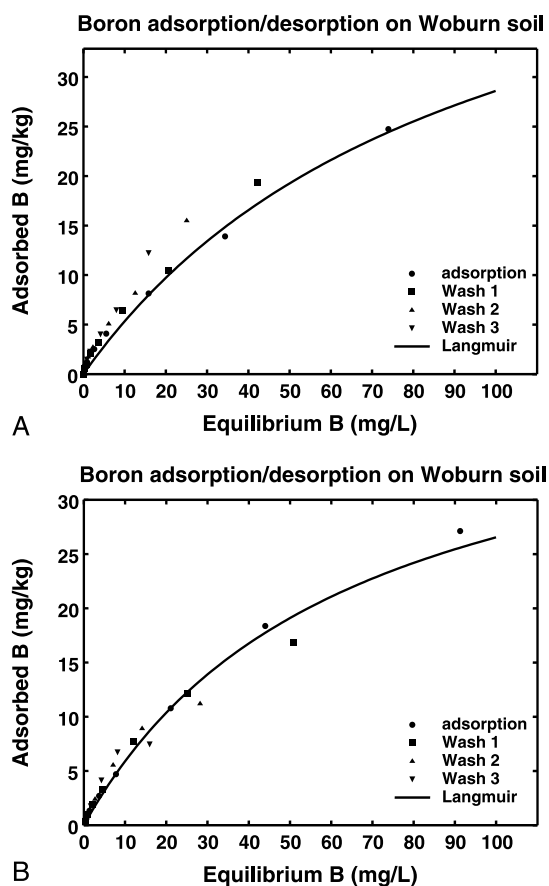


FIG. 5. Boron adsorption-desorption as a function of solution B concentration for Woburn soil: (A) soil was clumping during the experiment; (B) soil was completely dispersed during the experiment. Adsorption data are represented by circles, Wash 1 data by squares, Wash 2 data by up triangles, and Wash 3 data by down triangles. The solid line represents the fit of the Langmuir adsorption isotherm.

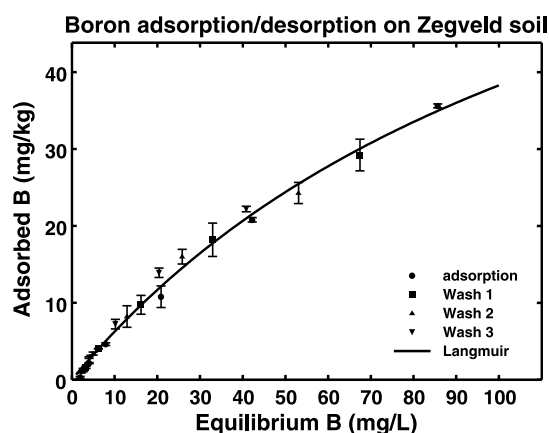


FIG. 6. Boron adsorption-desorption as a function of solution B concentration for Zegveld soil. Adsorption data are represented by circles, Wash 1 data by squares, Wash 2 data by up triangles, and Wash 3 data by down triangles. Error bars represent 1 S.D. from the mean of two replicates. If error bars are not visible, they are smaller than the symbol size. The solid line represents the fit of the Langmuir adsorption isotherm.

TABLE 2. Langmuir Adsorption Isotherm Parameters for the Soils

Soil	K	Maximum Adsorption	R^2
Hanford	0.0102	29.0 ± 3.08	0.996**
Glendale	0.0117	65.1 ± 7.95	0.996**
Elliott	0.0280	25.1 ± 1.69	0.994**
Pahokee	0.0131	106 ± 10.8	0.996**
Woburn	0.0106	43.5 ± 3.56	0.980**
Zegveld	0.00157	88.9 ± 14.7	0.980**

**Significant at the 95% level of confidence.

previously found hysteretic B desorption behavior for this soil. The 0.01 M CaCl₂ background electrolyte solution used in the adsorption-desorption experiments of Elrashidi and O'Connor (1982) would have precipitated additional calcium carbonate and could have trapped some B irreversibly in this calcareous soil (Fine-silty, mixed, superactive, calcareous, thermic Typic Torrifluent). In addition, the CaCl₂ treatment would have removed alkalinity and lowered the pH. Elrashidi and O'Connor (1982) observed a pH value of 7.6 in CaCl₂ solution compared with our pH value of 8.0 in NaCl solution. We did not observe hysteresis in a background electrolyte of 0.01 M NaCl. Our result is in contrast to that of Majidi et al. (2010), who found adsorption hysteresis in calcareous soils at low equilibrium B concentration.

As indicated in the previous paragraph, apparent B hysteresis might result from the experimental procedure itself. During the course of the initial experiment on the Woburn soil, we noticed that the soil was clumping at the bottom of the centrifuge tubes. It is likely that the clumping resulted in inaccessibility of some of the adsorbed B to the exchanging (B-free) electrolyte solution, thus resulting in poor equilibration of soil and solution and subsequent apparent hysteresis (Fig. 5A). In fact, after reducing the soil-solution ratio and paying special heed to thoroughly dispersing the soil, nonhysteretic behavior was observed (Fig. 5B). An additional procedural difficulty that might result in apparent B hysteresis is the loss of some soil particles in the supernatant during the washing steps.

These results do not support the hypothesis that B desorption hysteresis increases with increasing soil organic matter content. All of the soils examined in this study ranging in organic matter content from 0.7% for the Hanford soil to 47% for the Pahokee soil were found to be nonhysteretic. Clearly, if hysteretic behavior occurs for B adsorption-desorption, it would be due to a soil property other than organic matter content.

Boron desorption hysteresis was also evaluated by measuring the B released in the long-term B incubation experiment. Figures 7–9 depict B desorption as measured by the three extractants: (i) hot water, (ii) DTPA-sorbitol, and (iii) 0.1 M NaCl. Comparison between the three extractants revealed no statistically significant differences at the 95% level of confidence in amounts of B released for the Hanford and the Woburn soils after 1 month. For the Woburn soil after 11 months and the Zegveld soil, there were no statistically significant differences at the 95% level of confidence between the amounts of B released using hot water and 0.1 M NaCl, but the amounts of B released by DTPA-sorbitol were statistically significantly smaller than by the other two extractants at the 95% level of confidence.

For the Hanford soil, B releases after 11 and 23 months were not statistically significantly different at the 95% level of confidence relative to 1 month for all three extractants (Fig. 7). This

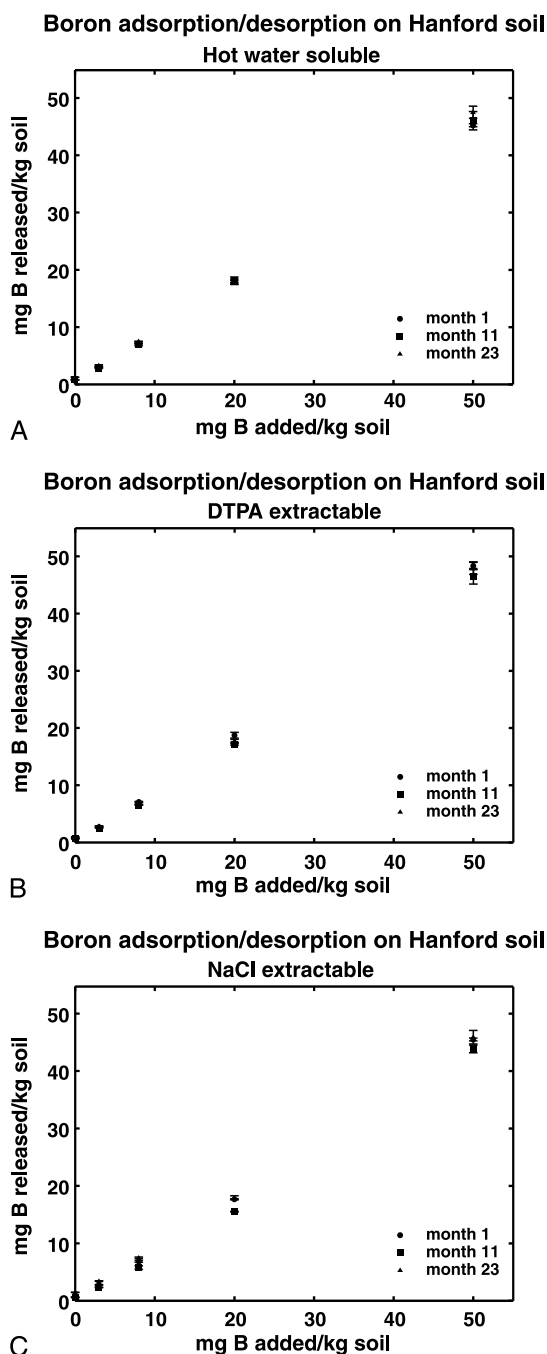


FIG. 7. Boron release as a function of B addition and incubation time from Hanford soil using (A) hot water, (B) DTPA-sorbitol, and (C) 0.1 M NaCl. Month 1 data are represented by circles, month 11 data by squares, and month 23 data by triangles. Error bars represent 1 S.D. from the mean of three replicates. If error bars are not visible, they are smaller than the symbol size.

observation agrees with the results of the B adsorption-desorption isotherm experiment described above for this soil. For the Woburn soil, B release was statistically significantly lower at the 95% level of confidence after 17 months than after 1 month of reaction time for all three extractants (Fig. 8). For the Zegveld soil, B release was statistically significantly lower at the 95% level of

confidence after 17 months than after 1 month of reaction time for DTPA-sorbitol extractable and 0.1 M NaCl extractable B. The hot water-soluble B for the Zegveld soil was not statistically significantly different at the 95% level of confidence after

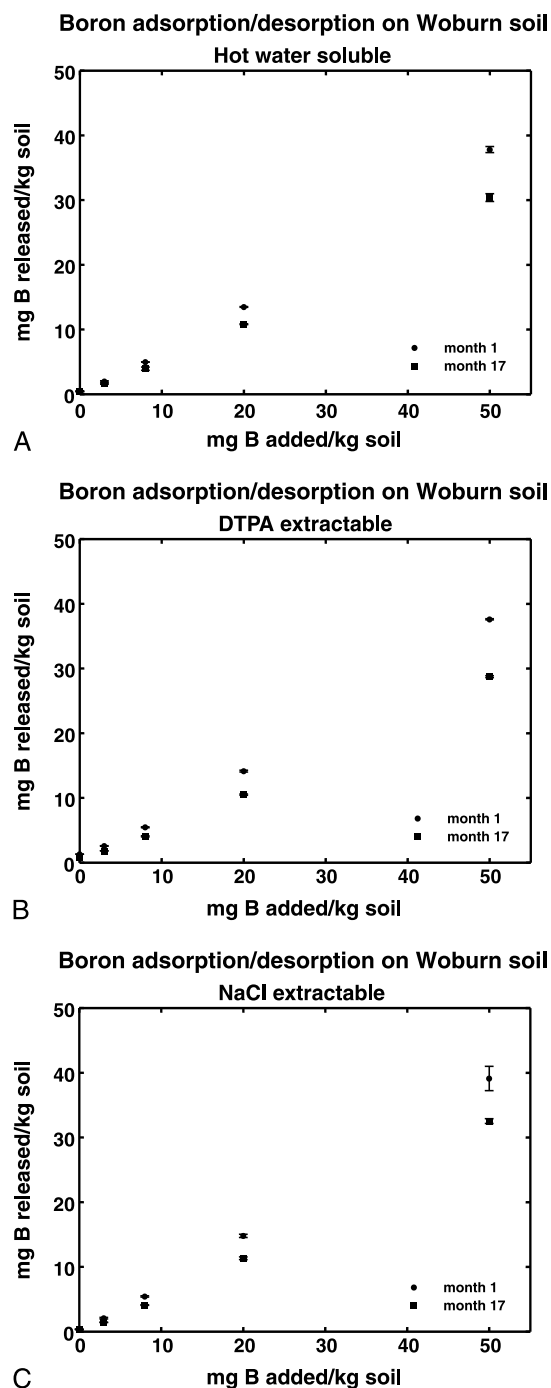


FIG. 8. Boron release as a function of B addition and incubation time from Woburn soil using (A) hot water, (B) DTPA-sorbitol, and (C) 0.1 M NaCl. Month 1 data are represented by circles, and month 17 data by squares. Error bars represent 1 S.D. from the mean of three replicates. If error bars are not visible, they are smaller than the symbol size.

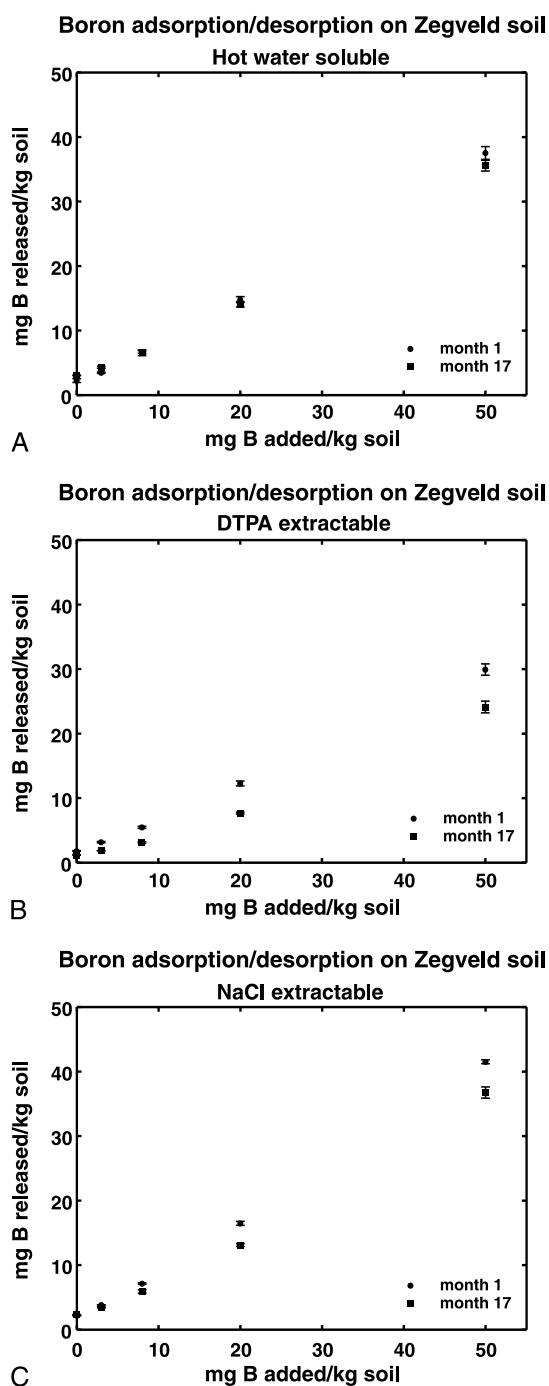


FIG. 9. Boron release as a function of B addition and incubation time from Zegveld soil using (A) hot water, (B) DTPA-sorbitol, and (C) 0.1 M NaCl. Month 1 data are represented by circles and month 17 data by squares. Error bars represent 1 S.D. from the mean of three replicates. If error bars are not visible, they are smaller than the symbol size.

17 months than after 1 month of reaction time, in agreement with the results of the B adsorption-desorption isotherm experiment.

CONCLUSIONS

Our results do not support the hypothesis that B desorption hysteresis is controlled by soil organic matter content since we

observed no hysteresis on the Pahokee soil containing almost 50% organic matter. Our short-term results also do not support the hypothesis that B desorption hysteresis is greatest at lowest soil pH. Our two highly acid soils, Zegveld and Pahokee, exhibited no hysteresis. The Woburn (pH 6.7) and Zegveld (pH 4.7) soils, both having a pH below neutral, were hysteretic in the long-term experiment in contrast to the Hanford soil having a higher pH (7.4), which was not. Our long-term results are in agreement with those of Chen et al. (2009), who found increasing hysteresis with decreasing soil pH. Hysteretic behavior can result from the experimental procedure itself. Careful attention to a standardized adsorption-desorption protocol that avoids clumping and soil loss is necessary.

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Role of Organic Matter on Boron Adsorption-Desorption Hysteresis of Soils: Erratum

In the article that appeared on page 417 of the July issue, Table 1 was reproduced incorrectly. The correct table is shown below. The publisher regrets the error.

TABLE 1. Chemical and Physical Characteristics and Classifications of Soils

Soil Series								
Classification and Clay Mineralogy	Depth (cm)	Clay (%)	pH	SA $m^2 g^{-1}$	IOC	OC	Fe	Al
					------%-----			
Hanford—Typic Xerorthent montmorillonite, illite, kaolinite	0–10	9.6	7.4	17.5	0.00525	0.72	0.489	0.0314
Glendale—Typic Torrifluvent montmorillonite, illite, kaolinite	Surface	20.0	8.0	98.4	0.33	1.06	0.786	0.0489
Elliott—Aquic Argiudoll illite, kaolinite, montmorillonite	0–15	23.0	6.9	51.6	<0.001	2.93	1.10	0.127
Woburn—Dystric Cambisol* montmorillonite, chlorite, kaolinite, illite	0–15	27.0	6.7	108	0.022	3.21	2.77	0.0818
Pahoee—Lithic Haplosaprist kaolinite, vermiculite	0–15	9.3	5.0	117	<0.001	47.0	1.08	0.178
Zegveld—Histosol* illite, kaolinite, vermiculite	0–15	20.0	4.7	12.2	0.013	29.1	0.881	0.251

*European Soil Classification System.

REFERENCE

Goldberg S, and D. L. Suarez. 2012. Role of Organic Matter on Boron Adsorption-Desorption Hysteresis of Soils. *Soil Sci.* 177:417–423.