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100 Years on Boron Research in Plants – Research article

Boron rates and residual effect of gypsum on soil chemical attributes and barley yield

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ABSTRACT: Studies on barley management practices are scarce, and farmers have employed boron (B) fertilization without technical criteria, relying primarily on field reports of yield gains. This study aimed to assess the efficacy of B fertilization of barley cultivars (cv.) under the residual effect of phosphogypsum. The experiment was started in 2009 using a randomized block design, with rates of 0, 3, 6, 9, and 12 $Mg_{[gypsum]}$ ha^{-1} for a long-term study. The plots were divided into four sections in the 2021 and 2022 winter seasons, each receiving a different B rate: 0, 1, 2, and 4 $kg_{[B]}$ ha^{-1} as boric acid (H_3BO_3). The barley cv. Danielle and Imperatriz were then sown in these sections. The data were analyzed using a split-split plot design. The yield components and grain yield were evaluated in Nov 2021 and 2022, while the soil was sampled following the barley harvest in 2022. The residual effects of gypsum on the evaluated soil chemical properties were nearly negligible, except for the yield of cv. Imperatriz, which exhibited an increase with gypsum rates in 2021, while no yield response was observed for cv. Danielle (2021 and 2022). The B fertilization increased soil-B availability and the yield of cv. Danielle in 2021 and 2022, while cv. Imperatriz exhibited an increase in yield only in the 2021 season. The cv. Imperatriz exhibited a higher yield than cv. Danielle in 2021 and 2022 seasons, with an increase of 18 % on average. The increases in soil B content (0.37 - 0.45 $mg_{[B]}$ dm^{-3}) and in grain yield as a result of B fertilization of barley cv. Danielle (+17 – 87 %) and Imperatriz (up to +6 %) indicate that the current critical soil B level (0.3 $mg_{[B]}$ dm^{-3}) should be reevaluated in the southern states in Brazil.

Keywords: Boric acid, CaSO4, critical level of boron, no-till

Introduction

Barley (*Hordeum vulgare* L.) is the fourth most cultivated cereal in the world, utilized both for human and animal consumption as well as in malt production (Nouri et al., 2019). Brazil is not self-sufficient in barley production. The industrial demand for barley grains exceeds 1.3 million tons for malt production (Mori and Minella, 2012), while 391 thousand tons (30 %) were harvested in 2023 (CONAB, 2024). The grain must be free of mycotoxins for malt production, exhibit 95 % germination, and contain 9.5 - 12 % protein (Geng et al., 2022). However, not all national production meets these requirements.

The national average barley yield was 2.9 t ha^{-1} on the 2023 harvest (CONAB, 2024). It is still possible to increase barley yield and production by adjusting plant nutrition. Boron (B) deficiency in barley has been reported in more than 80 countries, indicating that the germplasm is not adapted to soils with low B levels (Rerkasem et al., 2020). The deficiency symptoms include decreased spikelets, increased sterility, and low yield (Rerkasem et al., 2020). Toxic B levels are also linked to necrosis and low yield (Chen et al., 2021).

B leaches in the soil, and the soil critical level is set at 0.3 $mg_{[B]}$ dm^{-3} in southern Brazilian states (SBCS/NRS, 2016; SBCS/NEPAR, 2017), and up to 0.6 $mg_{[B]}$ dm^{-3} in other states (Cantarella et al., 2022). The proximity of deficient and toxic B levels presents a challenge for formulating fertilization recommendations, so the supply of B to crops is often reduced in relation to other nutrients.

Given the low B mobility in the phloem, fertilization is more effectively achieved via soil (Oliveira Neto et al., 2009). This approach has been demonstrated to reduce flower sterility in barley and enhance yield (Yau, 2010; Brdar-Jokanović, 2020), as well as increase ear length and number and weight of grains (Rehman et al., 2022).

In tropical and subtropical regions, soil profiles are typically deficient in B and other essential nutrients. The application of lime and/or gypsum is necessary to address soil acidity, and these materials serve as the primary sources of calcium (Ca^{2+}) to agricultural fields. The roles of Ca and B are related in plant nutrition, as these elements have been demonstrated to influence meristem integrity (Pereira et al., 2021), cell division and growth (Marschner et al., 2012), and flowering and fructification (Brdar-Jokanović, 2020). In light of these considerations, two questions are postulated: i) are the residual effects of gypsum and B fertilization relevant for increased barley grain yield? and ii) how accurate is the current soil B critical level established for barley? The objective of this study is to evaluate the impact of B fertilization on barley cv. in an experimental site under the residual effect of gypsum rates.

Materials and Methods

Study site

The experiment was conducted in the municipality of Guarapuava, Paraná state, Brazil (25°23′ S, 51°30′ W, altitude 1,026 m) under a mesothermic subtropical wet climate (Cfb) as defined by Alvares et al. (2013). According to the Brazilian Soil Classification System, the soil is a very clayey Typic Hapludox (Soil Survey Staff, 2014) or *Latossolo Bruno distrófico*. The soil was sampled in 2009 and characterized (Table 1) before the beginning of a long-term phosphogypsum (gypsum) trial (Michalovicz et al., 2014, 2019), in a land that was first subjected to the no-till crop system in 2003.

A soybean (*Glycine max* L.) black oat (*Avena strigosa* Schreb.) soybean succession was planted from the summer season of 2019/2020 to the summer season of 2020/2021. Prior to commencing the barley-soybean-barley succession for this study in Apr 2021, the soil was sampled and analyzed. In May 2021, 5.3 Mg ha^{-1} of dolomitic lime (relative neutralizing value 95 %) were broadcast on the soil surface with the objective of raising the base saturation to 70 %, as recommended for barley (SBCS/NEPAR, 2017). The mean initial B content (0.0 - 0.2 m) was 0.31 mg dm^{-3}. The chemical attributes of the soil prior to this study have been previously published by Souza et al. (2022) and Umburanas et al. (2023).

Experimental Design and Treatments

A randomized block design was employed, in a split-split plot scheme (5 × 4 × 2) with four replications. Five gypsum rates were initially applied to plots measuring 16 × 6.4 m: 0 (control), 3, 6, 9, and 12 $Mg_{[gypsum]}$ ha^{-1}. Gypsum was broadcast on the soil surface in Nov 2009, Nov 2010, and Nov 2011, at a rate of one-third of the total amount each year after the sowing of summer crops. In the winter seasons of 2021 and 2022, four B rates were applied in split-plots (4 × 6.4 m) with the following treatments: 0 (control), 1, 2, and 4 $kg_{[B]}$ ha^{-1}. Split-split plots (2 × 6.4 m) were utilized to examine the effects on two barley cv. The cv. Danielle and Imperatriz were used. Cv. Danielle has its origin from strains in Europe, where it has been adapted to temperate conditions and soils with good B availability. In contrast, cv. Imperatriz has its origin from a germplasm breeding in Brazil, where it has been adapted to a subtropical climate and more weathered soils, which are prone to B limitation (Schnurbusch et al., 2010). To apply the B rates, the required boric acid rate (H_3BO_3, containing 16.8 % of water-soluble B) was dissolved in 10 L of deionized water and applied to the soil surface during the pre-tillering stage of barley.

Barley cultivation and evaluation

The crop was sown in June 2021 and 2022, with a distance of 0.17 m between rows and an intended plant density of 250 plants m^{-2}. The fertilization regimen consisted of 80 kg ha^{-1} of P_2O_5 (triple superphosphate) applied in-furrow, and 60 kg ha^{-1} of K_2O (potassium chloride) plus 40 kg ha^{-1} of N (urea) broadcast on the soil surface at the crop's pre-emergence stage. Additionally, 100 kg ha^{-1} N (urea) was applied as a top dressing at the tillering stage, which corresponds to stage 2 of the Feeks scale (Large, 1954). The management of pests, weeds, and diseases was effectively addressed.

The barley yield evaluation was conducted at the physiological maturity stage 11 of the Feeks scale (Large, 1954), utilizing a plot combine harvester with a 1.7-meter-wide platform (10 barley rows) across a 5-m linear distance in the central region of each experimental unit (8.5 m^2 unit^{-1}). The moisture content of the grains was adjusted to 130 g kg^{-1}. Grain subsamples from each experimental unit were utilized to determine the 1000-grain weight in the 2021 and 2022 growing seasons and the hectoliter weight (HW) in the 2022 season.

Soil sampling and analysis

Following the barley harvest in 2022, soil samples were collected using a Dutch auger at three points per split-plot. Composite samples were formed for depths ranging from 0.0 to 0.1, 0.1 to 0.2, 0.2 to 0.4, 0.4 to 0.6, and 0.6 to 0.8 m in the soil profile. Subsequently, the samples were oven-dried (40 °C), crushed using a knife mill, and sieved through a 2 mm mesh. The pH was determined in 0.01 mol L^{-1} $CaCl_2$, and the potential acidity (H + Al) was determined by the Shoemaker-McLean-Pratt buffer. Exchangeable Al^{3+}, Ca^{2+}, and Mg^{2+} were extracted in 1 mol L^{-1} KCl and quantified through titration (Al^{3+}) and atomic absorption spectrometry (Ca^{2+} and Mg^{2+}). The soil B was extracted using barium chloride (1.25 g L^{-1}) in hot water, filtered, and then determined using spectrophotometry with azomethine-H (van Raij et al., 2001).

Table 1 – Soil chemical attributes and clay content of the Typic Hapludox (0.0 - 0.2 m) from the experimental field in 2009 prior to the beginning of this long-term experiment.

Depth	P (Mehlich-1)	OM	pH	Al^{3+}	H + Al	Ca^{2+}	Mg^{2+}	K^+	CEC	BS	Clay
m	mg dm^{-3}	g dm^{-3}	$CaCl_2$	------------	------------	cmol$_c$ dm^{-3}	------------	------------	------------	%	g kg^{-1}
0.0 - 0.1	20.7	49.8	5.3	0.0	4.8	4.9	2.6	0.3	12.6	61.8	720
0.1 - 0.2	19.7	49.5	5.3	0.0	5.1	4.5	2.4	0.3	12.3	58.3	720

OM = organic matter; CEC = cation exchange capacity at pH 7.0; BS = base saturation.

Meteorological data

The mean annual precipitation and temperature were 1,920 mm and 16.8 °C, respectively (IDR-PR, 2023). For the duration of the study, meteorological data, including precipitation and temperature (Figure 1), were collected from a distance of approximately 200 m from the experimental site by an official state meteorological station (SIMEPAR/Brazil) (25°21′ S, 51°30′ W, altitude 1,058 m).

At the beginning of the 2021 crop season, three frost events occurred: June 29th to 31st, July 19th to 21st, and July 28th to 30th, with temperatures reaching as low as −7.5 °C (Figure 1A). These frosts impaired and delayed early growth and development. Nevertheless, the crop was able to recover its growth during the tillering phase. The minimum, maximum, and average daily temperatures throughout the season were 10.7 °C, 22.9 °C, and 16.2 °C, respectively. These values align closely with the climatic norms for the same period, which are 11.0 °C, 22.1 °C, and 15.6 °C (IDR-PR, 2023). The precipitation levels were distributed in a relative uniform manner throughout the 2021 season, with a total of 749 mm recorded (Figure 1B).

In 2022, frost occurred on July 26th and Aug 22nd, yet minimal damage was inflicted to the crop, in contrast to the effects observed in 2021. From August onwards, multiple consecutive rainy periods resulted in an accumulated rainfall of 1,070 mm for the 2022 season (Figure 1D), with extended cloudy conditions. Effective pest and disease management strategies were employed.

Statistical Analysis

The results were initially evaluated using the Bartlett's test for homogeneity of variance, followed by analysis of variance (ANOVA) and regression analysis for quantitative variables. In instances where the ANOVA revealed significant effect of the treatments, the means were compared using the Tukey's test (α = 0.05), which was employed to elucidate instances of interaction between treatments. Regression models were selected based on the highest coefficient of determination, deemed significant at $p < 0.05$.

Results

Soil chemical attributes

The soil contents of Al^{3+} (Figure 2A and B) and Ca^{2+} (Figure 2C and D) in 2022 remained unaffected by the residual effect of gypsum or by different B application rates (Table 2). The 0.1 - 0.2 m layer exhibited elevated levels of Al^{3+}, while subsurface soil layers demonstrated diminished levels of Ca^{2+}. Nevertheless, Ca^{2+} levels within the 0.0 - 0.1 and 0.1 - 0.2 m layers remained above the critical level, in accordance with the official recommendation for Paraná state (SBCS/NEPAR, 2017).

Table 2 – Significance (p-values) from the analysis of variance of aluminum (Al^{3+}), calcium (Ca^{2+}), magnesium (Mg^{2+}), potassium (K^+), phosphorus (P) and boron (B) soil levels in response to the residual effect of gypsum rates (applied between Nov 2009 and Nov 2011) and boron rates (applied in July 2021 and July 2022), at the end of the 2022 barley season, in a Typic Hapludox under no-till in Guarapuava, Paraná state, Brazil.

Treatment	Soil Elements					
	Al^{3+}	Ca^{2+}	Mg^{2+}	K^+	P	B
Soil Depth: 0.0 - 0.1 m						
Gypsum (G)	0.455^{ns}	0.745^{ns}	0.446^{ns}	0.774^{ns}	0.609^{ns}	0.733^{ns}
Boron (B)	0.058^{ns}	0.731^{ns}	0.976^{ns}	0.984^{ns}	0.179^{ns}	**0.0001***
G × B	0.083^{ns}	0.935^{ns}	0.476^{ns}	0.129^{ns}	0.931^{ns}	0.895^{ns}
Soil Depth: 0.1 - 0.2 m						
G	0.399^{ns}	0.258^{ns}	0.835^{ns}	0.703^{ns}	0.175^{ns}	0.205^{ns}
B	0.560^{ns}	0.380^{ns}	0.539^{ns}	0.344^{ns}	0.819^{ns}	**0.0001***
G × B	0.169^{ns}	0.979^{ns}	0.122^{ns}	0.146^{ns}	0.811^{ns}	0.182^{ns}
Soil Depth: 0.2 - 0.4 m						
G	0.309^{ns}	0.735^{ns}	0.278^{ns}	0.354^{ns}	0.391^{ns}	0.990^{ns}
B	0.434^{ns}	0.731^{ns}	0.287^{ns}	0.566^{ns}	0.091^{ns}	0.546^{ns}
G × B	0.855^{ns}	0.935^{ns}	0.392^{ns}	0.421^{ns}	0.546^{ns}	0.504^{ns}
Soil Depth: 0.4 - 0.6 m						
G	0.872^{ns}	0.258^{ns}	0.158^{ns}	**0.021***	0.370^{ns}	**0.0076****
B	0.273^{ns}	0.380^{ns}	0.096^{ns}	0.920^{ns}	0.797^{ns}	0.747^{ns}
G × B	0.066^{ns}	0.979^{ns}	0.162^{ns}	0.876^{ns}	0.304^{ns}	0.098^{ns}
Soil Depth: 0.6 - 0.8 m						
G	0.769^{ns}	0.270^{ns}	0.220^{ns}	0.478^{ns}	0.277^{ns}	0.090^{ns}
B	0.074^{ns}	0.444^{ns}	0.106^{ns}	0.443^{ns}	0.454^{ns}	0.911^{ns}
G × B	0.104^{ns}	0.786^{ns}	0.061^{ns}	0.374^{ns}	0.126^{ns}	0.895^{ns}

*, **, ***, and ns denote significant values at $p \leq 0.05$, $p \leq 0.01$, $p \leq 0.001$ and non-significant values, respectively; bold-type numbers are $p \leq 0.05$.

The treatments had no discernible impact on soil Mg^{2+} levels (Table 2, Figure 3A and B), which generally remained above the critical level of 1.0 $cmol_c$ dm^{-3} (SBCS/NEPAR, 2017), except the 0.4 - 0.6 and 0.6 - 0.8 m layers for the 6 $Mg_{[gypsum]}$ ha^{-1} treatment.

With regard to soil K^+ contents, either the effect of B rates (Figure 3D) nor interaction effects were observed (Table 2). However, a residual effect was evident for gypsum in the 0.4 - 0.6 m layer (Figure 3C), where higher K^+ levels were observed with 6, 9, and 12 $Mg_{[gypsum]}$ ha^{-1} compared to the control without gypsum.

The application of B rates and gypsum did not result in any discernible effects on soil P levels (Figure 4A and B). They remained above the critical level of 6 mg dm^{-3} for Paraná state (SBCS/NEPAR, 2017) only in the 0.0 - 0.1 m layer, with a sharp decline observed in the 0.1 - 0.2 m layer, which is a common phenomenon in no-till areas.

Applying gypsum did not affect soil B contents at 0.0 - 0.1, 0.1 - 0.2 m layers (Figure 4C). However, the B rates had an impact on soil B availability (Figure 4D). In the 0.0-0.1 m layer, 4 $kg_{[B]}$ ha^{-1} treatment resulted in a higher B level compared to the control with no B application. In contrast, at 0.1 - 0.2 m, the 4 $kg_{[B]}$ ha^{-1} treatment resulted in a higher B content than the 1 and 2 $kg_{[B]}$ ha^{-1} treatments, which also increased soil B availability compared to the control.

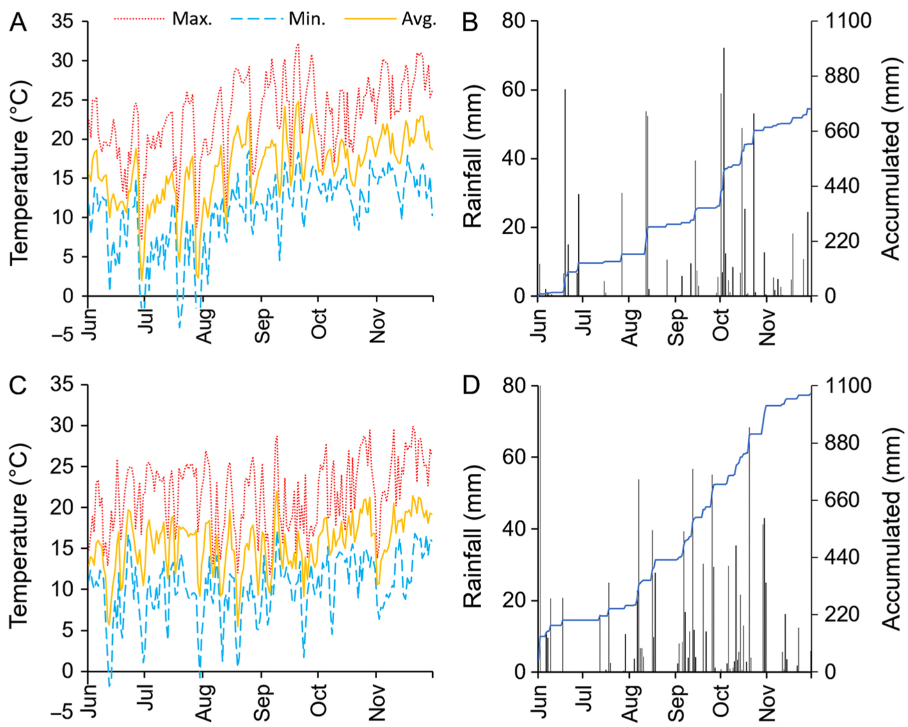

Figure 1 – A and B) Observed daily temperature and monthly (black bars) and cumulative (blue lines) rainfall at the experimental site during the winter crop seasons of 2021, and C and D) 2022 in Guarapuava, Paraná state, Brazil. Max. = maximum; Min. = minimum; Avg. = average.

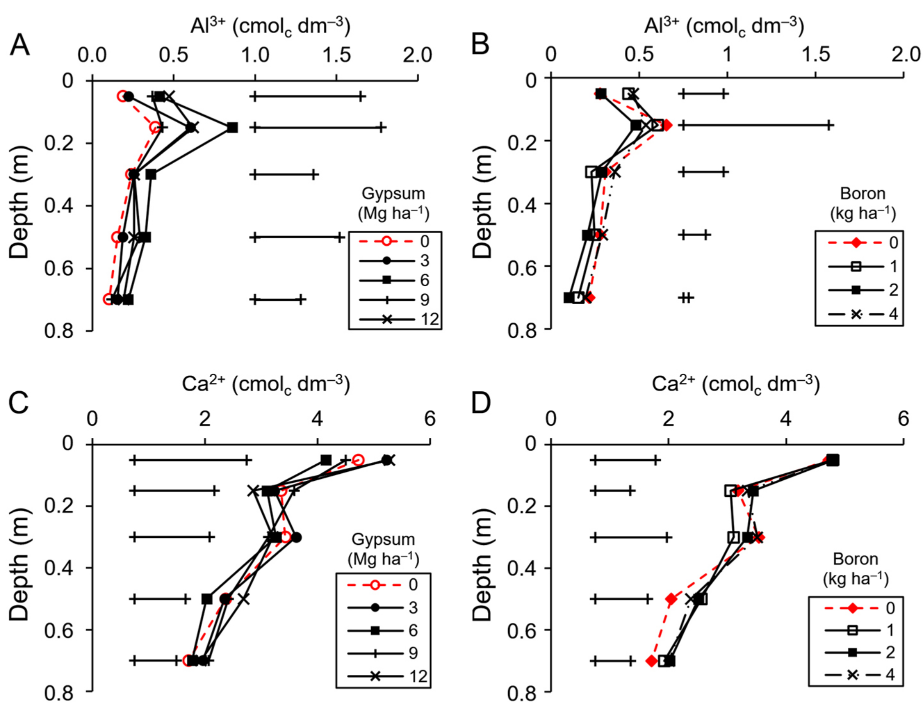

Figure 2 – A and B) Soil contents of aluminum (Al^{3+}) and C and D) calcium (Ca^{2+}) in 2022, under residual effects of gypsum rates applied in 2009 and boron rates applied at the pre-tillering stage of barley in the winter seasons of 2021 and 2022 in Guarapuava, Paraná state, Brazil. Horizontal bars are the minimum significant difference obtained from Tukey's test at 5 % probability of error.

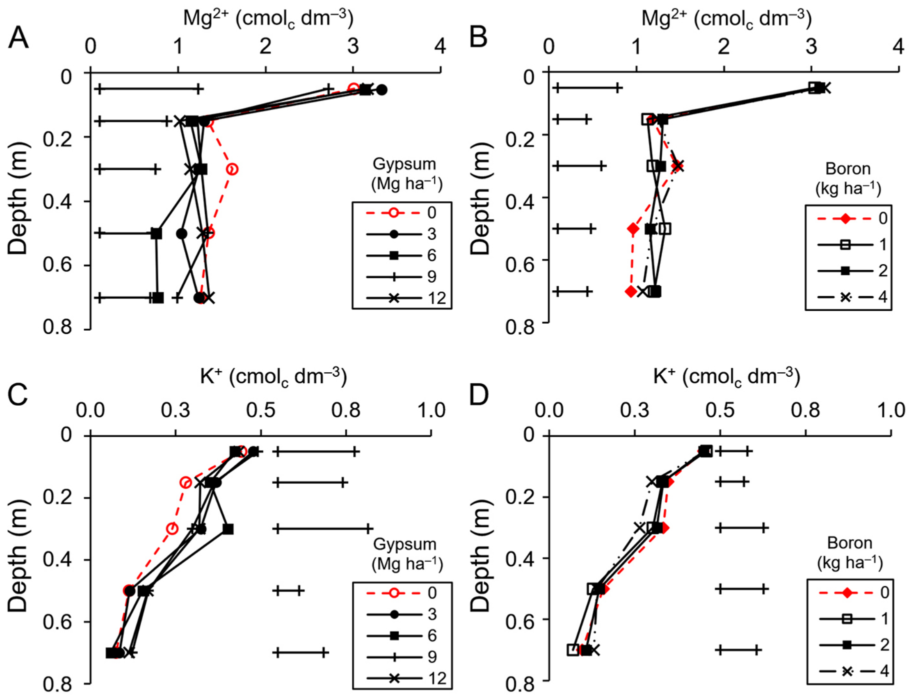

Figure 3 – A and B) Soil contents of magnesium (Mg^{2+}) and C and D) potassium (K^{+}) in 2022, under residual effects of gypsum rates applied in 2009 and boron rates applied at the pre-tillering stage of barley in the winter seasons of 2021 and 2022 in Guarapuava, Paraná state, Brazil. Horizontal bars are the minimum significant difference obtained from Tukey's test at 5 % probability of error.

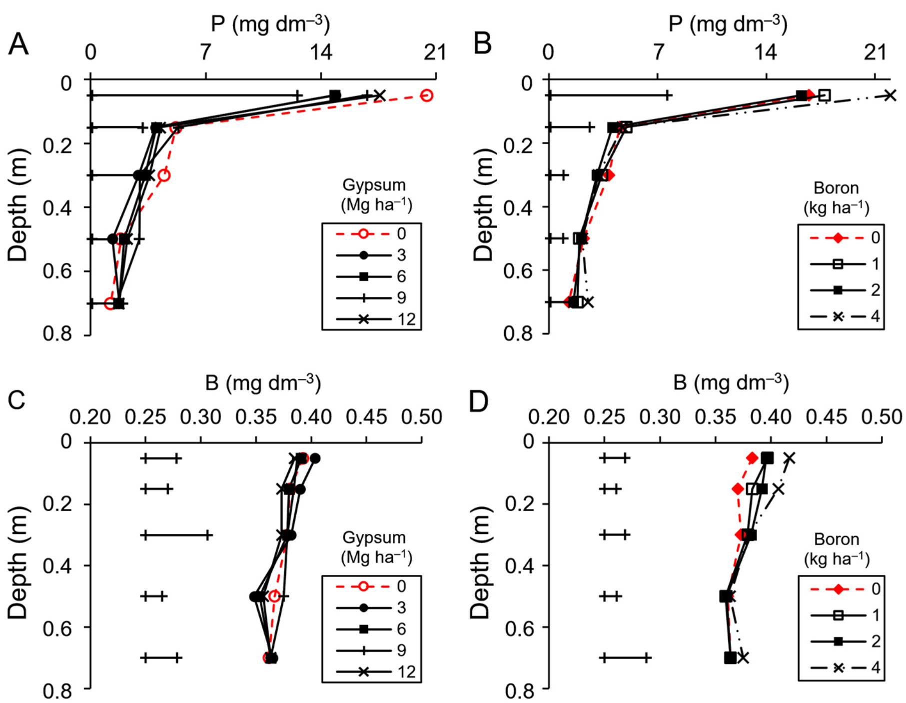

Figure 4 – A and B) Soil contents of phosphorus (P) and C and D) boron (B) in 2022, under residual effects of gypsum rates applied in 2009 and boron rates applied at the pre-tillering stage of barley in the winter seasons of 2021 and 2022 in Guarapuava, Paraná state, Brazil. Horizontal bars are the minimum significant difference obtained from Tukey's test at 5 % probability of error.

Table 3 – The *p*-value, significance, and coefficient of variation (CV) for barley grain yield and 1000-grain weight (1000-GW) in 2021 and 2022 crop season and hectoliter weight (HW) in 2022 crop season and regression models between each evaluated attribute.

Source of variation	2021		2022		
	Grain yield	1000-GW	Grain yield	1000-GW	HW
Gypsum (G)	ns[1]	0.001***	ns	ns	ns
Boron (B)	<0.001***	0.030*	ns	ns	ns
G × B	<0.001***	ns	ns	ns	ns
Cultivar (C)	<0.001***	0.002**	0.016*	0.008**	0.006**
C × G	0.006**	ns	ns	ns	0.040*
C × B	<0.001***	0.004**	0.001***	ns	ns
C × B × G	ns	ns	ns	ns	ns
$CV_{[Gypsum]}$ (%)	19	7	26	6	2
$CV_{[Boron]}$ (%)	14	6	18	7	3
$CV_{[Cultivar]}$ (%)	11	6	13	7	2

Factor		Regression model	R^2
Grain yield (2021) C × B	Imperatriz	$y = -0.068752x^2 + 0.26196x + 4.8830$	0.71*
	Danielle	$y = -0.28485x^2 + 1.6254x + 2.4745$	0.90**
Grain yield (2021) G × B	0 Mg ha^{-1}	ns	-
	3 Mg ha^{-1}	$y = -0.23683x^2 + 1.3553x + 3.160$	0.85**
	6 Mg ha^{-1}	$y = -0.24146x^2 + 1.1911x + 3.535$	0.97**
	9 Mg ha^{-1}	$y = -0.17726x^2 + 1.006x + 3.6858$	0.97**
	12 Mg ha^{-1}	$y = -0.18448x^2 + 1.0464x + 3.562$	0.99**
Grain yield (2021) C × G	Imperatriz	$y = 34.917x + 4{,}771$	0.93**
	Danielle	ns	-
1000-GW (2021) C × B	Imperatriz	ns	-
	Danielle	$y = -0.9563x + 47.416$	0.94**
1000-GW (2021) C × G	Imperatriz	$y = -0.0719x^2 + 1.0829x + 41.726$	0.74**
	Danielle	ns	-

*, **, ***, and ns denote significant values at $p \leq 0.05$, $p \leq 0.01$, $p \leq 0.001$ and non-significant values, respectively.

Barley grain yield and yield components - crop season of 2021

The barley grain yield in 2021 demonstrated the following interactions between treatments: B rates × cv., B rates × gypsum rates, and gypsum rates × cv. (Table 3). With regard to the grain yield interaction between B rates and cv. (Figure 5A), cv. Danielle exhibited no yield differences between the application rates of 1, 2, and 4 $kg_{[B]}$ ha^{-1} applied at tillering. Nevertheless, all the aforementioned rates yielded higher results (+83 to +96 %) than the control without B application. For cv. Imperatriz, the yield did not vary between 0, 1, and 2 $kg_{[B]}$ ha^{-1} application rates. However, the application of 4 $kg_{[B]}$ ha^{-1} resulted in an 8 % reduction of yield compared to 2 $kg_{[B]}$ ha^{-1}. The effect of B rate on grain yield was quadratic for both cv., with the maximum efficiency rate (MER) estimated at 1.91 $kg_{[B]}$ ha^{-1} for cv. Imperatriz and 2.85 $kg_{[B]}$ ha^{-1} for cv. Danielle.

The interaction of gypsum rates × cv. (Figure 5C) demonstrated that the grain yield of cv. Danielle was decreased at 6 $Mg_{[gypsum]}$ ha^{-1} compared to the control without gypsum, whereas the yield of cv. Imperatriz did not differ between the gypsum rates. No regression effect of gypsum rate was observed for cv. Danielle. However, the yield of cv. Imperatriz increased linearly with the gypsum rates, reaching an 8.7 % increase in yield with 12 $Mg_{[gypsum]}$ ha^{-1} compared to the control treatment without gypsum. Overall, grain yield was consistently higher (5 - 30 %) for cv. Imperatriz compared to cv. Danielle across all gypsum and B application rates (Figure 5A and C).

Regarding interaction of B rates × gypsum rates, grain yield increased with the applied B rates within each gypsum rate. This increase was quadratic, with the exception of the control treatment without gypsum (Figure 5B). In the range of 3 to 12 $Mg_{[gypsum]}$ ha^{-1}, the MER of B was between 2.46 and 2.86 $kg_{[B]}$ ha^{-1}. The application of gypsum did not affect yield within each B rate, with except for the control treatment without B, in which higher gypsum rates resulted in a reduction in yield compared to the control treatment without gypsum.

The 2021 crop season data indicate that the 1000-grain weight (1000-GW) was influenced by gypsum rates, B rates, cv. and the interaction between cultivar and B rates (Table 3, Figure 5D). For cv. Danielle, the 1000-GW was observed to be higher in the 0 $kg_{[B]}$ ha^{-1} control treatment compared to the 2 and 4 $kg_{[B]}$ ha^{-1} treatments, while the 1 $kg_{[B]}$ ha^{-1} treatment did not differ from the others. As the rate of B application increased, the 1000-GW decreased linearly, with an 8.6 % reduction observed at 4 $kg_{[B]}$ ha^{-1} compared to the control treatment without B application.

For cv. Imperatriz, the 1000-GW did not differ between B rates (Figure 5D) but increased with gypsum rates compared to the control without gypsum (Figure 5E). The effect was quadratic, and MER was estimated at 7.53 $Mg_{[gypsum]}$ ha^{-1}. Gypsum did not affect 1000-GW for cv. Danielle.

Barley grain yield and yield components - crop season of 2022

In the 2022 crop cycle, grain yield differed between cv. and was also influenced by the interaction between B rates and cv. (Table 3, Figure 6A and B). In 2022, the interaction between B rate and cultivar demonstrated that grain yield for cultivar Danielle was once again lower at the control compared to the 1, 2, and 4 $kg_{[B]}$ ha^{-1} rates, which did not differ. In contrast, for cultivar Imperatriz, B rates did not affect grain yield.

The 1000-GW exhibited differences between cv. (Table 3, Figure 6B) and was 3.1 % higher in the cv. Danielle compared to cv. Imperatriz. The 2022 harvest data revealed an interaction between cv. and gypsum rates with respect to grain HW (Table 3). The HW was observed to be higher in the cv. Danielle, exhibiting a 1.1 % increase compared to the cv. Imperatriz. The HW in the cv. Danielle did not exhibit any difference between the gypsum rates, while in the cv. Imperatriz, the HW was observed to be higher at 0 $Mg_{[gysum]}$ ha^{-1} (control) in comparison to the rate of 6 $Mg_{[gysum]}$ ha^{-1}. However, the HW at the other gypsum rates did not exhibit any notable differences from each other.

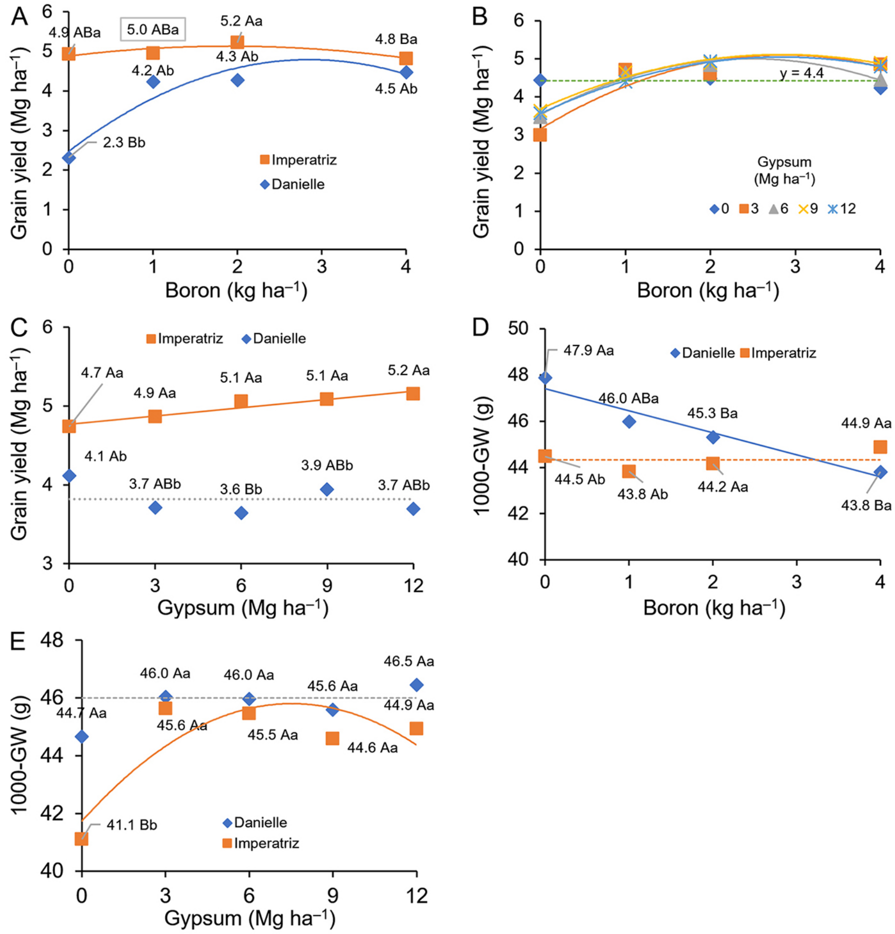

Figure 5 – A) Grain yield of barley as a function of boron rates × cultivars, B) residual effect of gypsum rates × boron rates, and C) gypsum rates × cultivars, D) 1000-grain weight (1000-GW) as a function boron rates × cultivars, and E) cultivars × gypsum rates in 2021 crop season in Guarapuava, Paraná state, Brazil. Capital letters compare rates of boron or rates of gypsum in the same cultivar, and lowercase letters compare cultivar in the same rate of boron or rate of gypsum, using the Tukey's test at 5 % of error probability.

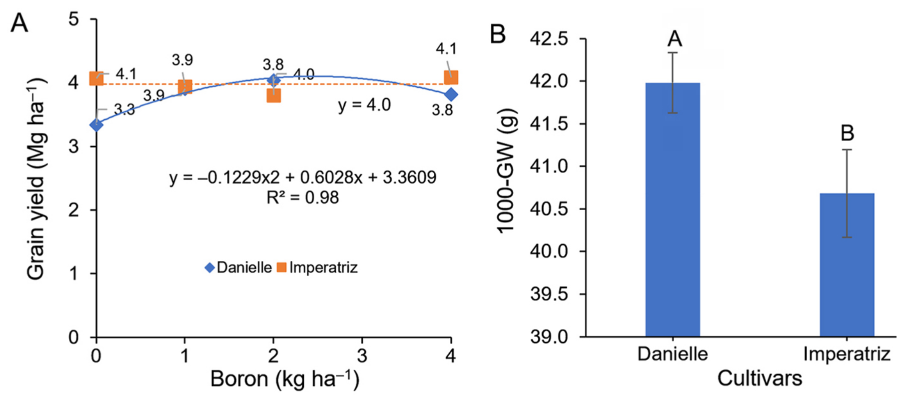

Figure 6 – A) Grain yield of barley as a function of boron rates x cultivars, B) 1000-grain weight (1000-GW) as a function of cultivars in 2022 crop season in Guarapuava, Paraná state, Brazil. Capital letters compare rates of boron or rates of gypsum in the same cultivar, and lowercase letters compare cultivar in the same rate of boron or rate of gypsum, using the Tukey's test at 5 % of error probability.

Discussion

Twelve years after the application of gypsum, its residual effects on soil Al^{3+} levels have dissipated. During the extended period without liming (2009-2021) at the experimental site, coupled with the continued application of N-fertilizers (NPK in the furrow and urea in top-dress) to crops (Michalovicz et al., 2014, 2019; Vicensi et al., 2020a), soil reacidification, plant nutrient uptake, and nitrification processes significantly influenced the comparison of Al^{3+} levels between the residual gypsum treatment and the non-gypsum treatment.

The concentration of Al^{3+} in the 0.1 - 0.2 m layer was found to be higher than that in the 0.0 - 0.1 m layer (Figure 2A and B), which is consistent with soil reacidification and the hypothesis that the surface layer was affected by a recent lime application in May 2021, due to the use of a no-till system (NTS) without mechanical incorporation into the soil.

Furthermore, the gypsum residual effects also ceased on the soil contents of Ca^{2+} (Figure 2C and D) and Mg^{2+} (Figure 3A and B), reinforcing the loss of gypsum effectiveness. The reduction in Ca^{2+} levels in subsurface soil layers may be attributed to: the extended period following gypsum application (2009-2022) and the extraction of previous crops. In contrast, the 0.0 - 0.1 m layer exhibited higher Ca^{2+} levels due to the recent application of lime at the soil surface in 2021. In NTS, the vertical mobility of lime may be limited, leading to concentrated liming effects in the topsoil layer (Crusciol et al., 2016). This phenomenon provides a rationale for the testing and use of gypsum in NTS to manage soil acidity, given the prevalence of NTS in South America.

The elevated Mg^{2+} levels observed in the 0.0 - 0.1 m soil layer can be attributed to the recent surface liming process, which involved the use of a dolomitic material. The Mg^{2+} levels decreased sharply from the 0.0 - 0.1 m layer to the 0.1 - 0.2 m layer. This phenomenon can be attributed to the low vertical mobility of lime in the soil and the historical leaching of Mg^{2+} through the profile as a long-term effect of gypsum application. At the 0.2 - 0.4 m layer, slightly elevated Mg^{2+} levels in the control compared to the gypsum rates (Figure 3A), even without significance in 2022, illustrate this leaching effect in the experiment as previously observed by Michalovicz et al. (2014, 2019) and Vicensi et al. (2020b) with significance in past years.

Furthermore, the lower yield of Poaceous crops observed in the control without gypsum in contrast to the yields observed with gypsum rates of 3 and 6 $Mg_{[gypsum]}$ ha^{-1} in the initial years and 6 and 9 $Mg_{[gypsum]}$ ha^{-1} in the subsequent years of this long-term experiment (Michalovicz et al., 2014, 2019; Vicensi et al., 2020a), revealed a reduction in nutrient exportation in the control without gypsum application, which in turn resulted in elevated Mg^{2+} levels in the soil.

In addition to Al^{3+}, the reaction of gypsum can displace ions such as K^+ and Mg^{2+} from the solid phase to the soil solution, where leaching may occur with excessive soil water (Michalovicz et al., 2014, 2019; Vicensi et al., 2020a). The intensity of this impact is contingent upon the gypsum application rate, the volume of precipitation, and the crops cultivated within the production system. Ultimately, leaching may result in these nutrients reaching or falling below the soil critical level, which could impede the yield of subsequent crops (Vicensi et al., 2020b).

In the layers between 0.4 and 0.8 m (Figure 3A), Mg^{2+} remained below the critical level of 1.0 $cmol_c$ dm^{-3} (SBCS/NEPAR, 2017) for the 6 $Mg_{[gypsum]}$ ha^{-1} rate, however, it did not limit barley yield because the diagnostic layer for the crop (0.0 - 0.2 m) was well supplied with this nutrient. In this long-term experiment, Mg^{2+} was not resupplied by fertilizers and lime (the primary source of Mg^{2+} for cultivated soils in Brazil) for years as a deliberate decision, with the objective of evaluating the longevity of gypsum effects and to estimate the period for reapplication. The implementation of specific nutrient replenishing and/or liming, in the case of Ca^{2+} and Mg^{2+}, would introduce additional factors of variation and potentially compromise the assessment of gypsum residual effects.

Compared to Mg^{2+}, the impact on soil K^+ availability was less pronounced. However, the application of 12 $Mg_{[gypsum]}$ ha^{-1} resulted in elevated K^+ levels in the 0.4 - 0.6 m layer when compared to the control treatment without gypsum (Figure 3C). As no gypsum leaching effect was previously recorded for K^+ in this experiment (Michalovicz et al., 2014, 2019; Vicensi et al., 2020a), it can be inferred that the observed effects on soil K^+ are likely because plants absorb significantly greater quantities of K^+ than Mg^{2+}. Crop plants received K^+ through the application of NPK fertilizers during each growth season at a higher quantity than K^+ was recycled by crop residues under gypsum application. This phenomenon increased grain and biomass yield of Poaceous crops, and root growth (Vicensi et al., 2020b), counteracting natural K^+ leaching and providing higher K^+ contents to the soil.

The elevated B content in the 0.4 - 0.6 m soil layer with 9 $Mg_{[gypsum]}$ ha^{-1} in comparison to 3 $Mg_{[gypsum]}$ ha^{-1} (Figure 4C) can be attributed to a combination of factors. As the yield gains for Gramineae species were higher with 3 $Mg_{[gypsum]}$ ha^{-1} over time, it may have resulted in higher B export by harvested grains and forage biomass since 2009. The gypsum utilized is a residue derived from the phosphorus fertilizer industry that exhibits residual levels of nutrients (van Raij et al., 2001). In consideration of the potential for B supply by gypsum in the past, the 3 $Mg_{[gypsum]}$ ha^{-1} rate may have contributed to a smaller amount of B to be recycled by the crops over time than the 9 $Mg_{[gypsum]}$ ha^{-1} rate.

In all treatments and soil layers, the B contents of the soil (Figure 4D) remained above the critical level of 0.3 mg dm^{-3} for Paraná (SBCS/NEPAR, 2017), Rio Grande do Sul, and Santa Catarina (SBCS/NRS, 2016), the three southern states that collectively account for more than 90 % of Brazil's barley production. Nevertheless, the application of B rates resulted in an increase in soil B levels within the 0.0 and 0.2 m depth, which is the diagnostic layer for barley. Furthermore, the barley grain yield exhibited a quadratic adjustment to B rates for both cv. Danielle and Imperatriz in 2021, and cv. Danielle in 2022.

Given that H_3BO_3 does not bind to soil particles and is easily leached into deeper soil layers (Marschner, 2012), and considering that no effect of B rates was observed below 0.2 m depth in 2022 after a barley season with 1,070 mm of precipitation during the second consecutive year of B fertilization in winter, it is likely that the B rates tested were insufficient to cause B leaching. The grain yield response of the cv. indicates that the B rates used were appropriate.

The findings indicated that applying gypsum for soil acidity management necessitates implementing additional measures, namely B fertilization, in areas designated for barley cultivation. The lower yield of barley cv. Danielle observed under the residual effect of gypsum, when coupled with the absence of B fertilization, and the favorable response of cv. Imperatriz to gypsum, even 12 years after its application underscores the necessity of understanding the specific characteristics of the cv.

In this long-term experiment, the application of 3 and 6 $Mg_{[gypsum]}$ ha^{-1} has been observed to increase the yield of Poaceous crops, including corn, barley, and wheat, since the initial crop seasons (Michalovicz et al., 2014). Furthermore, the application of 9 and 12 $Mg_{[gypsum]}$ ha^{-1} has demonstrated enhanced relative performance in subsequent years, attributed to a greater residual effect (Vicensi et al., 2020b). The higher yield results under gypsum rates led to increased nutrient extraction and export, resulting in greater nutrient depletion in the soil compared to the gypsum control. This depletion was particularly evident in the B content, which affected the cv. Danielle more intensely.

The yield of cv. Imperatriz exhibited an increase due to the residual effect of application of gypsum rates in 2021. However, this effect was not observed in the subsequent rainier crop season of 2022. The rainfall throughout the barley growth cycle in 2021 was 30 % lower than in 2022 (Figure 1). It may be posited that the residual effect of gypsum on soil fertility and root environment in subsurface layers resulted in improved conditions for water and nutrient absorption, thereby increasing yield. This hypothesis has been previously demonstrated in this same experiment for barley (Michalovicz et al., 2014).

In 2021, cv. Imperatriz exhibited a yield up to 30 % higher than that of cv. Danielle. This difference can be attributed to the two cv. origin from different geographical germplasm banks (Rerkasem et al., 2020).

The observed increase in grain yield in both cv. in the 2021 season and in cv. Danielle in the 2022 season as a function of the B rates suggests that the current soil critical level of 0.3 $mg_{[B]}$ dm^{-3} in southern Brazilian states may require reevaluation. Modern barley cv., particularly those derived from European germplasm banks, such as cv. Danielle, may exhibit not only enhanced yield potential, but also elevated B requirements. This necessitates a reassessment of B interpretation values for soil tests and the formulation of novel recommendations for B fertilization.

Furthermore, the increase in barley grain yield in response to 1 $kg_{[B]}$ ha^{-1} B application has been documented in other studies (Wróbel, 2009; Rahman and Schoenau, 2020). For cv. Danielle, the MER was 2.85 $kg_{[B]}$ ha^{-1} (Figure 5A). Therefore, based on the 2021 crop cycle, rates between 1 and 2.85 $kg_{[B]}$ ha^{-1} are possible for cv. Danielle. For cv. Imperatriz, the MER was estimated at 1.91 $kg_{[B]}$ ha^{-1} in 2021 and the rate of 4 $kg_{[B]}$ ha^{-1} was observed to reduce grain yield, potentially due to an excess of B and its toxic effect. A similar effect of B toxicity was observed at the rate of 4 $kg_{[B]}$ ha^{-1} in sunflower (Archana and Pandey, 2021) and rice (Riaz et al., 2021).

The 1000-GW in cv. Danielle was found to decrease in a negative linear model as a function of B, whereas grain yield was observed to increase with the application of B rates. Boron is a nutrient associated with pollen tube formation (Oldoni et al., 2018). Consequently, a plant better nourished with B will increase the number of formed seeds and have more drains to distribute the photoassimilates, reducing the individual weight of seeds. This partially explains the behavior of increased yield and reduced 1000-GW due to B fertilization in cv. Danielle.

The 1000-GW exhibited a response to the residual effect of gypsum only in the cv. Imperatriz, aligning with the positive impact of gypsum on the grain yield of this cultivar. These findings highlight the existence of cultivar-specific differences that warrant further investigation. While cv. Danielle exhibited a yield level that was consistent across both crop seasons, the average grain yield of cv. Imperatriz in 2022 was 20 % lower compared to 2021. Furthermore, the average grain yield of cv. Imperatriz in 2022 was only 5 % higher than that of cv. Danielle, which demonstrated lower yield potential in both crop cycles, but exhibited yield stability.

The greater occurrence of precipitation in 2022 was also associated with a greater occurrence of cloudy days, lower levels of solar radiation, lower average temperatures, and soil waterlogging. These conditions challenged the crop performance compared to 2021; nevertheless, they significantly impact yield only for cv. Imperatriz. Although frosts were more frequent in 2021, particularly at the beginning of the crop season, in 2022 the average temperature was 0.7 °C lower compared to that in 2021 (Figure 1C). Cultivar Imperatriz originates from national breeding in subtropical environments in southern Brazil, while cv. Danielle originates from a breeding program in a temperate environment, which is more adapted to cold temperatures.

The increases in soil B contents and grain yield observed in barley cv. Danielle (2021 and 2022) and Imperatriz (2021) following B fertilization indicate that the current soil critical level of B should be reevaluated in southern Brazilian states, particularly in light of the advent of modern barley materials.

The residual effect of gypsum rates did not affect the grain yield of cv. Danielle; however, it did increase the yield of cv. Imperatriz in the 2021 crop season, which was not as cold and rainy as the 2022 season. Without B fertilization, the long-term effect of gypsum decreased barley productivity due to the depletion effect of the higher productivity, and subsequent nutrient export, provided by gypsum in past harvests.

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Conceptualization: Muller MML. **Data curation**: Bombardelli TJ, Umburanas RC, Michalovicz L, Muller MML. **Formal analysis**: Bombardelli TJ, Umburanas RC, Michalovicz L, Camilo EL, Pott CA, Muller MML. **Methodology**: Bombardelli TJ, Umburanas RC, Michalovicz L, Camilo EL, Pott CA, Muller MML. **Writing-original draft**: Bombardelli TJ, Muller MML. **Writing-review & editing**: Bombardelli TJ, Umburanas RC, Michalovicz L, Camilo EL, Pott CA, Muller MML.

Conflict of interest

The authors declare that the research was conducted in the absence of any commercial or financial relationships that could be construed as a potential conflict of interest.

Data Availability statement

The raw data supporting the conclusions of this article will be made available by the authors upon request, without undue reservation.

Declaration of use of AI technologies

The authors declare that no AI technologies were used in this manuscript.

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